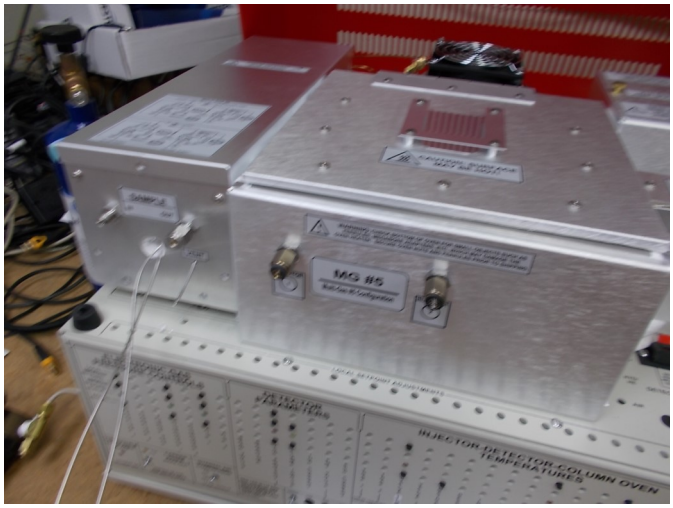
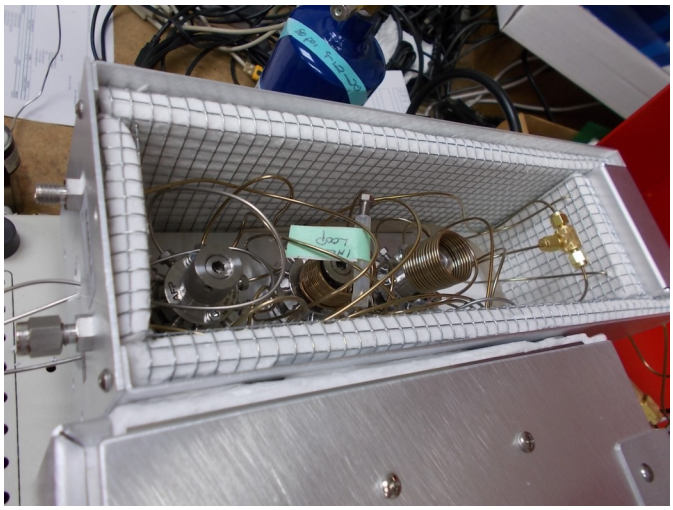
Silicone Tube Loop

May 2021

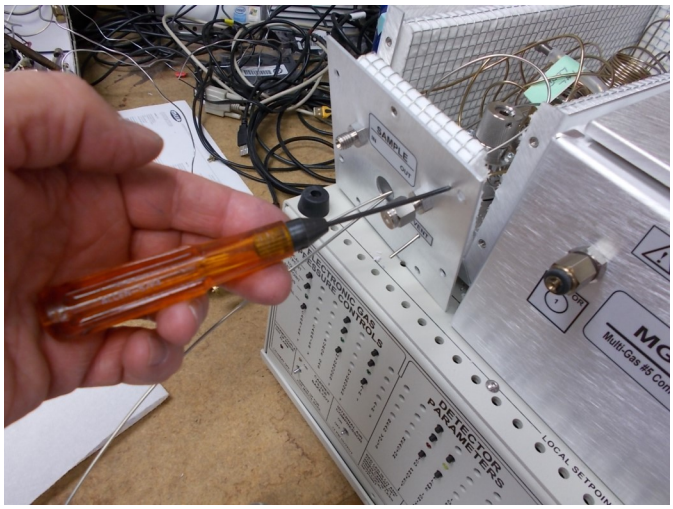
A silicone loop can be added to any 8610C SRI GC as long as there is a place for the 10port valve which is required. A multiple Gas #5 GC configuration is shown in the photo.



In this GC there are three 10 port valves in the heated valve oven.



The front panel oven of the valve oven is easily disconnected by removing six 5/32” hex head screws.



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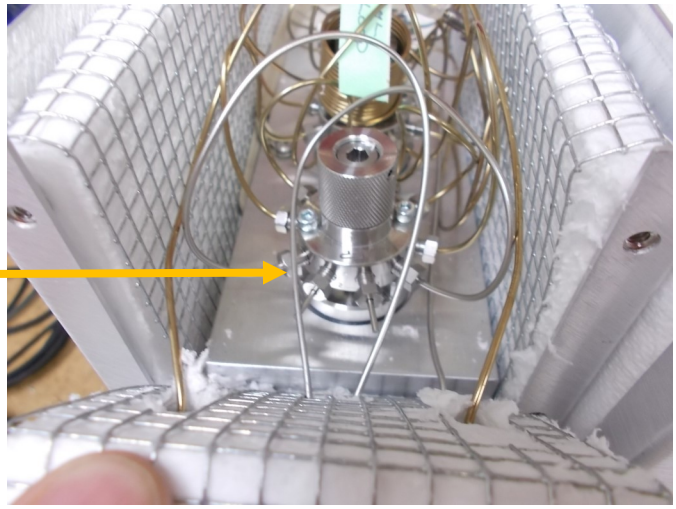
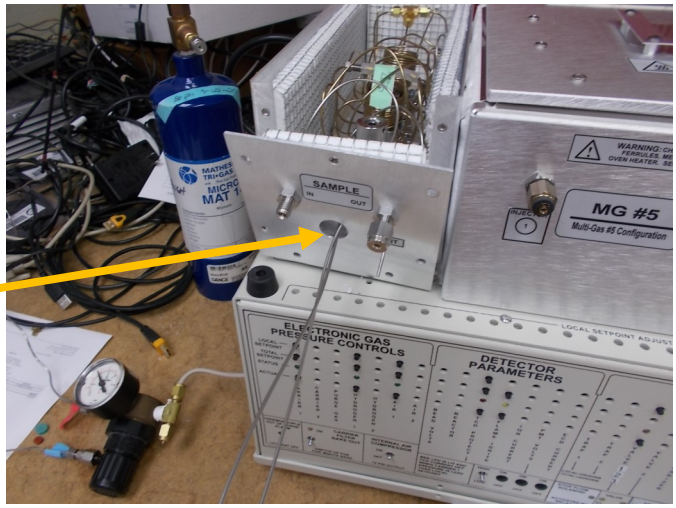
Ideally the 3rd valve will be located at the front where the loop connections (ports 3 and 10) are easiest to get to.

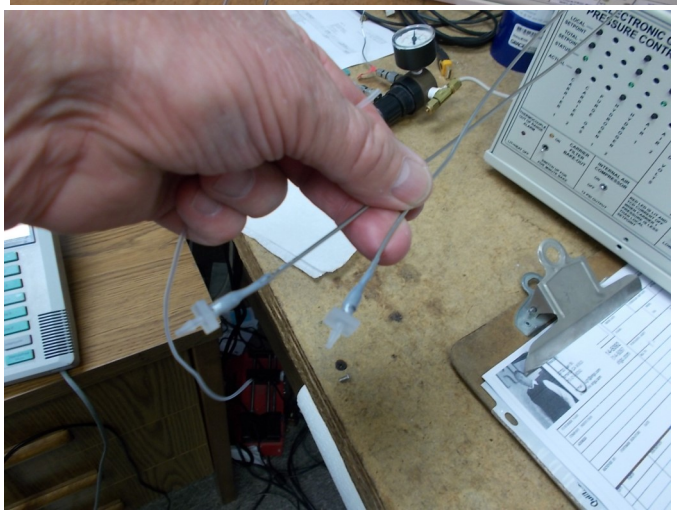


Feed the silicone loop through the front plate and attach to ports 3 and 10 of the valve. If there is a regular loop already attached to those ports you will need to remove it in order to attach the silicone tube loop.



The silicone loop consists of two 1/16” stainless steel tubes with a short section of silicone tubing attached to the ends. The stainless tubing is .010 diameter to minimize the volume of the stainless tubing.

.010” tubing has a volume of about 1.3ul/inch so two 24” long stainless tubes have a total volume of about 64 ul



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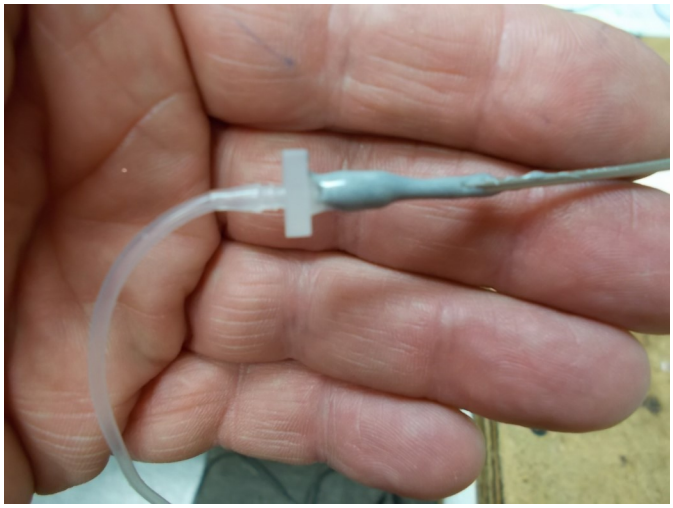
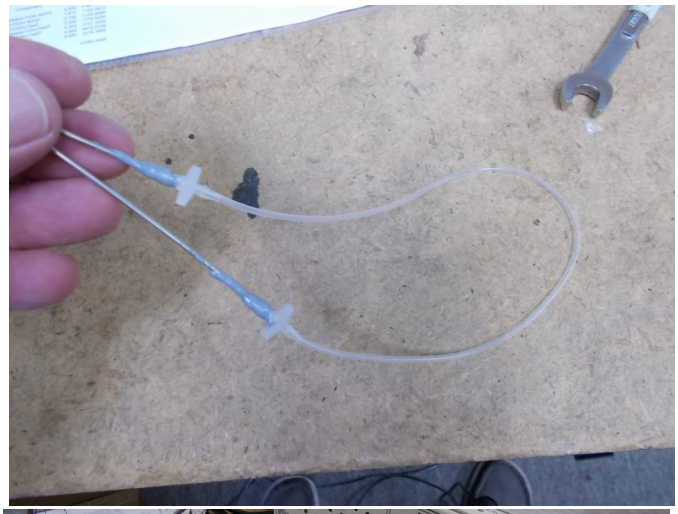
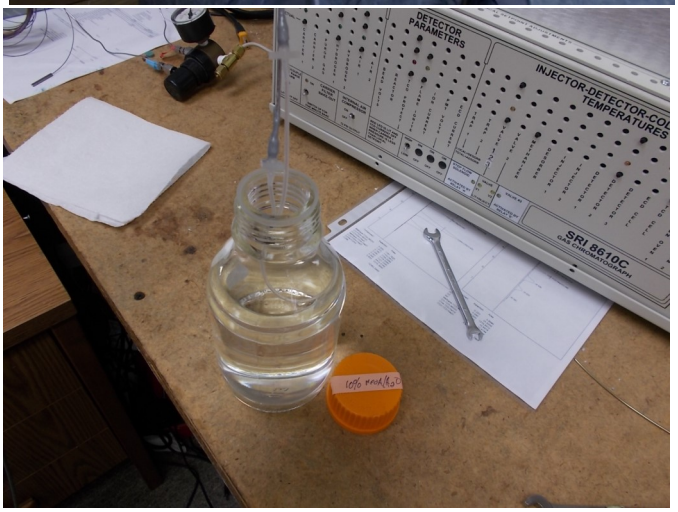
The ends of the stainless tubing are epoxied to a plastic barb fitting. These fittings are commonly availa-ble.in various plastics or metals. Sili-cone tubing is attached to the barb fittings.



The tube shown here is 12 inches long and has an internal diameter of .058 which has a volume of 43ul/ inch for a total of 516 ul. Longer or shorter tubing can be substituted as desired.



The silicone part of the loop is insert-ed into the liquid to be measured.



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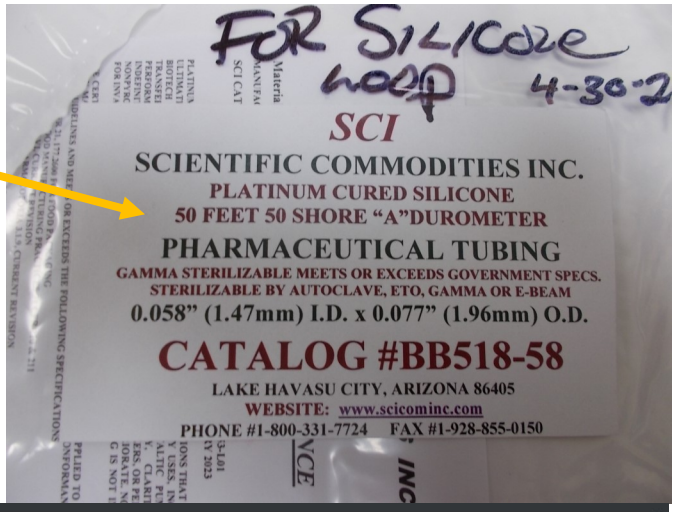
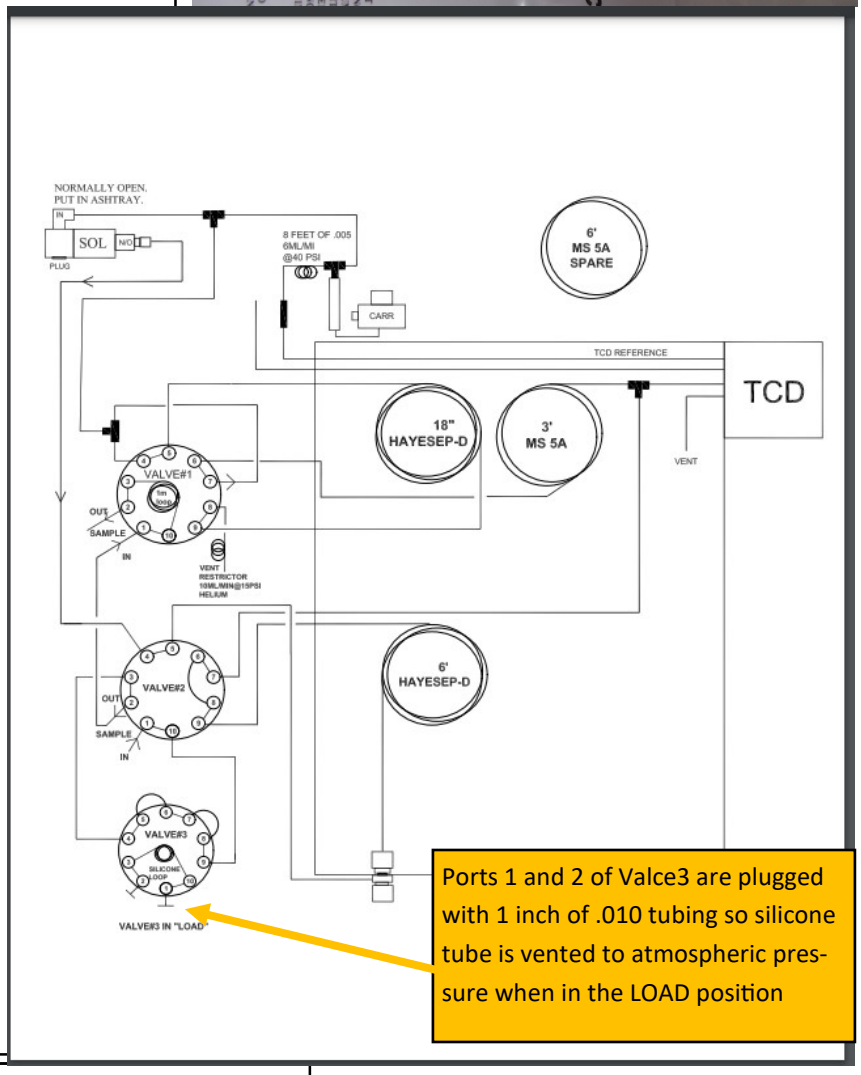
The silicone tubing can be any size or wall thickness, but we have used this which was available on-line.



The valve diagram shows how Valve3 either isolates the loop (no gas flow) or places the loop in the flow path of valve2.

As the silicone loop sits in the liquid many of the compounds in the liquid will permeate through the silicone wall into the interior of the loop. When valve2 rotates and then Valve3 also rotates, whatever is in the silicone loop is pushed into the column.

Valve 3 is rotated back to the Load position (loop isolated) after a few seconds to avoid a detector signal shift which occurs if the carrier gas continues to extract the loop after it is initially emptied.



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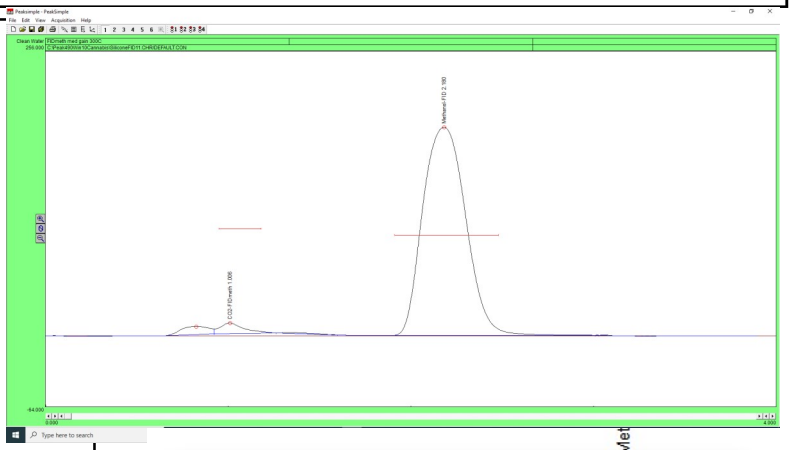
As an example, the silicone tube loop (MultipleGAs#5 GC with TCD and FIDmethanizer) was alternately immersed in 10% methanol/water mix and clean water. This is the chromatogram of the methanol. You can see some small CO2 peak in the beginning also.

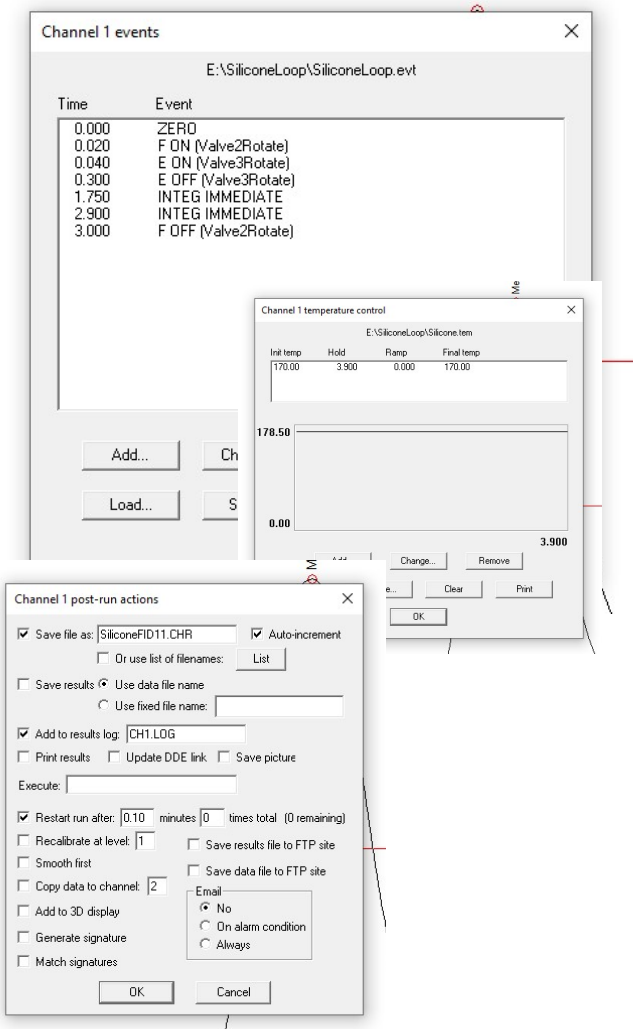


These are the operating conditions.

The most important point is that the analysis has be a repetitive sequence exposing the silicone tube to the sample for a constant amount of time each run.

In this case the runtime was 3.9 minutes and the Postrun Delay before re-starting was .1 minutes. So there was a 4 minute cycle.

The results of many sequential runs were automatically added to the log file which is shown on the next page.



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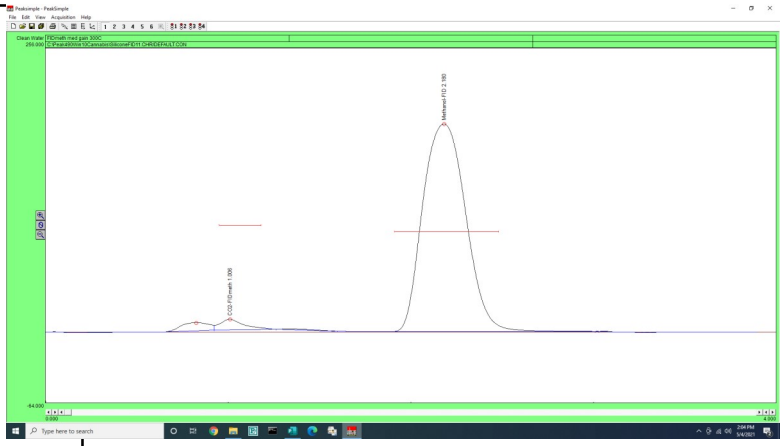
Here is the log file which shows the data from each run as the sample was switched from 10% Methanol/Water to Clean Water and then back again to 10% Methanol/Water.



Note the Relative Standard Deviation (%RSD) for the first 7 runs was 1.27%.

Also note how the silicone tube takes several runs to equilibrate to the new sample. This may take longer with more viscous fluids or other target molecules.

For this reason, you have to sample on some repeated interval so the sample has the same amount of time every time to permeate through the wall of the silicone tubing.

*CH1 - Notepad

File Edit Format View Help

```
Start test by placing siliconeloop in 10% methanol/water
andriy88.CHR    5/4/2021        12:26:55        "Methanol-FID"  2.160   3065.7094
andriy89.CHR    5/4/2021        12:30:55        "Methanol-FID"  2.166   3089.2204
andriy90.CHR    5/4/2021        12:34:56        "Methanol-FID"  2.170   3027.6563
andriy91.CHR    5/4/2021        12:38:56        "Methanol-FID"  2.173   2983.7288
andriy92.CHR    5/4/2021        12:42:56        "Methanol-FID"  2.173   3009.3998
andriy93.CHR    5/4/2021        12:46:56        "Methanol-FID"  2.160   3048.5872
andriy94.CHR    5/4/2021        12:50:56        "Methanol-FID"  2.163   3031.9966
Calculate %Relative Sandard Deviation for above 7 runs =1.27%
Switch to clean water.  Change data file names
SiliconeFID01.chr       5/4/2021        12:54:56        "Methanol-FID"  2.083   249.7427
SiliconeFID02.chr       5/4/2021        12:58:56        "Methanol-FID"  2.086   14.9428
SiliconeFID03.chr       5/4/2021        13:02:56        "Methanol-FID"  2.100   3.4962
SiliconeFID04.chr       5/4/2021        13:06:56        "Methanol-FID"  0.000   0.0000
Notice how it takes several runs to reach zero.
Switch back to 10% methanol in water
SiliconeFID05.chr       5/4/2021        13:10:56        "Methanol-FID"  2.090   1.1803
SiliconeFID06.chr       5/4/2021        13:14:57        "Methanol-FID"  2.203   2772.3704
SiliconeFID07.chr       5/4/2021        13:18:57        "Methanol-FID"  2.183   3092.3008
SiliconeFID08.chr       5/4/2021        13:22:57        "Methanol-FID"  2.183   3067.3421
SiliconeFID09.chr       5/4/2021        13:26:57        "Methanol-FID"  2.180   3048.2049
SiliconeFID10.chr       5/4/2021        13:30:57        "Methanol-FID"  2.183   3125.6104
SiliconeFID11.chr       5/4/2021        13:34:57        "Methanol-FID"  2.180   3055.5135
```

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