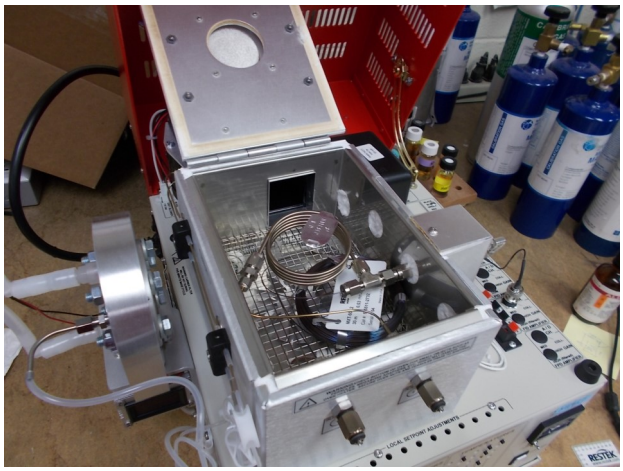
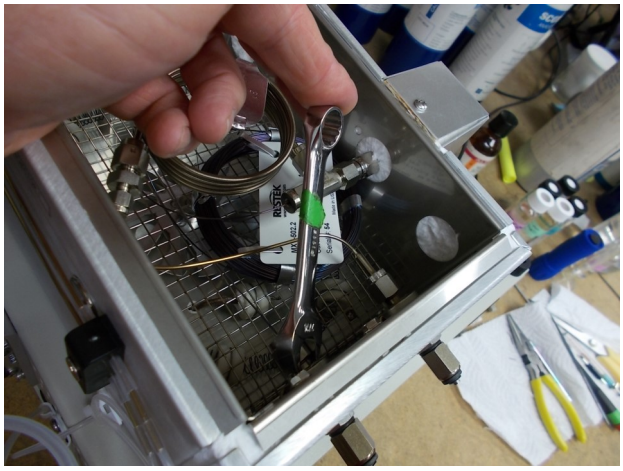
Column Rinsing

April 2021

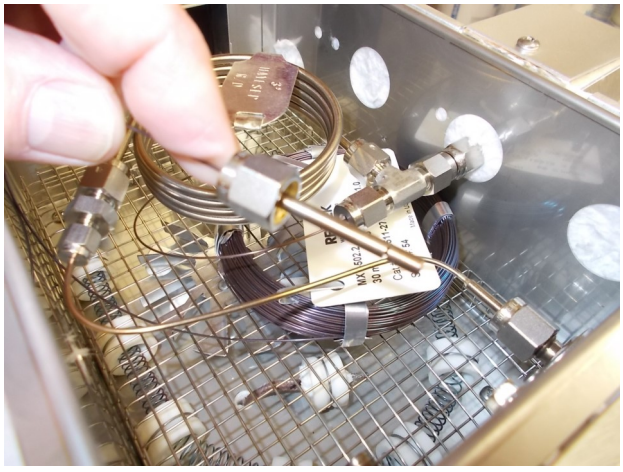
Sometimes it is useful to solvent rinse a column to remove contaminants which can not be removed by heat or time alone. This GC has two columns. Only the capillary column can be rinsed, not the packed column.



Remove the column from the GC by removing the nuts and graphite ferrules at the ends. Keep track of which end is which because its better to rinse from the exit back towards the inlet.



Keep the graphite ferrules away from the solvent because they absorb the solvent like a sponge and take a long while to get clean if saturated with solvent. The injector ferrule is usually soft graphite, while the detector side is often a hard graphite type.



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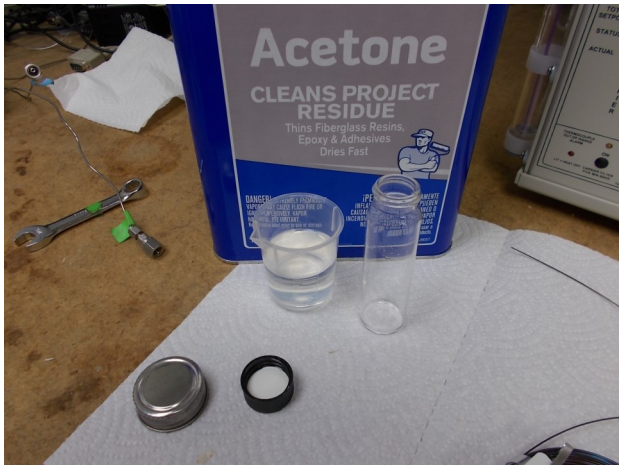
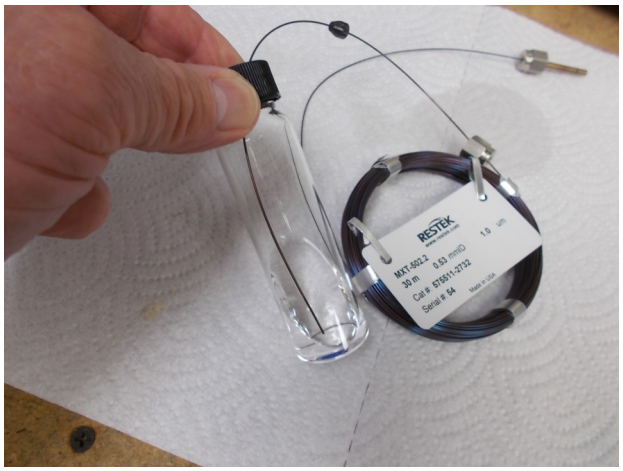
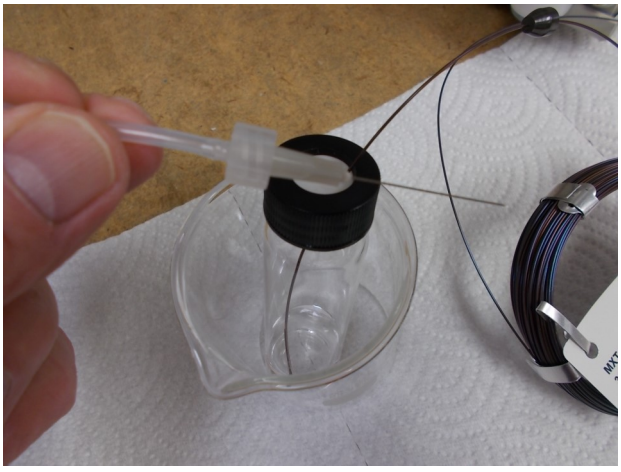
Acetone makes a good solvent, but other solvents like methanol, ethanol etc can also be used. Pick a solvent you suspect will dissolve any contaminant which might be in the column like olive oil, MCT oils, butter etc.

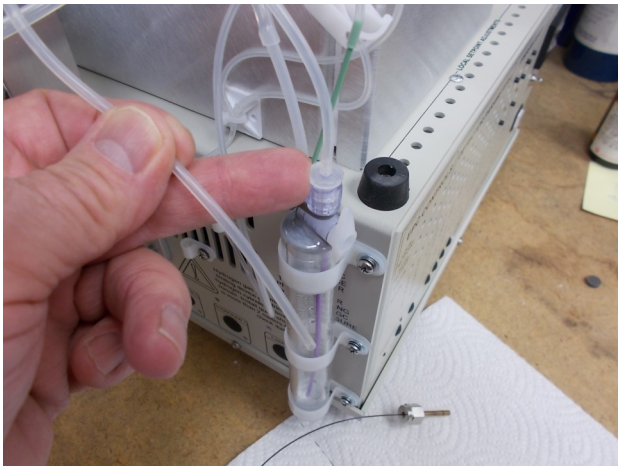


Fill a 40ml extraction vial (septum cap) with 5-10ml of the solvent. Then poke the exit side of the column through the septum and to the bottom of the vial.



Connect a needle to the 1/16” silicone tube drain line of the hydrogen generator and poke that through the septum also. The Luer type needle and fitting are available from SRI or you can buy them locally.





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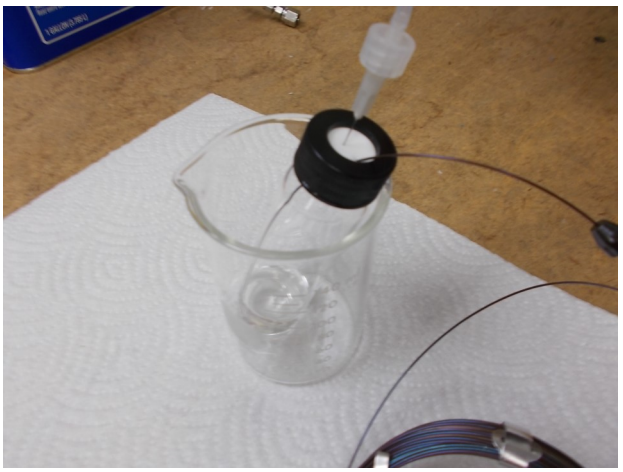
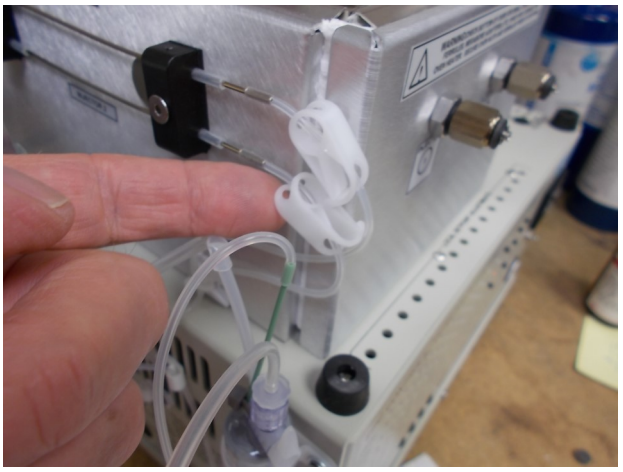
Put the 40ml vial in a beaker or jar to hold it up-right and also to protect against the vial breaking.



Insert the needle through the septum, turn on the GC and rotate the stopcock so the Hydrogen flows out the drain tube. Clip closed both of the white clips so all the gas goes out the drain tube to pressurize the solvent in the vial.



Place the inlet side of the column on a paper towel until the first bit of solvent comes out. This may take a few minutes. The first bit of solvent often comes out dirty looking so you will know you are rinsing something out.

Note that the nut and graphite ferrule have been removed to keep them from getting soaked by the solvent.

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The column is stiff so arrange the 40ml vial and column so the solvent runs into a beaker or jar.

Let the entire 5-10 ml of solvent flush through the column and when the solvent is gone the hydrogen gas will purge most of the solvent out of the column.

You should see hydrogen bubbles once the solvent is mostly purged. There will still be a lot of solvent remaining in the column however, so the FID detector signal will be very high for some minutes after the column is re-installed.

Return the hydrogen generator to the normal operating configuration (white clips set, stopcock closed etc).

Keep the oven temperature at 150 or below for the first few minutes, then run a few blank temperature programs to get rid of any remaining solvent in the column.

Eventually the signal will return to the normal level. Inject the calibration standard several times to see if the rinsing improved the column performance.