Lucidity

miniGC – Analyzing Your GC-FID Test Mix Chromatogram

LUCIDITY

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1. What is the Test Mix

This is the GC-FID Test Mix. It is a standard part from Restek (PN:35108, https://www.restek.com/catalog/view/33563) that can be purchased from their website. It is also included in the Accessory Kit that comes with the miniGC. It is used to QC every miniGC during checkout at Lucidity before it leaves our facility. And it is the primary means for troubleshooting your miniGC.

The Test Mix contains 3 hydrocarbons (C12, C14, and C16) at a concentration of 20ug/mL each in a solution of hexane.

2. How we run the Test Mix

After production, every miniGC goes into checkout before boxing and shipment. During checkout, we run 3 runs of the Test Mix in the miniGC and these runs are capturing in the results for the system so that they can be viewed by the user when the system arrives at their facility. Each run is made using the

GC-FID Method loaded in the system during checkout that remains in the system when it arrives at the user's facility.



The method can also be found on our website (https://luciditysystems.com/products/minigc/instructional-troubleshooting-documents/). A 1uL injection is made using the GC-FID method. The method details are as follows:

Name: GC-FID
Carrier Gas: Helium
Control Type: Constant Pressure
Flow: 2.00mL/min
Split Ratio: 10:1
Inj Temp: 250C
Det Temp: 325C
Temp Stages:

1. *Hold at 40C for 2.0 min*
2. *Ramp at 10C/min to 220C*
3. *Hold at 220C for 0.0 min*

Column: MXT-5 (30m x 0.25mm x 0.25um)

At a 10:1 split rate, 1 uL injections, and injected concentrations of 20ug/mL, the on column amount of each compound is around 2ng, or roughly 2ppm.

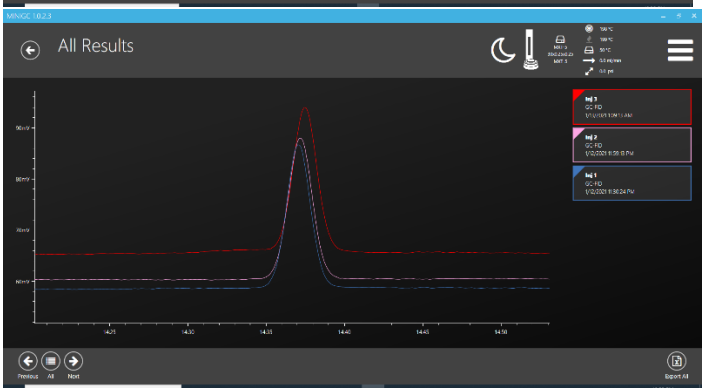
3. What are the passing criteria?

The passing criteria of a GC-FID Test Mix run is as follows:

- Peak height for each of the 3 component peaks (C12, C14, and C16) should be between 25-45 mV (this is measured from the top of the peak to the baseline)
- Peak area run-to-run consistency for the 3 runs should be <2% RSD when injected with the autosampler (<5% RSD for manual injections)
- Retention times should be as follows:
    - C12 11.75 min +/- 0.50 min (between 11:15 and 12:15)
    - C14 14.50 min +/- 0.50 min (between 14:00 and 15:00)
    - C16 17.00 min +/- 0.50 min (between 16:50 and 17:50)
- Retention time run-to-run consistency for the 3 runs should be <2% RSD when injected with the autosampler (<5% RSD for manual injections)
- Solvent peak should begin between 1.50 min and 1.75 min (between 1:30 and 1:45)
- Solvent peak width should be 0.75 min +/- 0.15 min (between 0:39 and 0:51)
- Baseline should be between 30 mV and 75 mV

You can expect some contamination peaks on the first run, but you should still be able to see the 3 component peaks clearly. If there are excessive other peaks these should go away after a couple of runs. If they do not, then it is recommended to run the Bakeout method that is loaded into every system.

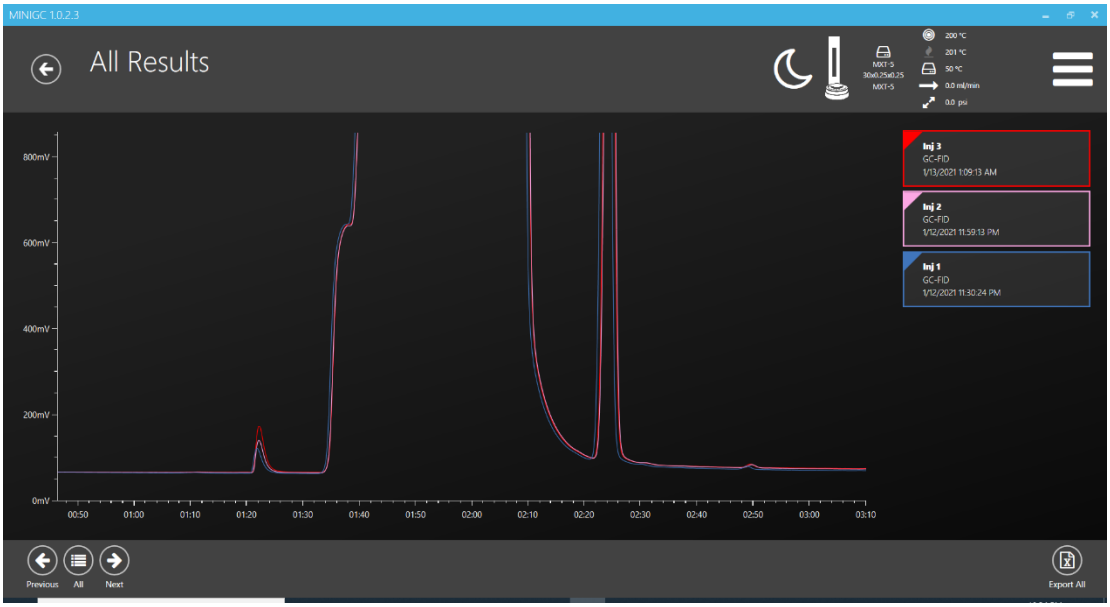
These are some examples of how the Test Mix Chromatogram should look:



4. Troubleshooting the results of the GC-FID Test Mix

Solvent Peak:

The first thing to observe is the solvent peak. Here is an example of what it should look like:



If you don't see a solvent peak (or any other peak), this could mean that the flame in your FID is out. Proceed to the "How To Check if the Ignitor is Working in the miniGC" document to see if this is the issue and if so how to correct it.

If you have a solvent peak, make sure it is the correct width.

If it is too narrow this can indicate a bad injection or a leak. If it is a leak in the system you should see a yellow warning triangle on the top of the interface indicating a low split flow error. This leak could be coming from:

- The injection port – check or replace the liner nut o-ring (the outer o-ring in the top of the injection port), make sure the liner nut is tight (needs only to be slightly tighter than finger tight, use the supplied tool), check or replace the septum, and make sure the septum nut is tight (needs only to be slightly tighter that finger tight).
- The column sealing correctly with the system – remove the column caddie, make sure the column holder is situated correctly on the column caddie, and reinsert the column caddie in the system. If you encounter much resistance when inserting the column caddie, it may be a sign that the unit is cold and needs to warm up before the column will fully seal with the system. Load a method or manually increase the injection port and detector temps to at least 200C and try inserting the column again after a few minutes.

If the solvent peak is too wide this can indicate a leak around the liner o-ring (the inner o-ring in the top of the injection port) or a missing liner. If the liner is missing, or the liner o-ring is not installed properly, the split flow will not operate properly and you will be injecting a much more concentrated sample on to your column, so the peaks will all appear bigger. Check or replace the liner o-ring, make sure the liner is in place, and make sure the liner nut is tight (needs only to be slightly tighter than finger tight, use the supplied tool).

Baseline:

If the baseline is below 30 mV this can indicate that the FID is not lit. Proceed to “How to Check if the Ignitor is Working in the miniGC”.

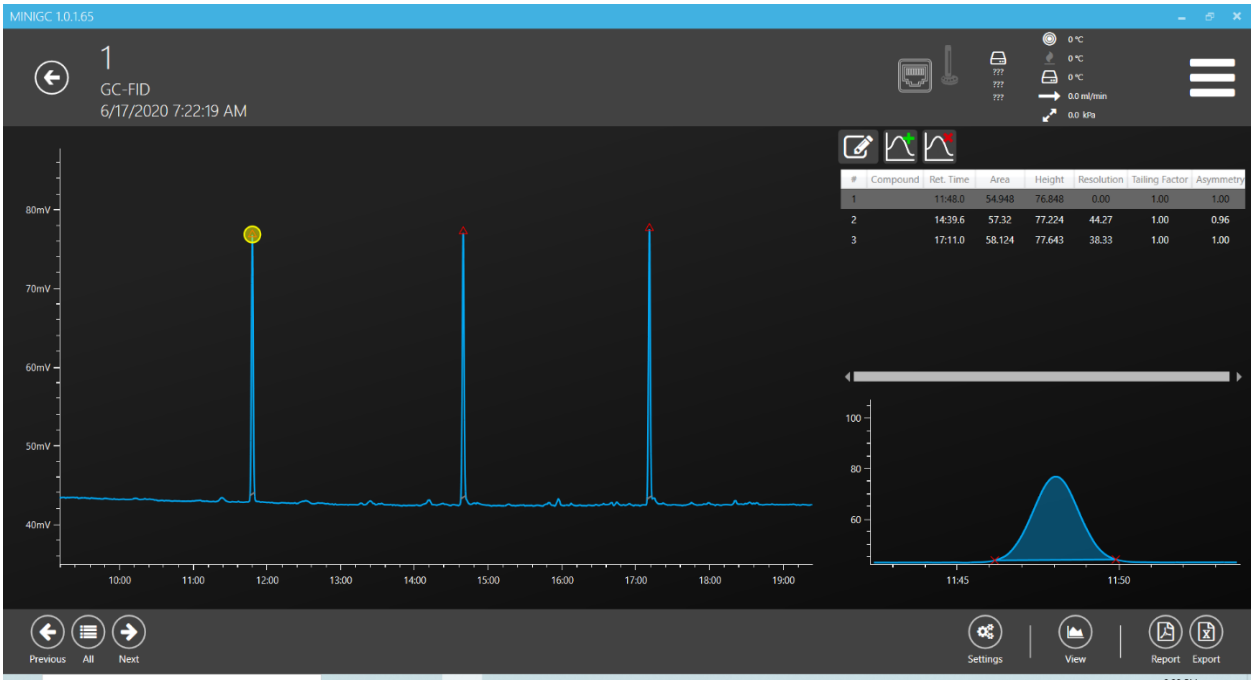
If the baseline is above 75 counts mV, it's possible it is due to excessive contamination. If there is enough contamination on the column, then instead of a lot of extra or unexpected peaks, you will see what appears to be a high baseline as these components come off of the column and into the detector. Run the Bakeout method one or more times (make sure that the bakeout method is compatible with column you are using and does not go to higher temps than are recommended for the column you are running). The standard Bakeout method that comes preloaded in the system is for MXT-5 columns, which have a fairly high max allowable temp. If you are running a column with a lower max allowable temp, then you will need to create a new bakeout method with a lower max temp, otherwise you can destroy your column, and you will in turn see a higher baseline. If the baseline does not begin to come down to the desired level after multiple bakeouts, then there may be another issue.

Retention Times:

Peak Heights:

The next thing to observe is the peak heights of the 3 components (C12, C14, and C16). The peak height of each component should be at least 25 mV and not more than 45 mV.

Here are some examples of what the peaks should look like:



If the peaks are too low in height, this may indicate a bad injection (or possibly a leak). If it is a leak, you should also notice a solvent peak that is too narrow. If the solvent peak is the appropriate width, but the component peaks heights are too low, it is mostly due to a bad injection. Whether you are doing a manual injection or an autosampler injection, check the syringe once you pull up the standard and make sure you have 1uL of liquid in the syringe. If the autosampler is not pulling up 1uL of liquid, make sure

the injection volume is set to 1uL in the method, and make sure that the syringe position in the vial is not too low – if it is it will bottom the needle out on the bottom of the vial and not be able to pull the full injection volume into the syringe. This can be resolved by resetting the “vial” depth position of the autosampler in settings.

If the peaks are too high and the solvent peak is too wide, this would indicate a leaking liner o-ring or missing liner, which means that the split is not accurate and you’re getting more sample than expected on the column and into the detector.

If the peaks are too high and the solvent peak is the proper width, this could indicate that the GC-FID Test Mix needs to be replaced. If the Test Mix is left opened too long or with a septum that has been puncturing a lot, it will evaporate and the 3 hydrocarbon components will be more concentrated in the solution since they evaporate at a slower rate than the solvent.

Repeatability of Retention Times and Peak Areas:

If the peaks do not meet the above criteria for retention time repeatability but all else is fine, there may be an issue with the oven temperature or oven cooling fan. Contact us.

If the peaks do not meet the above criteria for area repeatability but all else is fine it may be a case of intermittently bad injections or a leaky liner o-ring. Replace the liner o-ring and monitor the syringe on the injections to make sure the syringe is filled with liquid up to the 1uL mark for every injection you are comparing.

Here is how to calculate the %RSD for the peak retention times and peak areas:

1) Convert retention times into minutes from minutes:seconds. For example 12:15 is 12.25 minutes.
2) Calculate the average retention time and area for each of the 3 peaks for 3 different runs
3) Calculate the standard deviation of the retention time and area of each of the 3 peaks for these runs
4) %RSD = 100 * (Standard Deviation / Average)

For the 3 runs shown in these examples the %RSD values would be:

Ret Time

	C12	C14	C16
Inj 1	11.73	14.62	17.14
Inj 2	11.73	14.62	17.14
Inj 3	11.74	14.62	17.15
AVG	**11.73**	**14.62**	**17.14**
STDEV	**0.01**	**0.00**	**0.01**
%RSD	**0.0%**	**0.0%**	**0.0%**

Area

	C12	C14	C16
Inj 1	42.4	46.4	47.2
Inj 2	42.1	45.8	46.2
Inj 3	42.5	45.3	50.1
AVG	**42.3**	**45.8**	**47.8**
STDEV	**0.21**	**0.55**	**2.03**
%RSD	**0.5%**	**1.2%**	**4.2%**

These 3 injections were done manually (rather than with the autosampler); therefore, this system would pass the performance test since the %RSD values are all less than 5%. If these injections were done using an autosampler these %RSD values would all need to be <2%. To do some further analysis on these 3 runs, the C16 peak in the 3rd injection is the only one that keeps all the peak areas from being

within 2%, and if you observe this peak, you will notice that it appears there is a slight amount of contamination in the run that appears just on the back end of this peak, possibly slightly affecting its peak area. Keep in mind these compounds (C12, C14, and C16) are only 2 ng on column (around 2 ppm), so even very small amounts of contamination can affect these peaks.