

**GC Method Optimization Trade Study for RESOLVE:
20-meter Column v. 8-meter Column**

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GC Method Optimization Trade Study for RESOLVE: 20-meter Column v. 8-meter Column

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Nomenclature

<i>C</i>	=	Celsius
<i>GC</i>	=	Gas Chromatograph
<i>Id</i>	=	inner diameter
<i>m</i>	=	meter
<i>MS</i>	=	mass spectrometer
μV	=	micro volts
<i>mm</i>	=	millimeter
<i>ppm</i>	=	parts per million
<i>PLOT</i>	=	porous layer open tubular
<i>psig</i>	=	pounds per square inch gauge
<i>RSD</i>	=	relative standard deviation
<i>s</i>	=	seconds
<i>TCD</i>	=	thermal conductivity detector

I. Abstract

RESOLVE is the payload on a Class D mission, Resource Prospector, which will prospect for water and other volatile resources at a lunar pole. The RESOLVE payload's primary scientific purpose includes determining the presence of water on the moon in the lunar regolith. In order to detect the water, a gas chromatograph (GC) will be used in conjunction with a mass spectrometer (MS). The goal of the experiment was to compare two GC column lengths and recommend which would be best for RESOLVE's purposes. Throughout the experiment, an Inficon Fusion GC and an Inficon Micro GC 3000 were used. The Fusion had a 20m long column with 0.25mm internal diameter (Id). The Micro GC 3000 had an 8m long column with a 0.32mm Id. By varying the column temperature and column pressure while holding all other parameters constant, the ideal conditions for testing with each column length in their individual instrument configurations were determined. The criteria used for determining the optimal method parameters included (in no particular order) (1) quickest run time, (2) peak sharpness, and (3) peak separation. After testing numerous combinations of temperature and pressure, the parameters for each column length that resulted in the most optimal data given my three criteria were selected. The ideal temperature and pressure for the 20m column were 95°C and 50psig. At this temperature and pressure, the peaks were separated and the retention times were shorter compared to other combinations. The Inficon Micro GC 3000 operated better at lower temperature mainly due to the shorter 8m column. The optimal column temperature and pressure were 70°C and 30psig. The Inficon Micro GC 3000 8m column had worse separation than the Inficon Fusion 20m column, but was able to separate water within a shorter run time. Therefore, the most significant tradeoff between the two column lengths was peak separation of the sample versus run time. After performing several tests, it was concluded that better detection via good peak separation with a longer run time is a better asset than moderate peak separation with a shorter run time. Even given that RESOLVE is highly interested in water and that mission timeline is of significant importance given the short seven-to-ten-day mission timeline, worse detection with an 8m column may lead to overlooking other substances existing on the moon that could advance planetary science. Thus, I recommend the 20m column. However, if mission timeline and water separation are deemed the highest priority, the 8m column should be selected due to its ability to separate water within a shorter run time than the 20m column.

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II. Introduction

Regolith and Environment Science and Oxygen and Lunar Volatile Extraction (RESOLVE) is the payload on a short-duration Class D lunar mission. A Gas Chromatograph (GC) will be included in the payload instrument suite. A GC is an instrument used for separating compounds in a gaseous state. These compounds may either exist in a gaseous state or be vaporized without being decomposed. Some uses of a GC include determining the identity or purity of a certain substance. Another use is for separation of the different components of a gaseous mixture, which is what my experiment entailed.

The purpose of this experiment was to determine what GC column length should be selected given the requirements for good separation in a short run time where water is the commodity of most interest. It is important to note that although water identification and quantification is the critical priority of the Resource Prospector, the quality of data should not be overlooked while focusing on water.

Two different GCs were used in this experiment, the Inficon Fusion and the Inficon Micro GC 3000. The two instruments had different column lengths, so the differences in the data when testing may be observed. A tedlar bag was used which was filled with a mixture of gases containing 3.99% hydrogen, 5.0% carbon monoxide, 4.02% methane, 5.0% oxygen, and 5.0% helium in a balance of nitrogen with added water saturated at room temperature. Throughout testing helium was used as a carrier gas at all times. Helium is commonly used as a carrier gas because it is inert and will not react with the gases being tested.

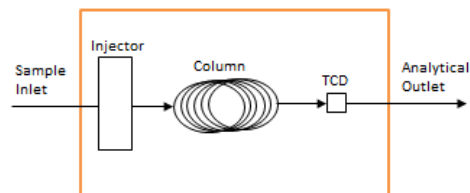


Figure 1. GC Components

Inside a GC, there is a column which is responsible for the separation of the various gasses in the sample. After separation, the gases are detected by a Thermal Conductivity Detector (TCD) which works by comparing the difference in thermal conductivity between the carrier gas and sample gas. A representation of the column and the TCD can be seen in Figure 1. The different samples injected into the GC exit the column at different times, which is known as the retention time. The retention time is used to differentiate between the various gases in the sample. The retention time usually increases proportionally with column length.

The Inficon Fusion has a 20m long column with a 0.25mm .25 mm inner diameter (Id). The run time used for this column was 130 seconds. The Inficon Micro GC 3000 has an 8m long column and with 0.32mm Id. The run time used was 60 seconds. Both of these run times are appropriate for their respective column length. The purpose of this experiment was to determine the best GC column length for RESOLVE by evaluating the data acquired using optimal column temperature and pressure for good peak separation in an acceptable run time. Using a tedlar bag with known gases, temperature and pressure varied to observe which combination resulted in the best data. The best results fulfilled the pre-determined criteria most effectively. These criteria included (1) short run time, (2) peak sharpness, and (3) separation of the peaks

A. How a Column Works to Separate Substances by Their Retention Time

The column in a GC represents the stationary phase, while the carrier gas represents the mobile phase. The different substances injected into a GC are separated based on their interaction with the stationary phase. If the interaction is stronger, it takes more time to react with the stationary phase and therefore the retention time is greater. If the interaction is weaker, the substance requires less time to interact with the stationary phase and therefore has a shorter retention time. The columns used in the two GCs were both capillary porous layers open tubular (PLOT). There are other factors that affect the separation of compounds, such as column temperature and column length.

Throughout the experiment, a very high column temperature produced shorter retention times but the resulted in poor separation of the substances. The column length varies between different GCs based on the needs of the user. In general, a longer column may improve the separation of the substances but retention time may increase and the observed peaks on the graph may be broader. When testing with a shorter column, lower temperatures provided better results compared to the GC with the longer column which performed better at a higher temperature.

III. Procedure

The GCs used in this experiment were the Inficon Fusion Gas Analyzer and the Micro GC 3000. This Fusion contains a Thermal Conductivity Detector (TCD) with a 1ppm detection limit. It also contains a 20m long column with a 0.25mm Id. Once a sample was injected into the GC, it was then analyzed and the results were transferred to a laptop. After the data was transferred to the laptop, it was then examined it and compared to other data. The data

was also examined using Microsoft Excel. After determining the optimal parameters for the GC/column, tests were performed using a different GC with a shorter column. The Micro GC 3000 has an 8m column with a .32mm ID. I then compared the data and confirmed each GC/column performs best at unique temperatures and pressures. I was able to then conclude which of the two columns provided the best separation under their optimal conditions and offer my recommendation to the RESOLVE team for inclusion in the flight payload.

A. Preparing the GC

Before powering on the GC, the carrier gas must be flowing. The carrier gas used in this experiment was helium, since it is inert. The instrument-grade helium gas was supplied from a K-bottle and the pressure was regulated to approximately 60 psig. The tedlar bag providing the mixed gas sample was then attached to the GC inlet located on the outside of the GC. To prevent leakage from the tedlar bag when the GC was not operating, the valve was kept closed. Once testing began, the valve was opened to allow the gases to flow out of the tedlar bag and into the inlet. In between operations the GC was not typically turned off; it was held in a standby method at 30 °C and 20 psig. The GC operated better when it was not turned off, but put into a standby state. That way the instrument did not cool down completely and water did not condense in the instrument.

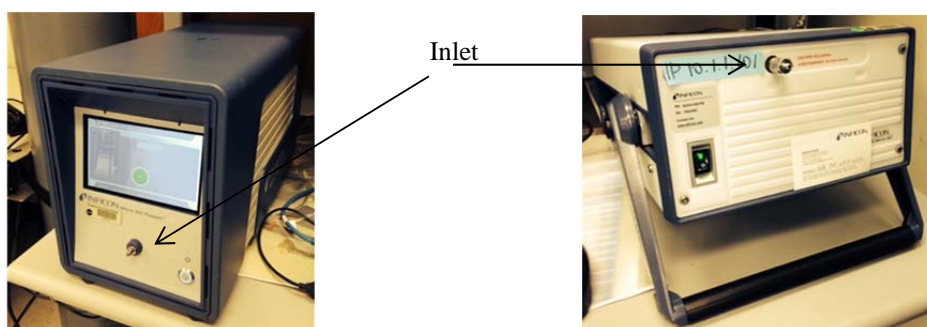


Figure 2. Inlets of Fusion GC, 20m Column (left) and Micro GC 3000, 8m Column (right)

B. Running Tests

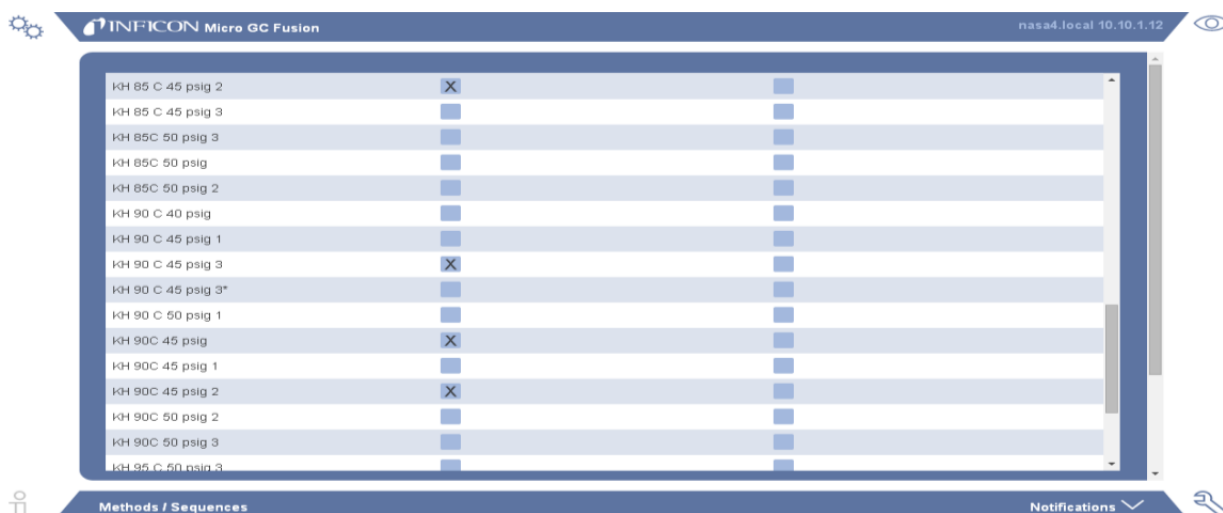


Figure 3. Fusion Gas Chromatograph Methods

The primary goal of this experiment was to determine the optimal column temperature and pressure to accomplish the best separation, leading to a recommendation of the best column length. The data resulting from each GC run was a plot with peaks containing one or several of the gases in the tedlar bag sample. Ideally, the sample gases should be separated into distinct peaks which are not too broad. In order to achieve these results, the GC had to be run using an optimal combination of column temperature and column pressure. The column temperature

describes the temperature of the heated column, while the pressure describes the head pressure at which the sample gases are pushed through the column.

As seen in Figure 3, numerous methods were created containing a variety of set points for the GC (Fusion shown here). A new method was used to test each combination of column temperature and pressure, while keeping the run time the same (130 seconds for the Fusion and 60 seconds for the Micro 3000). With a 20m column, it was not recommended to shorten the run time since it takes a significant amount of time for the gases to work their way through a column of that length. Each method was run three times to secondarily determine the accuracy of the instrument from run to run, resulting in three separate plots. Once a test was performed and a graph was produced (reference Figure 4), the data was imported into Excel to begin the analysis and calculate the percent RSD (Relative Standard Deviation). Figure 4 is an example of a graph produced by the GC. Once all of the data was uploaded to a computer and graphed, it was examined and the best possible temperature and pressure to be used for that particular GC was determined. For comparison, the Inficon Micro GC 3000 with a shorter column length was evaluated to determine which instrument produced the best separation. The Micro 3000 had an 8-meter column, far shorter than the 20-meter column in the Fusion GC which was evaluated initially.



Figure 4. Fusion GC Chromatogram

IV. Data and Results

A. Peak Separation

Various temperature and pressure combinations were tested to find the best possible peak separation. For the GC Fusion, the temperatures ranged from 70°C to 120°C, while the pressure ranged from 40psig to 50psig. The data was outputted in a graphical form where optimal separation would result in four distinct peaks. The largest peak is the inert gases and the 3 other peaks are methane, carbon dioxide and water – from left to right with increasing retention time. At certain column temperatures, the methane peak was connected to the large inert peak as a shoulder, meaning that there was another peak but it was still connected and not distinctly separated. One goal of this experiment was to separate methane from the inert gases peak. (PLOT-Q columns do not typically have the ability to separate the inert gases, so they all elute from the column as one peak. Other columns with specific column phases can separate the inert gases.) In an attempt to separate methane, data was acquired at the following column temperature and pressure combinations: 120°C at 45psig; 110°C at 45psig; and 100°C at 45psig.

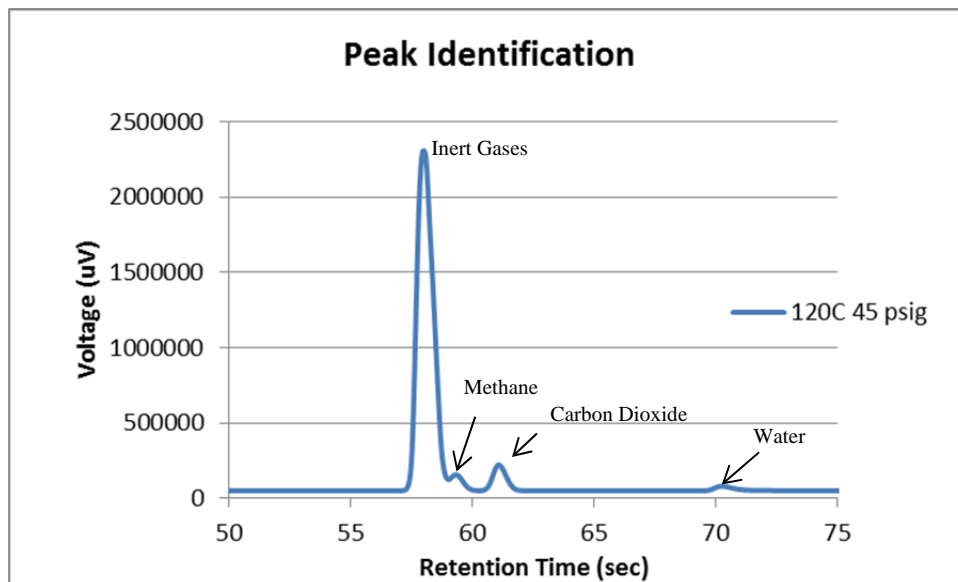


Figure 5. Fusion GC Data 120°C 45psig

This data can be seen in Figure 5 through Figure 7. As seen in Figure 5, the methane peak is barely separated from the inert gas peak. The area under each peak is proportional to the amount of that substance present in the sample. From Figure 5 - Figure 7, there is a large amount of the inert gases and a very little amount of water. In Figure 6, the methane peak is slightly more separated from the inert gas peak. In Figure 7, the methane is even more separated. Maintaining a constant pressure of 45psig and gradually lowering the temperature by increments of 10°C, the methane peak was separated from the inert gases peak.

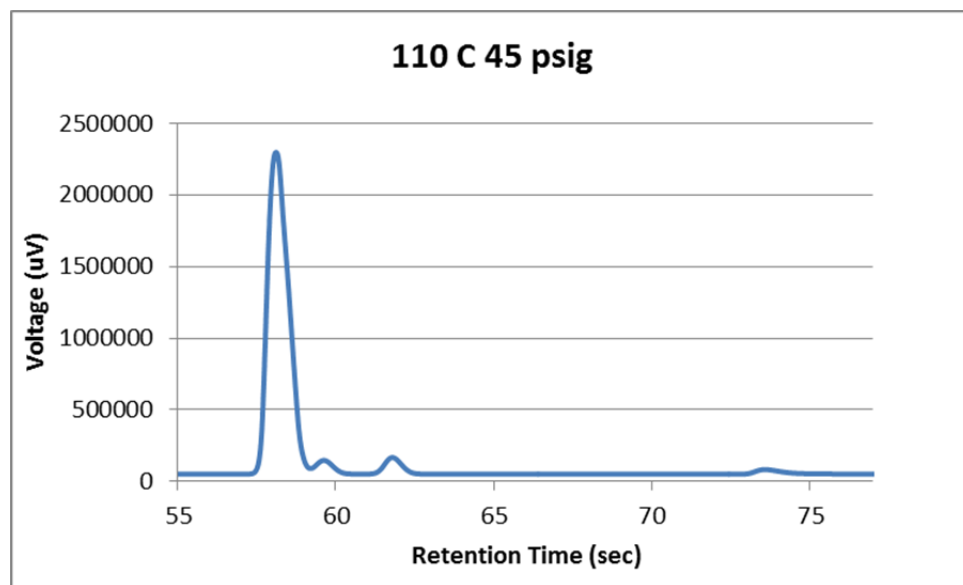


Figure 6. Fusion GC Data 110°C 45psig

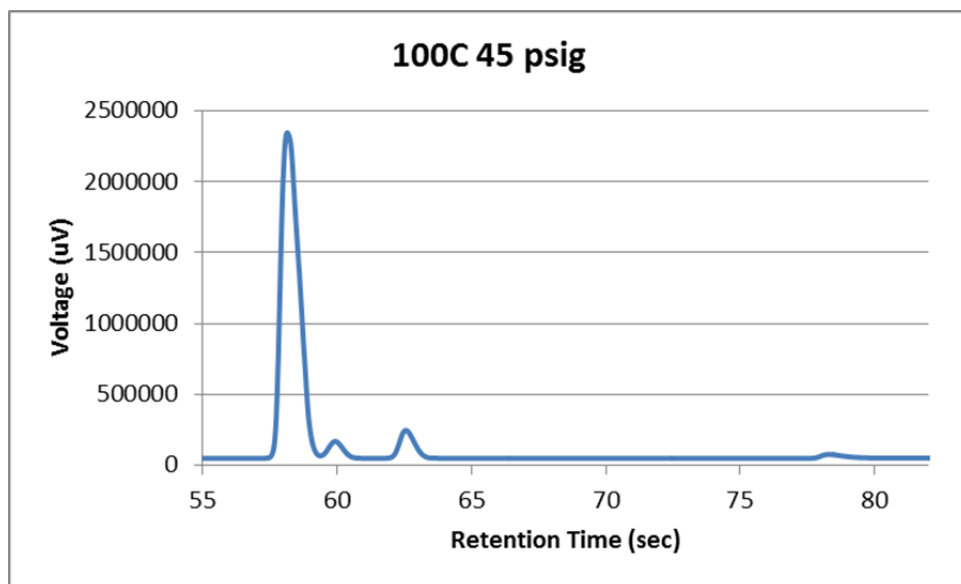


Figure 7. Fusion GC Data 100°C 45psig

The peak separation using the Micro GC 3000 was not as distinct, compared to the data seen using the Fusion. When testing with Micro GC 3000, a method with 90°C and 20psig for column temperature and pressure was created. This GC operated better at a lower temperature compared to the Fusion. As seen in Figure 8, the carbon dioxide peak (second peak) is barely separated from the inert gas peak. Using the Fusion, although the methane peak (second peak) is not fully separated from the inert gases, the carbon dioxide peak (third peak) is still fully separated as can be seen in Figure 7.

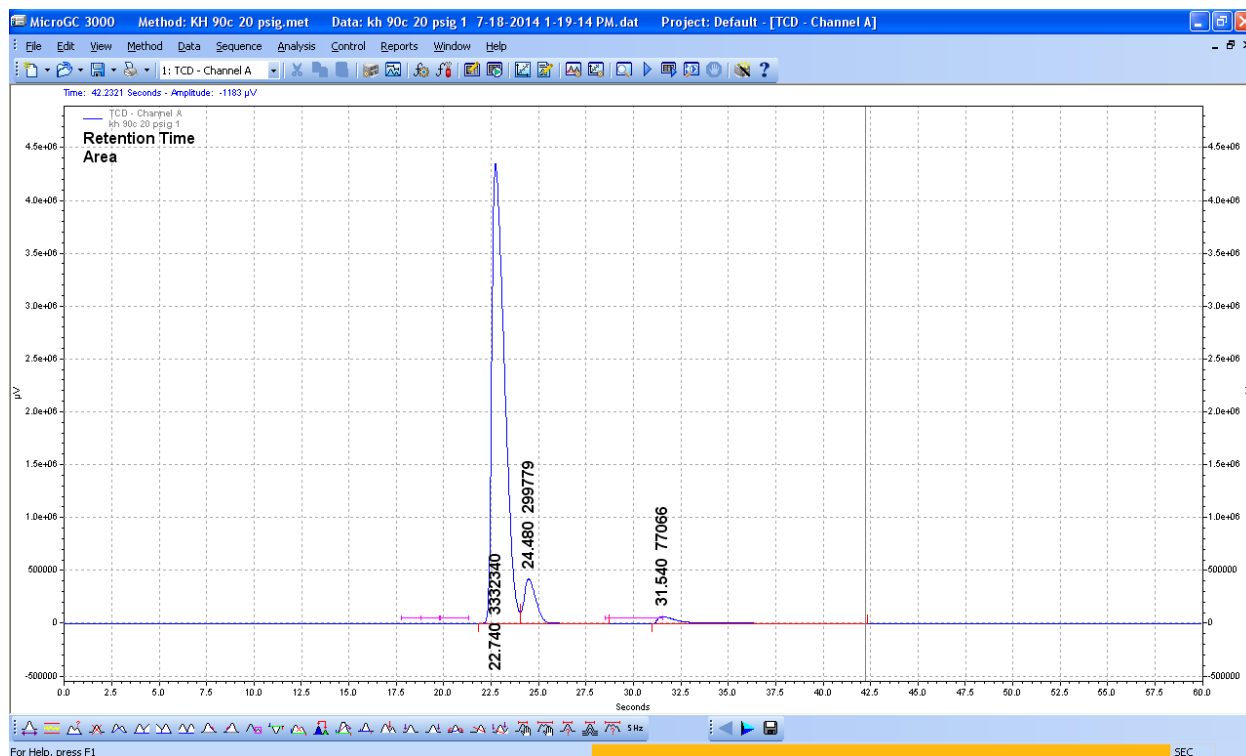


Figure 8. Micro GC 3000 at 90°C and 20psig

B. Comparing the Degree of Peak Separation

As testing progressed, the peaks from the Fusion appeared to separate as the temperature was decreased and the pressure remained the same. However, leaving the pressure constant at 45psig and decreasing the temperature increased the retention time and made the peaks wider. Although separation was achieved, the increased retention time was not ideal due to the increase in timeline this would cause during RESOLVE's operation on the moon. As seen in Figure 9, multiple runs with the column pressure set to 45psig and varying temperatures are compared with one run set to a lower column pressure. At 45psig column pressure, as the column temperature decreased, the graphs moved farther right which means that the retention time is increasing. At 90°C and 45psig, the retention time was the largest. In Figure 9, this conclusion is best seen with the water peaks staggered on the far right of the graph.

When testing with the Micro GC 3000, the peak separation was not as good compared to the peak separation using the Fusion. The methane peak was not separated and the carbon dioxide peak was significantly closer to the inert gas peak, compared to the data seen using the Fusion. Comparing Figure 11 and Figure 14, it can be seen how the peak separation varies between the two instruments.

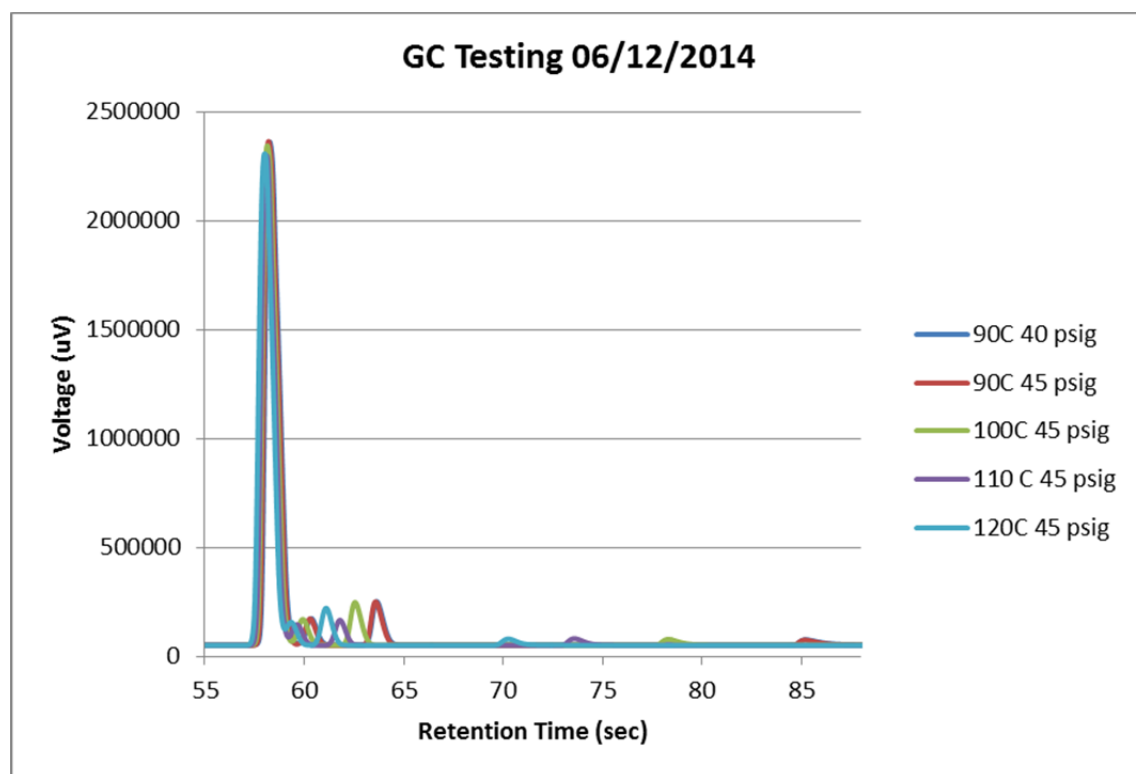


Figure 9. Fusion GC Data Comparing Constant Column Pressure and Variable Temperature

C. Changes in Pressure Using Inficon Fusion

Once the peaks were separated, the goal was to keep them separated without increasing the retention time too much or making the peaks very broad. Increasing the retention time means that the run time increases, which is not ideal for this short-duration mission. If the same data can be achieved with a lower run time, then it is more desirable. When the peaks are too broad, they have a larger retention time. To determine the effect of a column pressure change on the data, the column temperature was held constant and the pressure was changed. As seen in Figure 9, a slight change in pressure (5psig) did not have an obvious effect on the graph. In fact, the plots looked nearly identical. In Figure 9, the plots for 90°C at 40psig and 90°C at 45psig seem to overlap. However in Figure 10, a slight pressure increase does have a small effect on the data. A change of 10psig resulted in a more significant change in the data. The graphs do not overlap, and are clearer.

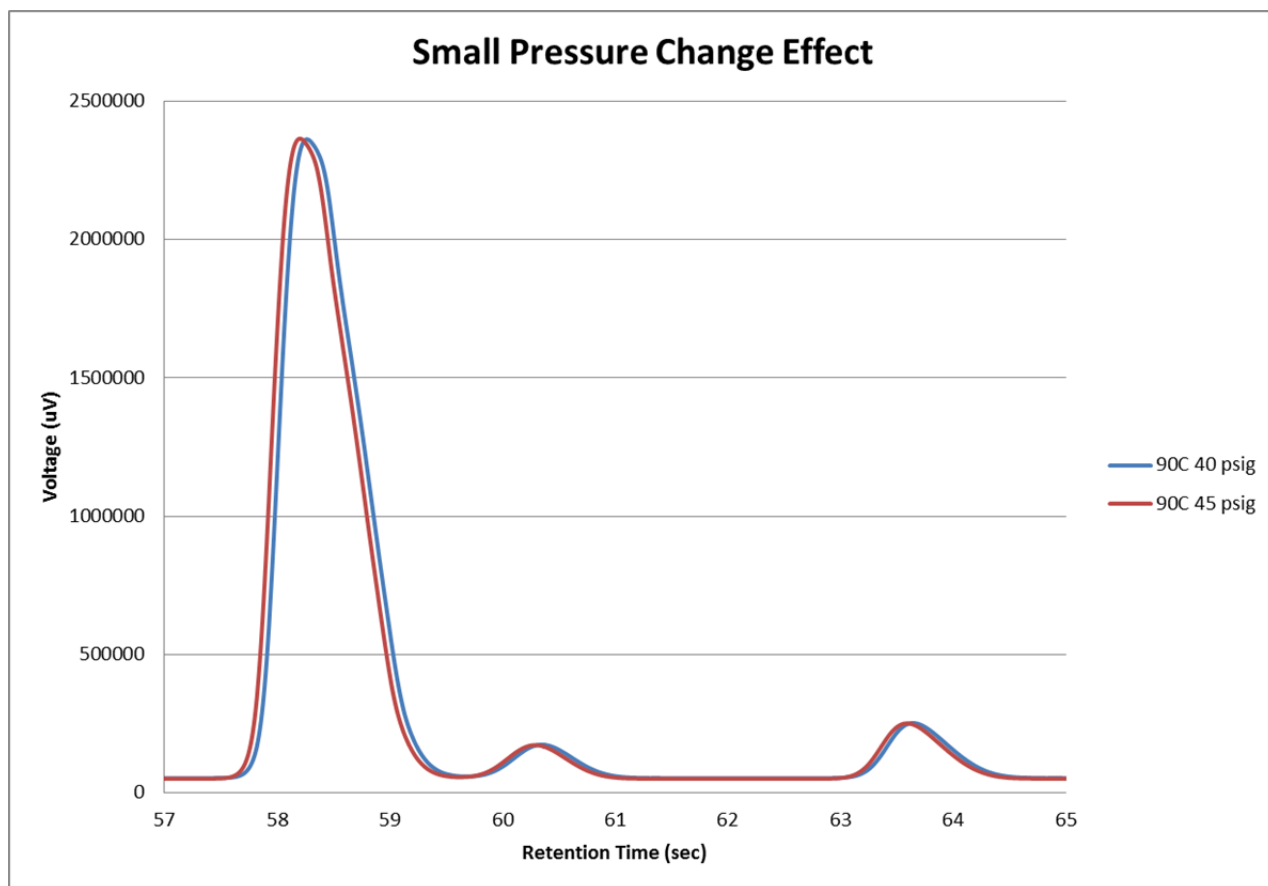


Figure 10. Fusion GC Small Pressure Change Effect

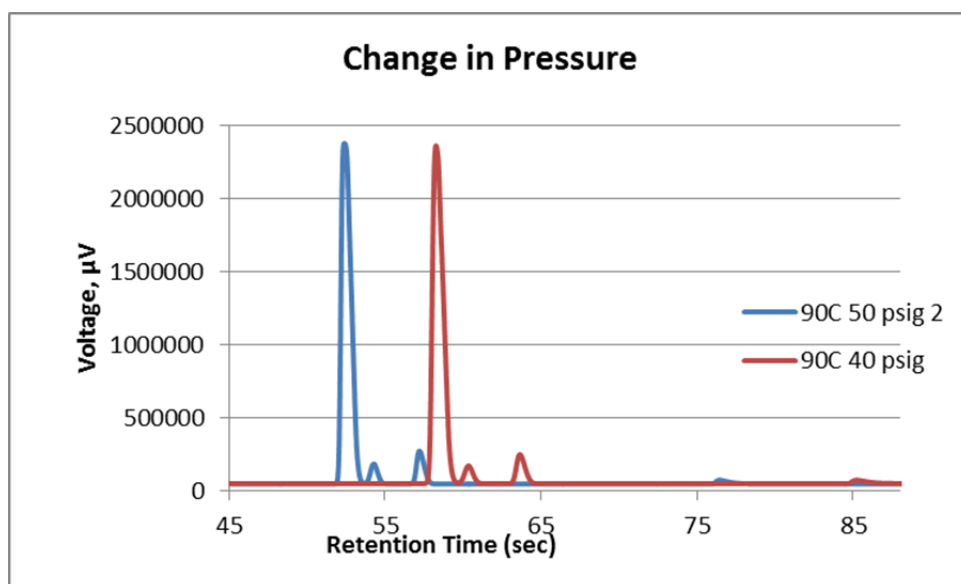


Figure 11. Fusion GC Data Comparing Variable Pressure at 90°C

As seen in Figure 11, the separation between the peaks remained constant, meaning that good separation was maintained. At 50psig, the graph was shifted toward the left showing lower retention times compared to 40psig. The same result may be seen at a different temperature, such as 85°C. In Figure 12, the temperature stayed constant at 85°C, but the pressure was increased in increments of 50psig. At 50psig, the peaks had the shortest retention times.

The separation of peaks throughout the graph remained constant, but the position of the graph changed, shifting left as pressure increased. The same may be observed at a different lower temperature, such as 75°C.

As seen in Figure 13, the test with the highest pressure resulted in the lowest retention time. At 52psig, the plot was the farthest on the left, compared to the graph at 45psig which was located farthest right. As seen in Figure 10 through Figure 13, as the column pressure increased, the peaks shift farther left with lower retention times.

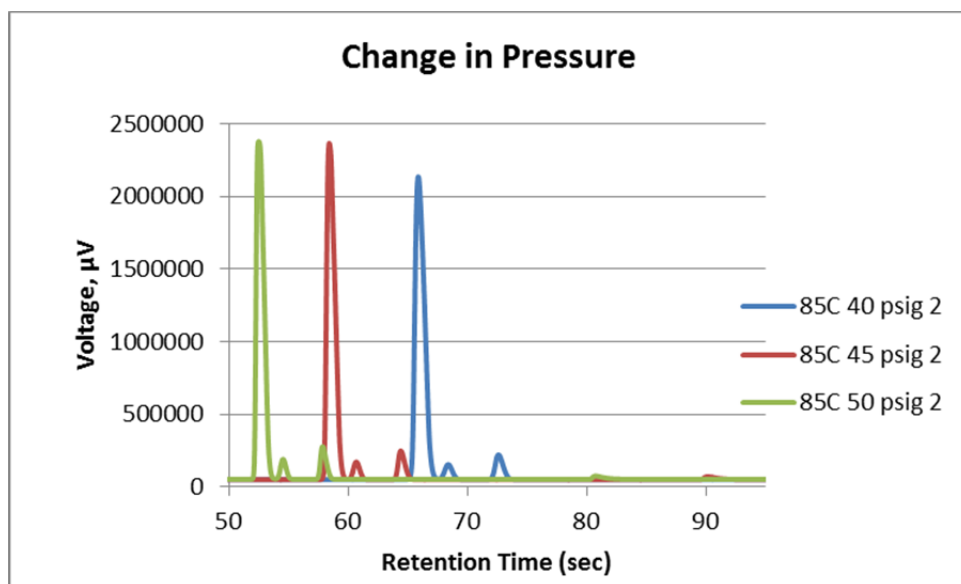


Figure 12. Fusion GC Data Comparing Variable Pressure at 85°C

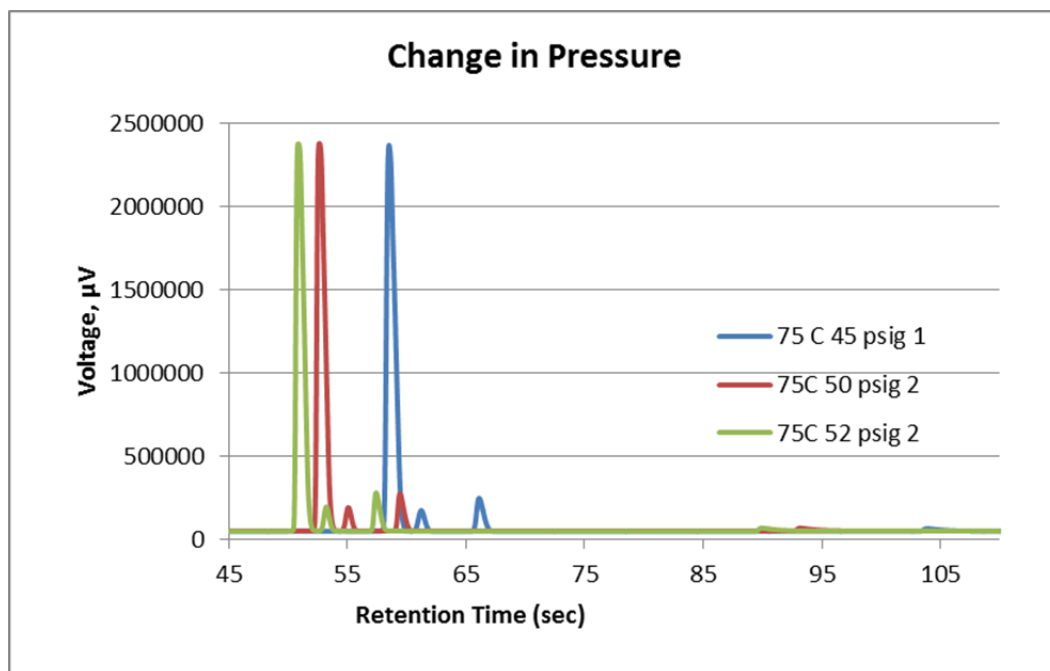


Figure 13. Fusion GC Data Comparing Variable Pressure at 75°C

D. Changes in Pressure Using Inficon Micro GC 3000

The effect of pressure change was tested using the Micro GC 3000 as well. An increase in pressure using this shorter column resulted in narrower peaks. This is comparable to the results observed using the longer column in the

Fusion. The two instruments react in similar ways to the changes in pressure but relative to the column. For example, the range of pressures tested in the Micro GC was from 20psig – 30psig, which was significantly lower compared to the Fusion where pressures ranged from 40psig to Fusion's range of 40psig- 50psig.

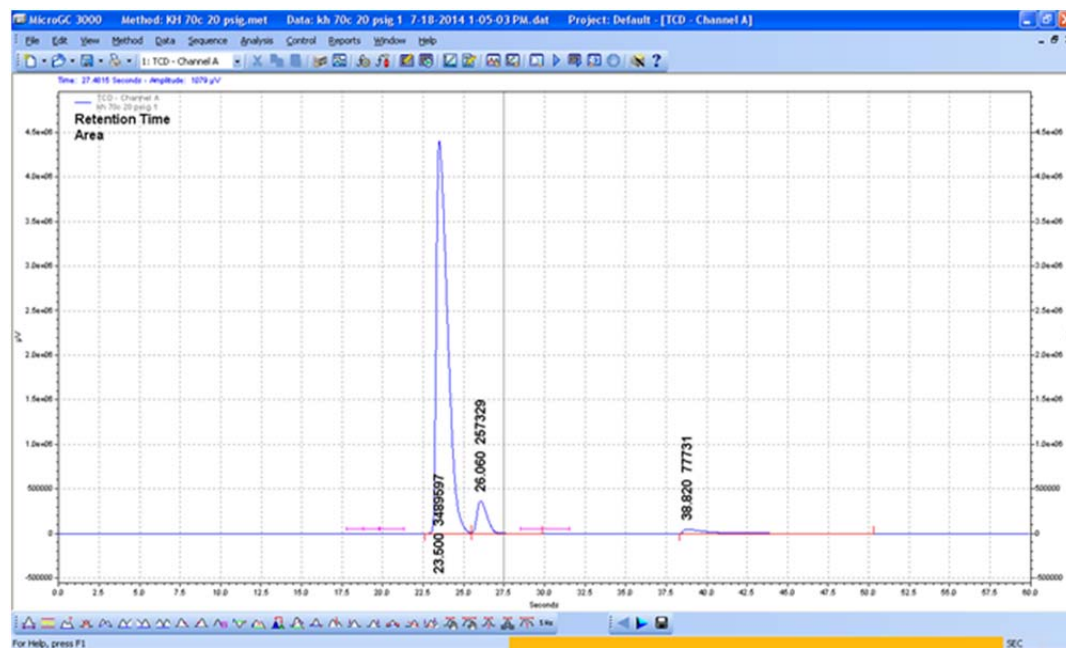


Figure 14. Micro GC 3000 Data at 70°C and 20psig

As seen in Figure 14, the inert gas peak is present but there is no separation of the methane peak. The carbon dioxide peak is also very close to the inert gas peak. As seen in Figure 12, using the longer column in the Fusion, the carbon dioxide peak is relatively far from the inert gas peak, and the methane peak is separated. Even at 40psig, which is a relatively low pressure in the Fusion, the carbon dioxide peak and the methane peaks were separated. With the Micro GC 3000 8m column, the peaks became narrower at 70°C and 30psig compared to the peaks at 20psig.

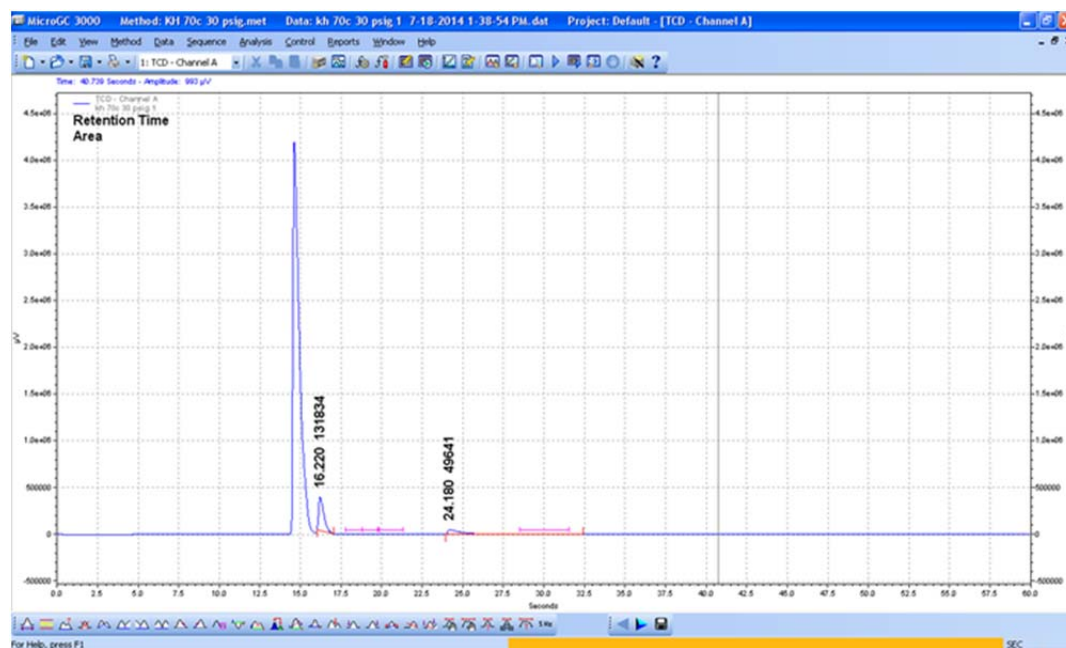


Figure 15. Micro GC 3000 Data at 70°C and 30psig

As seen in Figure 15, an increase in pressure made the peaks more narrow, which is comparable to the results using the Fusion. Although the peaks did narrow and the retention time was lower, there was still no separation of the methane peak and the water peak was difficult to observe.

E. Change in Temperature Using Inficon Fusion

In addition to changing the column pressure, the Fusion column temperature was changed to determine how the data was impacted. The goal was to understand which temperature that would deliver the most optimal results. From initial testing, temperatures above 100°C were not ideal; the peaks were not fully separated. The methane peak was a shoulder on the inert gas peak when the temperature was above 100°C. Below 100°C, the peaks were separated, but now the goal was to establish at what temperature the retention times were shorter and where the peaks were not too broad. In Figure 16, at 90°C and 45psig, the graph has the lowest retention times and the separation of peaks is also good. At 75°C, the separation of peaks was also very good but the retention times were largest and the peaks became broader. As the temperature decreased, the water peaks had a larger retention time in addition to being flatter. In Figure 16, the water peaks on the far right are very flat, compared to the water peaks toward the left.

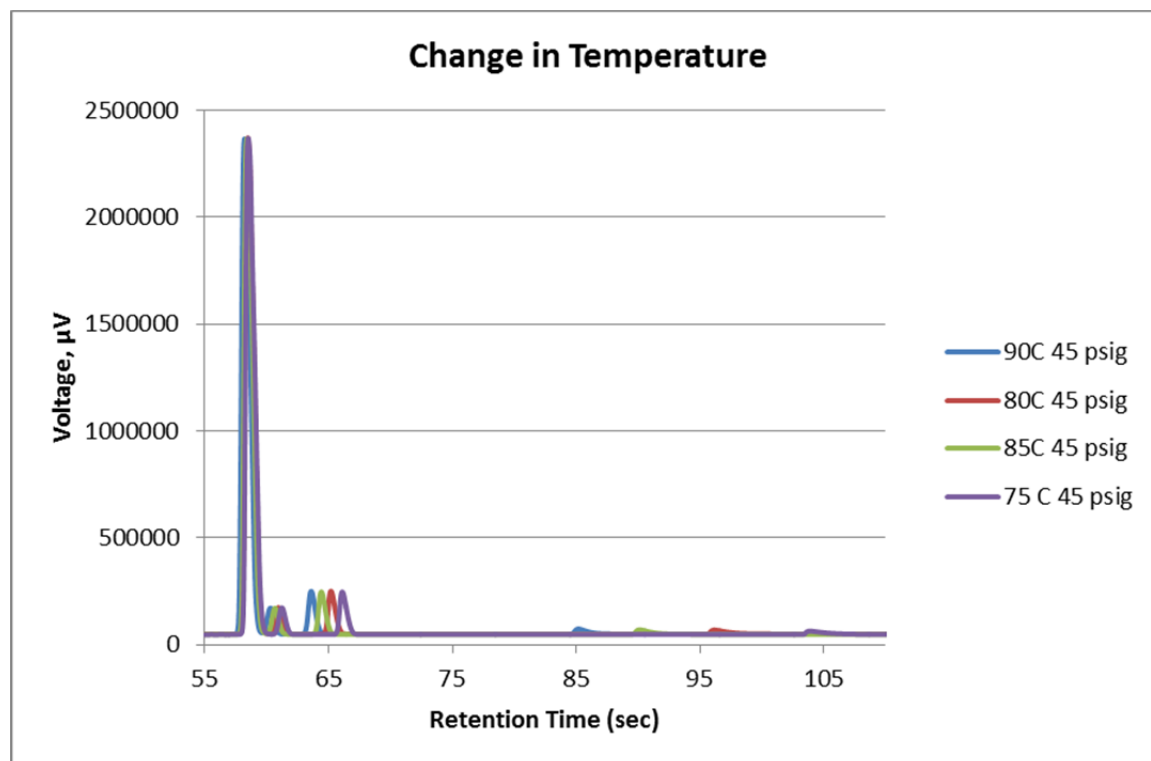


Figure 16. Fusion GC Data Comparing Variable Temperature at 45psig

In Figure 17, similar observations are seen to the ones shown in Figure 16. This time, the temperatures were varied at 50psig instead of 45psig. The separation of peaks is good, but the retention time and the broadness of the peaks are affected. At 95°C, the retention time was the shortest and the water peaks were the sharpest, meaning they were not broad. At 70°C, the water peak was farthest right and broader than the other water peaks. It is nearly flat.

Comparing the data in Figure 16 and Figure 17, the higher column temperatures generally resulted in lower retention times and good separation of peaks. That same high temperature at a higher column pressure resulted in good peak separation in addition to even lower retention times. As seen in Figure 16, a column temperature of 90°C results in good separation of peaks and a shorter retention time compared to the lower temperature. Decreasing temperature increased peak separation but also caused the peaks to become broader. This is why data below 80°C was not ideal. The best data resulted within the range of 85°C to 95°C. Regarding pressure, better data was observed with an increase in pressure.

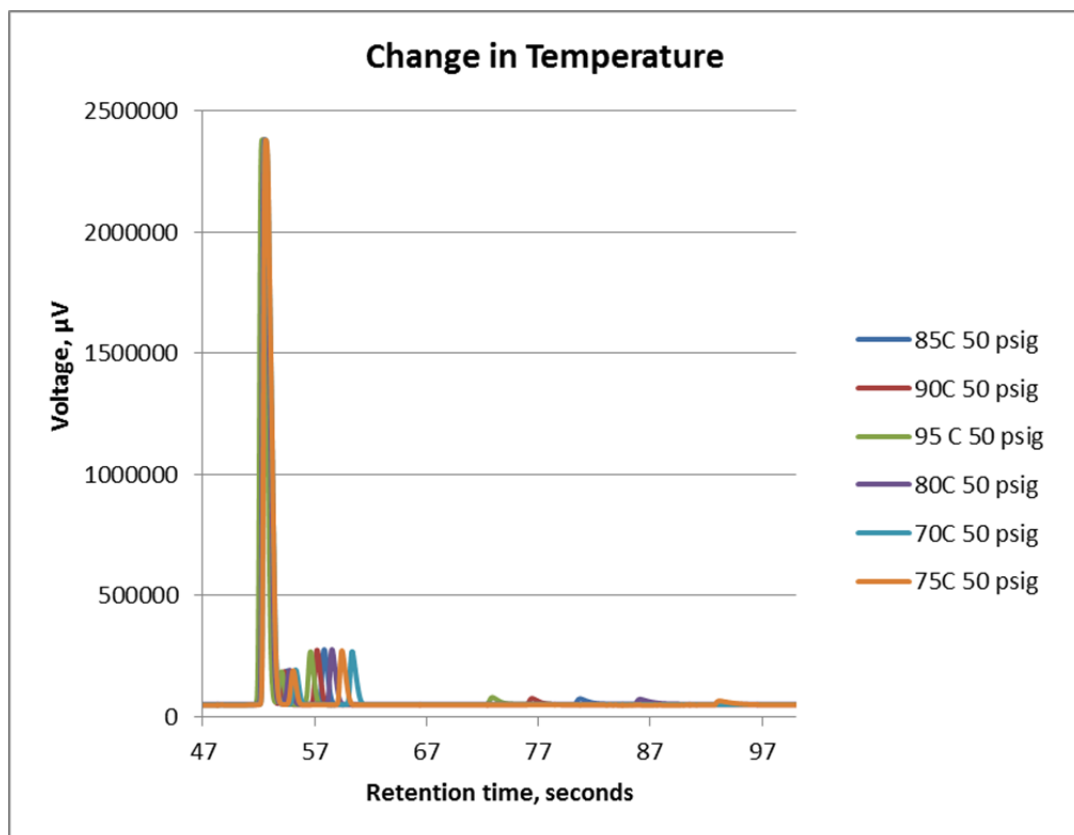


Figure 17. Fusion GC Data Comparing Variable Temperature at 50psig

F. Change in Temperature Using Inficon Micro GC 3000

Using the Micro GC 3000, a decrease in temperature, caused the peaks to become broader and the separation became more defined. This observation was comparable to the results of the Fusion. A decrease of 10°C had a significant impact on the peak separation using the shorter column in the Micro GC 3000. As seen in Figure 18, the carbon dioxide peak is slightly separated from the inert gas peak but not completely at 80°C. It is also relatively narrow. In Figure 19, the peaks are slightly more separated and a little broader at 70°C compared to the ones seen in Figure 18. Using the Micro GC 3000, it can be confirmed that decreasing the temperature increased peak separation, but not as significantly as seen using the Fusion.

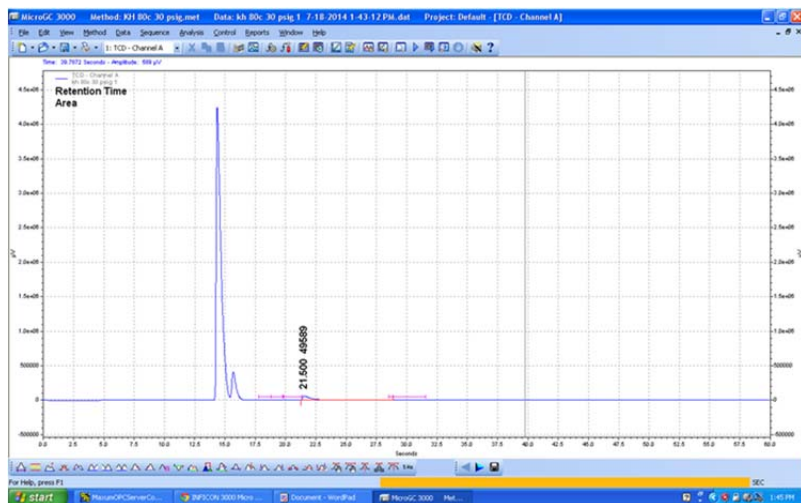


Figure 18. Micro GC 3000 Data at 80°C and 30psig

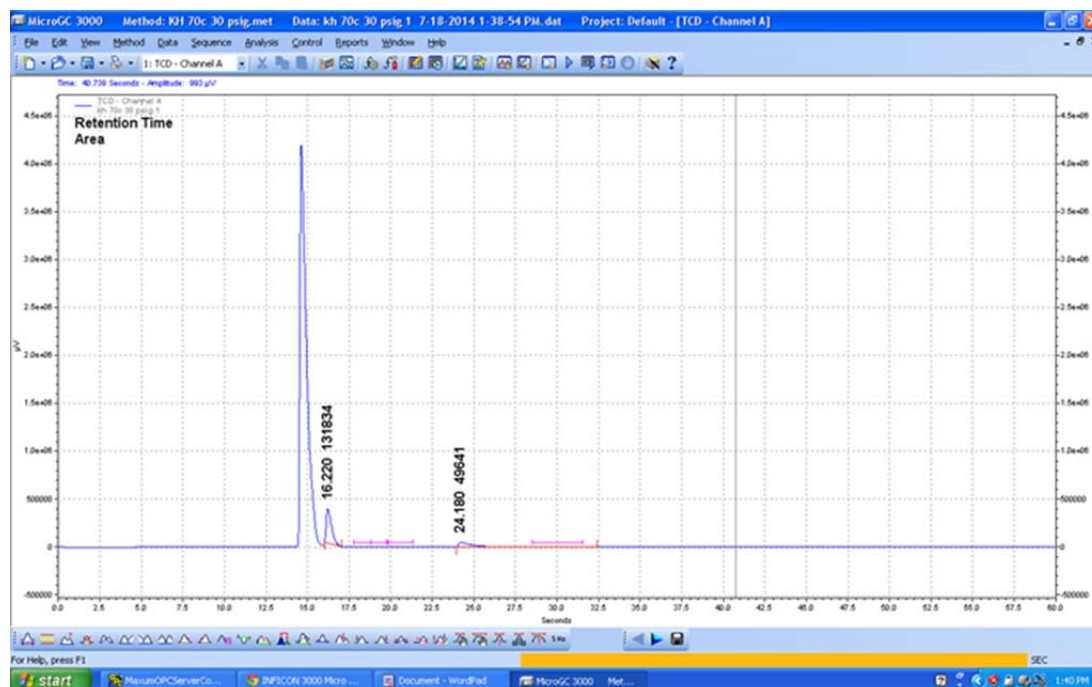


Figure 19. Micro GC 3000 Data at 70°C and 30psig

G. Comparing the Areas of the Peaks

The area of the peaks was proportional to the amount of the substance in the sample. In the graphs, four peaks are seen, one of them distinctly largest. The biggest peak is the inert gas peak, which contains more than one substance. The PLOT column does not have the ability to separate those substances, therefore they elute in one peak. The water peak, seen on the far right of the plot, is the smallest peak, which shows that there is not a large amount of water in the tedlar bag. By monitoring the area, it can be seen whether the peaks grow smaller or larger with changes to column temperature or pressure.

After performing two runs, one at 95°C at 50psig and the other at 100°C at 50psig the peaks were affected in different ways. At 100°C and 50psig, the inert gas peak was slightly smaller than at 95°C and 50psig. As seen in Figure 20, the area of the inert gas peak is slightly larger at 95°C and 50psig.

Inert Gases Peak	Run 1 (uV)	Run 2 (uV)	Run 3 (uV)
100 C 50 psig	1351907.673	1334579.373	1348268.991
95 C 50 psig	1380417.418	1347930.845	1348525.173

Figure 20. Fusion GC Area of Inert Gas Peak with Changing Temperature

Inert Gases Peak	Run 1 (uV)	Run 2 (uV)	Run 3 (uV)
80 C 40 psig	1920919	1906565	1905844
80 C 45 psig	1375244	1369321	1355807

Figure 21. Fusion GC Area of Inert Gas Peak with Changing Pressure

In Figure 21, a decrease in pressure increased the area of the peak. This means that the peak may have become broader. A decrease in temperature also causes the peak area to grow larger, which is why the peaks start to look broad if the temperature is too low. The increase in pressure decreases the area, making the peaks look sharper rather than broader.

H. Choosing the Optimal Conditions

After thorough testing with the Fusion GC, the data was divided into the three best parameters for column temperature and pressure. In order to make this decision, multiple tests were performed, varying temperature and

pressure, keeping one constant and varying the other. The top three conditions that had the best results were 85°C at 50psig, 90°C at 50psig, and 95°C at 50psig. From prior testing, received the best results were observed at 50 psig, where the peaks were separated and not too broad. The temperature which resulted in the best data had to be determined. After examining the data, the best data was observed at 95°C and 50psig. The peak separation was good, the retention time was relatively short, and the peaks were not too broad. Figure 22 confirms these observations. The graph at 95°C and 50psig is farthest left, which means it has the shortest retention times. Also, the peaks for 85°C and 90°C at 50psig are broader. The water peak is a good way to see this. The water peaks are located on the far right side of the graph and are relatively small.

The best possible method using the Micro GC 3000 was also determined. The methane peak was not separated but a slight shoulder was seen. After a few tests, it became evident that the Micro GC operates best at lower temperatures and pressures compared to the Fusion, due to its shorter column. Changes in temperature and pressure have the same effects on both machines but just at difference scales. For example, with the Fusion, the most effective pressure ranged from 40psig to 50psig. With the Micro GC 3000, the pressure ranged from 20psig to 30psig. The best temperature and pressure combination on the Micro GC 3000 was 70°C and 30psig. The slight methane peak and the other peaks were not too broad. For RESOLVE's purposes, water is most important, and was observed at 70°C and 30psig.

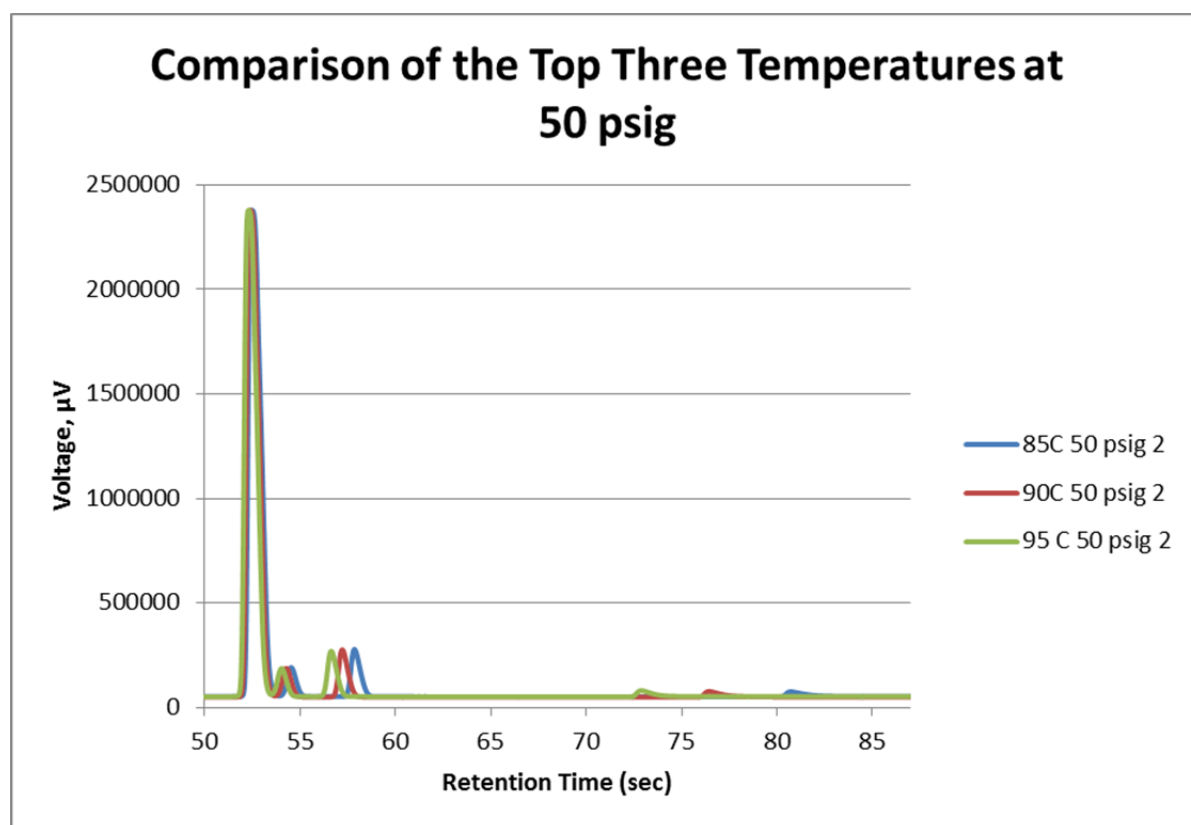


Figure 22. Comparing Top Three Column Pressure and Temperature Combinations for the Fusion GC

I. Consistency of the GC

Throughout the experiment to determine the ideal temperature and pressure for testing, the consistency of the Fusion GC was also monitored. The reason for this test was to determine if accurate data was recorded every time the GC was used. This analysis did not contribute to the ultimate column length recommendation. All of the data collected was not acquired in one day, and it was important to ensure that accurate data was received every time the instrument was used. The data proved to be consistent except when the instrument was turned off. When the GC was turned off and then turned back on, the data was inconsistent and could not be used. Therefore, the GC should not be turned off completely or the initial two to three runs should be thrown out as the instrument stabilizes. Throughout the duration of my testing, the GC was set to Standby mode every day after operation, in order to maintain an

acceptable temperature to prevent water from condensing inside. The Standby method set the GC to 30°C and 20psig. This can be seen in Figure 23.

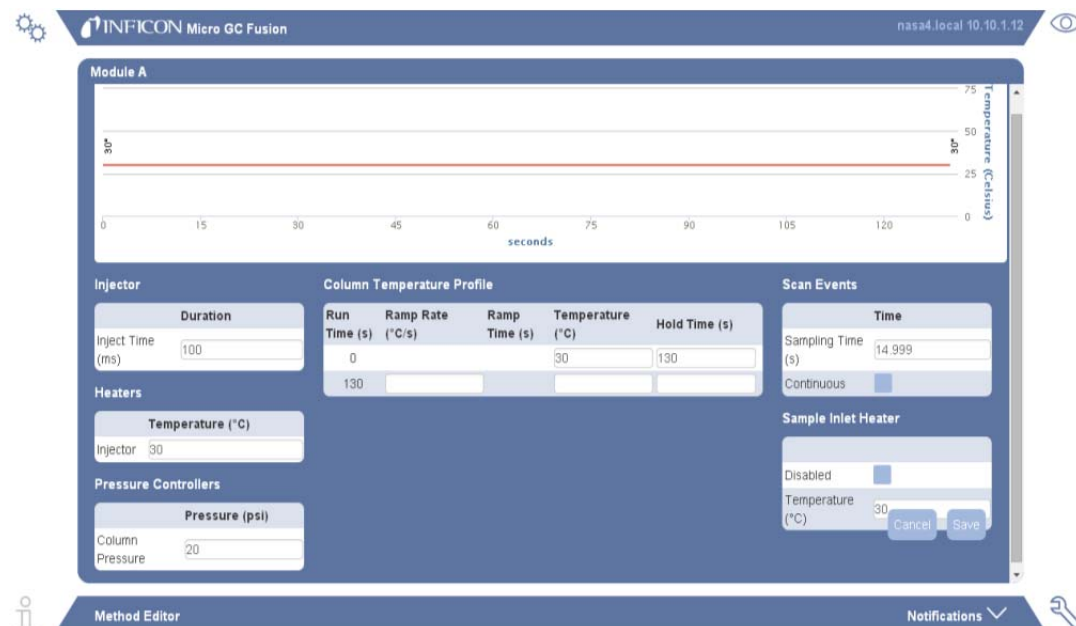


Figure 23. Fusion GC Cool Down Method (Standby)

To determine the consistency of the Fusion GC, the percent RSD was calculated for the retention times at different temperatures and pressures. The percent RSD was calculated using Excel. The formula for %RSD is

$$\%RSD = \frac{stdev(x)}{average(x)} \times 100$$

Equation 1. % RSD

where x is the retention time in seconds or peak area in uV. Industry standard states that an acceptable percent RSD is less than 2%, with increased consistency at lower percent RSD's. In Figure 24, the percent RSD's for all of the peaks at 95°C and 50psig were less than one. All of the percent RSD's in Figure 25 were also less than one, which shows that the data was consistent. At other temperatures and pressures, the percent RSD remains below one. The percent RSD does not factor into the peak separation or broadness, it just shows the consistency of the instrument from run to run and day to day if data runs were separated across days. These tests were done over a span of a few weeks and the percent RSD remained less than one. This shows that throughout the weeks of testing, the machine remained consistent with the data it was producing.

Retention Time for 95 C 50 psig				
	Run 1 (sec)	Run 2 (sec)	Run 3 (sec)	%RSD
Inert gas	52.773	52.736	52.736	0.040497886
Methane	54.045	54.027	54.027	0.019233255
Carbon dioxide	56.645	56.627	56.627	0.018350264
Water	72.6	72.9	72.927	0.249285207

Figure 24. Fusion GC Retention Time %RSD at 95°C and 50psig for Three Runs

Retention Time for 100 C 50 psig				
	Run 1 (sec)	Run 2 (sec)	Run 3 (sec)	%RSD
Inert gas	52.664	52.645	52.7	0.053037068
Methane	53.827	53.809	53.873	0.061306274
Carbon dioxide	56.155	56.109	56.2	0.081027844
Water	70.127	69.918	70.173	0.193949525

Figure 25. Fusion GC Peak Area %RSD at 100°C and 50psig for Three Runs

J. Comparing Data with Different Column Length

To compare how different column lengths may affect the results, tests were performed on two different GC instruments. An Inficon Micro GC 3000 was used. This GC has a shorter column length (8m) than the Fusion GC (20m). Initially, it was planned to test the top three Fusion methods to determine if they result in similar data on a different GC with a different column length. Quickly it was confirmed that each GC operates optimally at different parameters. The GC which was used for most of the testing had a 20m column, and the other one had an 8m column. The column length affects the results on a GC which is why it is important to determine which length is most optimal for the RESOLVE flight payload. The reason for the same parameters not matching is due to the change in column length, change in Id and change in the thickness of the column.

The same parameters did not result in the same data. 50psig would be too high on an 8 meter column. The maximum pressure used for testing was 35psig. The results on the Inficon Micro GC 3000 8m column were not as good as the results from the Inficon Fusion 20m column. The methane peak was not separated from the inert gas peak using even the best combination of pressure and temperature. This column did not have that ability due to the shortened column length and other factors. Although great separation of peaks was not seen using the Micro GC 3000, the water was still visible. For RESOLVE's purposes, water is most important. When on the moon, the mission will last between seven and ten days. Time will be very important and will be used wisely. Using the 8m column, the run time may be decreased which will save time when performing many runs. However, the quality of the data will suffer. The methane on the GC will not be fully separated and will have to rely on the MS for enhanced quantification.

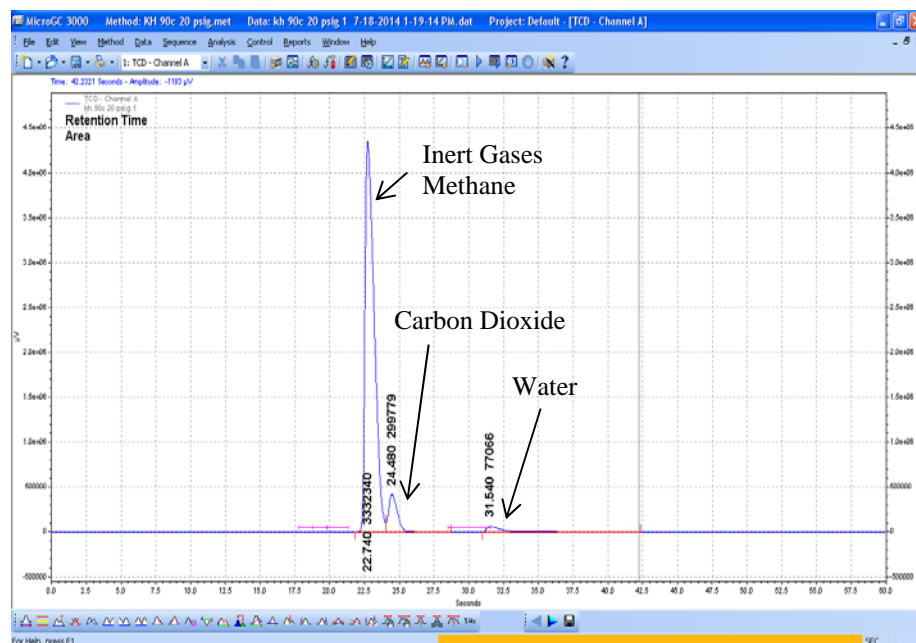


Figure 26. Inficon Micro GC 3000 Chromatogram Data at 90°C and 20psig

As seen in Figure 26, the peak separation is not that good. The carbon dioxide peak is barely separated from the inert gases and the methane peak is nowhere to be seen. When using the Inficon Fusion, 90°C was a very good temperature and separation was good. After further testing it became evident that this GC operated better at lower

temperatures such as 75°C. When using the Inficon Fusion, 75°C was too low because it would drastically increase the retention time. Although methane was not fully separated from the inert gas peak, a slight shoulder was achieved. This was the most separation that could be acquired. Since the column was shorter (8m), the run time for this GC was shorter than with the Inficon Fusion. The retention times were also shorter. The Inficon Micro GC 3000 operated better at a lower pressure because the column was significantly shorter and required less pressure to push the sample through. Although the parameters for the optimal data differ significantly between the two GCs, they are both relative. The column impacts several parameters, including run time. Although the Inficon Fusion had a longer run time and retention times, the benefit was better separation of substances. For example, methane was separated out completely.

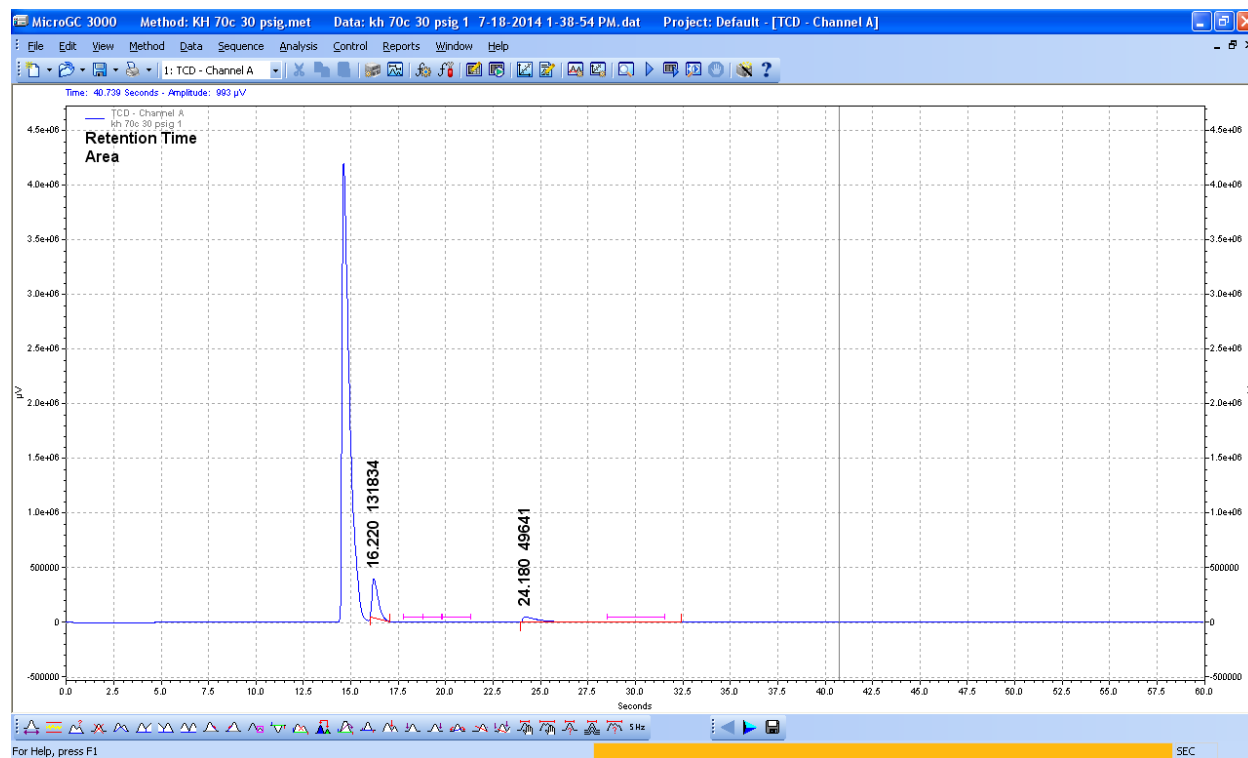


Figure 27. Inficon Micro GC 3000 Chromatogram Data at 70°C and 30psig

V. Conclusion

A Gas Chromatograph is used throughout many laboratories in addition to chemical plants, and performs useful tasks, such as identifying a substance or distinguishing between different substances. In this case, two GCs with different column lengths were used, to evaluate how the column lengths affect the data. For the RESOLVE project, the main objective is to identify and quantify water, which can technically be seen with both instruments. The data is shown in the form of a plot, or chromatogram, showing the various peaks within the sample. In order to draw a conclusion about the data, the graph must be acceptable for analysis. This means that the peaks are distinct and not too broad. Choosing an ideal temperature and pressure may be difficult. A lower temperature improves separation of peaks while a higher pressure decreases the retention time and makes the graphs narrower. Finding a suitable middle ground between temperature and pressure was the goal of this experiment in order to recommend an optimal method for each column length and a preferred column length.

After thorough testing and analysis of the 20m column in the GC Fusion, the data was narrowed down to three temperatures, all performed at one pressure. The final decision resulted from analyzing the graphs at each temperature and then plotting them all on one graph together. The best data observed for the Fusion was at 95°C and 50psig. The peaks were all separated and the retention times were not too large. Also the peaks were not too narrow.

Tests were also performed on a Micro 3000 GC (8m column) to see if the results compare or if one column length stands out over the other. The two GCs required different parameters to achieve the best results due to the column length variation. The GC with the 8m column operates better at a lower temperature and pressure than the GC with the longer column. The best parameter for the Micro GC 3000 was 70°C and 30psig. The benefit of the

machine with the shorter column was a shorter run time, but the separation of peaks was not ideal. When using the Inficon Fusion, there was a sacrifice in run time in return for better quality results. Reference Figure 28 for a breakdown of the pros and cons of each column length.

With the 20m column, much better separation of peaks was seen, but there was a longer run time. With the 8m column, there was only moderate separation of peaks, especially methane, but a shorter run time. Water can be seen with both instruments, which is most important. However, in my opinion, I believe that we should select the 20m column and run GC analysis for slightly longer.

	20 m	8 m
Pros	Better separation of peaks (methane)	Shorter run time
	Separate water peak	Separate water peak
	Sharper peaks	
Cons	Longer run time	Cannot separate methane
		Broader peaks
Best parameter	95C (Column temperature)	70C (Column temperature)
	50 psig (Column pressure)	30 psig (Column pressure)

Figure 28. Comparison Between 20m and 8m Columns

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