95-0013 User Guide- TD-GC-Lonestar: GC column removal and installation

Contents

1 Introduction

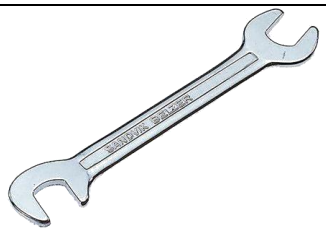
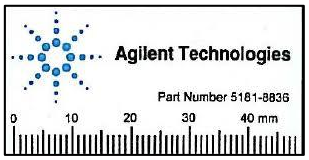
This document explains how to remove and install a HP-5 GC capillary column in the Thermo Trace 1310 GC oven. It includes guidelines on essential column installation checks, such as leak checks and column blanks.

2 Definitions

Abbreviation/Term	Definition
TD	Thermal Desorption. The TD-GC-LNS system has the Markes UNITY-xr TD Unit integrated.
UNITY-xr	Thermal desorption platform with a sorbent tube oven integrated in where sample tubes can be loaded and desorbed for VOCs injection onto the GC column.
VOC	Volatile Organic Compound
GC	Gas Chromatographer. The TD-GC-LNS system has the GC Thermo Trace 1310 integrated.
LNS	Lonestar System – its FAIMS chips enables to detect chemical species in gaseous state based on its characteristic ion mobility speed under an asymmetric electric field.
FAIMS	Field Asymmetric Ion Mobility Spectrometry
PPE	Personal Protective Equipment
HP-5	GC column installed in the Thermo Trace 1310
He	Helium, acts as carrier gas in the TD-GC-LNS system
N2	Nitrogen, acts as purge gas in the TD-GC-LNS system.
OML	Owlstone Medical Ltd

3 Tools and supplies.

Quantity	Item	Use	Part number	Picture
1	HP-5 GC column (30m x 0.32mm x 0.25µm)	To provide chromatographic separation (other GC column should be installed depending on the analysis).	Agilent part number 19091J-413	

2	Capillary column silicone caps.	To cap and protect both ends of the GC column, preventing moisture and air from entering the column during storage.	Restek Cat.# 22858 **Note:** columns are manufactured capped with similar silicone caps. Please, keep them.	
2	BAHCO spanner 1931M-5 (5mm x 5mm spanner)	To (un)tighten the Siltek MXT VALCO union connecting the TD transfer line and the GC column.	RS Stock No. 305-4246	
1	Siltek MXT connector kit for 0.32mm ID columns	MXT VALCO union used to provide a leak-free connection between the TD transfer line and the GC column. Each new kit includes MXT union; 1/32-inch nuts (2); VALCO one-piece union body, and fused silica adaptors (2)	Restek Cat.# 21385	
2	1/32-Inch VALCO Adaptor Ferrules	Ferrules inserted in between each male nut and the MXT VALCO union body. The GC column goes through it.	Restek Cat.# 20140	
2	3/8" x 7/16" open end spanner	The 7/16'' end is required to o (un)tighten the 1/8'' Swagelok nut connecting the GC column with the LNS transfer line.	Any spanners with a 7/16'' end can be used.	
1	Vespel Graphite Ferrules 1/8-0.5mm	It goes inside the 1/8' Swagelok nut located to the right side of the GC oven (connecting with the GC-LNS transfer line)'. It seals the connection, protecting from gas leakage. Suitable for 0.32mm columns.	Restek CAT# 20255	
1	Ceramic wafer	To cut the GC column.	Agilent Part Number:5181-8836 (or any other suitable for GC column trimming)	

1	9x Magnifier	To inspect and check the column cut edge is clean and without sharp ends.	RS Stock No. 544-055	
1	Ruler	To measure 21cm of GC column at the end connecting with the LNS transfer line	Part number#KF01109 (Any 30cm long ruler can be used).	
1	High-temperature string (not essential)	It can be used to restring capillary columns and hold the column in the GC oven rack. Capable of withstanding temperatures to 400 °C.	10m pack – Restek Cat.# 20109	
1	Gas leak detector	To detect He leaks from unsealed/loose fittings which require further tightening	Agilent G3388B leak detector (other leak detectors can be used)	
1	Liquid Tipp-Ex	To mark the surface of the column at 21cm of length.	Any Tipp-Ex can be used. Alternatively, the column can be marked with a pen marker or a thermostable GC septum.	
3 minimum	Swagelok 316 Stainless Steel blanking nut for 1/8''	To cap the LNS and seal the female nut used to insert and connect the GC column to the LNS transfer line. This is required when performing an LNS pressure test (see section 5.3)	Swagelok Part No. SS-200-P	

4 TD-GC-LNS system diagram

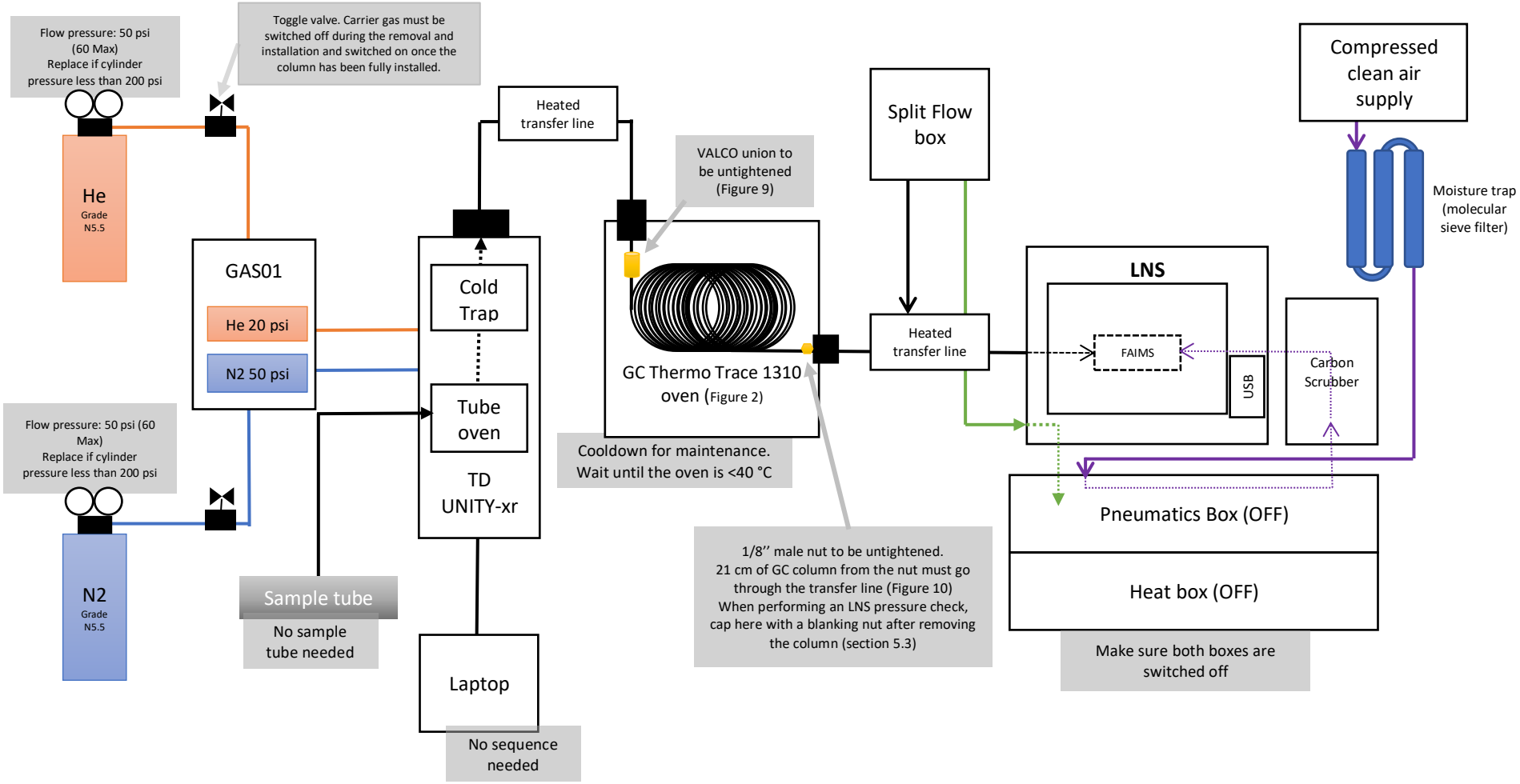


Figure 1. TD-GC-LNS diagram, with key steps to follow when removing a GC column.

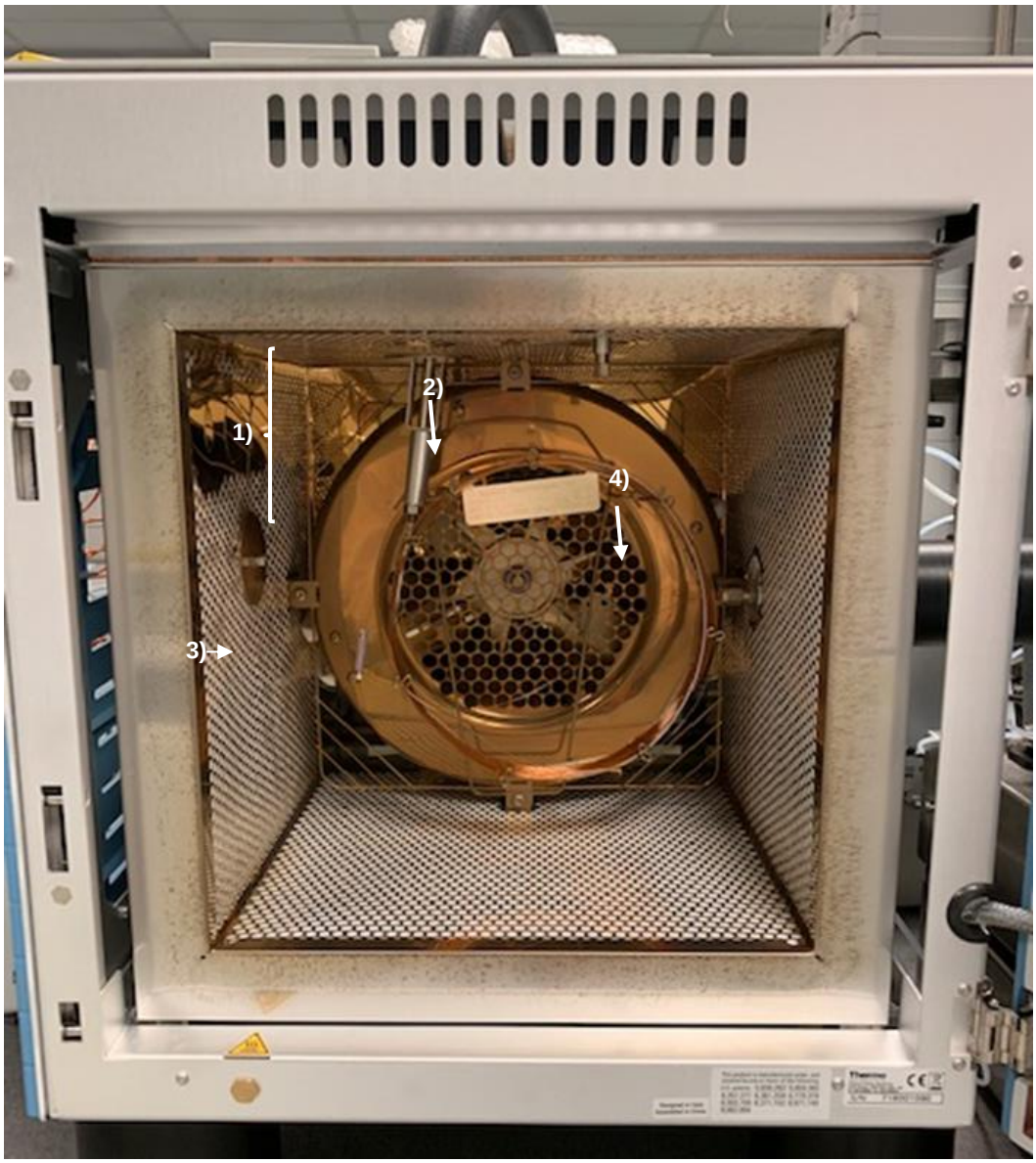


Figure 2. GC Thermo Trace 1310 oven.1) TD Transfer line inlet, 2) HP-5 GC column held in the oven rack, 3) Siltek MXT VALCO union connecting the TD transfer line to the GC column (GC column end needs to be loosened), 4) GC column outlet. 21 cm of column goes through the 1/8'' nut and connect with the LNS transfer line (needs to be loosen).

5 Procedure

PPE (lab coats, safety specs and gloves) must always be worn when replacing the GC column.

Note: The photos presented in the following sections have been taken without gloves and using defective parts in order to show as clearly as possible the procedure explained.

5.1 System preparation for GC column replacement.

5.1.1 The GC oven will require to be cool down if its temperature exceeds 40°C. To do so, set the GC oven to cool down by pressing "*Maintenance*" > "*Cool for Maintenance*" from the interface main screen (Figure 3).

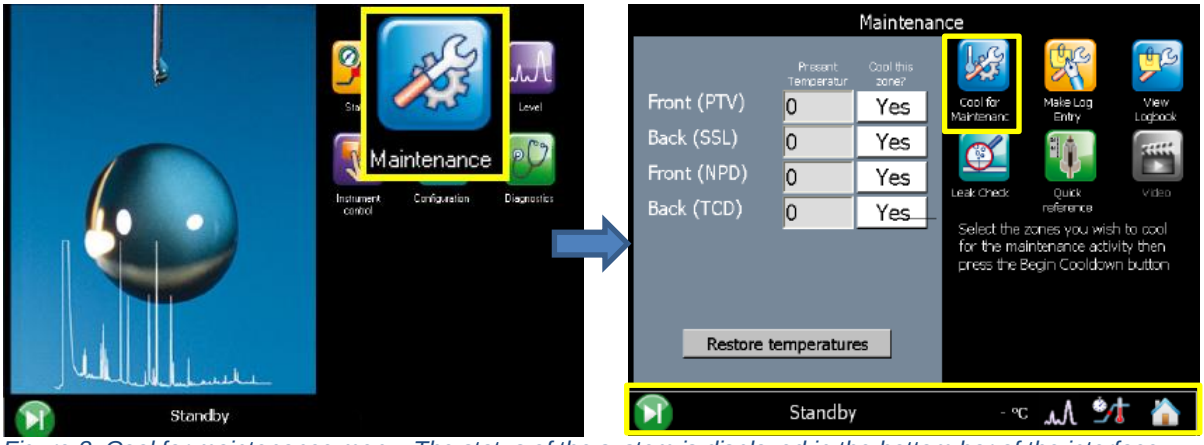


Figure 3. Cool for maintenance menu. The status of the system is displayed in the bottom bar of the interface.

5.1.2 The panel shown in Figure 4 will appear to the left of the interface. Select yes on all the entries in the "*Cool this zone?*" column and then press "*Begin Cooldown*". Due to the TD-GC-LNS settings, only the "*Front (SSL)*" should be available.

The status of the system will then change from "*Standby*" /" *Ready to inject*" to "*Cooling for Maintenance*". Please, wait until the temperature is around 40 °C before opening the GC oven.

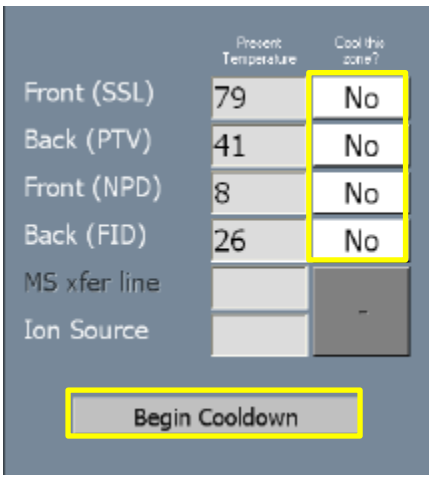


Figure 4. How to start the GC oven cooling down. Select "Yes" on the "Cool this zone?" column and then press "Begin Cooldown". The message "Cooling for maintenance" will appear in the status bar at the bottom of the interface. This option will not work if the oven it is already at around 40°C.

5.1.3 Close the air supply from the ATLAS pneumatic box, by turning the “air supply” valve as shown in Figure 5. The pressure regulator dial should drop to zero. DO NOT modify the flow from the pressure regulator.

Figure 5. ATLAS pneumatic control box diagram. 1) Air supply valve, 2) Pressure Regulator, 4) Pressure dial.

5.1.4 Switch off the heat box. The “TEMP 1 LIMIT” and “TEMP 1 CONTROL” green lights should be off. The light above the switch is illuminate if the ATLAS heat box has power, as shown in Figure 6.

Figure 6. ATLAS heat box. The light panel displayed is how it should look like when the heat box is switched off.

5.1.5 Switch off the carrier gas line. This can be done either by closing the toggle valve installed next to the primary gas regulator or by closing the GAS01 valve.

5.1.5.1 To close the carrier gas line from the primary gas regulator, turn the valve 90 degrees, so that the valve is placed perpendicularly to the gas line (Figure 7).

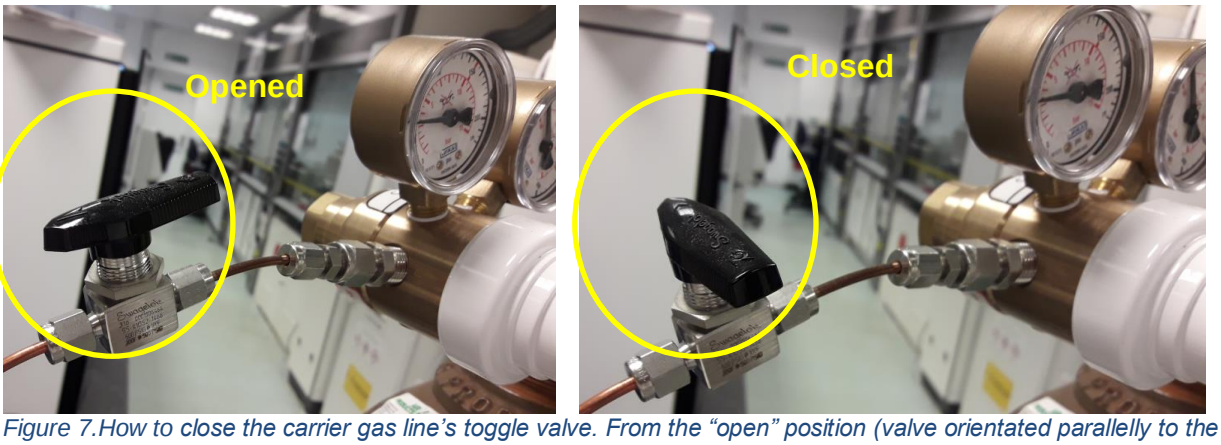


Figure 7.How to close the carrier gas line’s toggle valve. From the “open” position (valve orientated parallelly to the gas line), turn it 90 degrees (valve orientated perpendicularly to the gas line) to close the carrier gas.

5.1.5.2 If there is no toggle valve installed, it is possible to switch of the He flow by switching off the carrier gas regulator at the GAS01 (Figure 8).

Figure 8. GAS01 secondary regulator. Use the carrier gas switch to close the line.

⚠	***Do not remove and/or install a GC column with the carrier gas opened. Ensure the gas line is closed.***

5.2 Column removal.

5.2.1 Use the two Bahco spanners (5mm x 5mm) to untighten (anticlockwise) the male nut connecting the GC column to the MXT VALCO union connector. It is not required untighten the male nut on the transfer line side unless the transfer line needs to be replaced or leak is suspected.

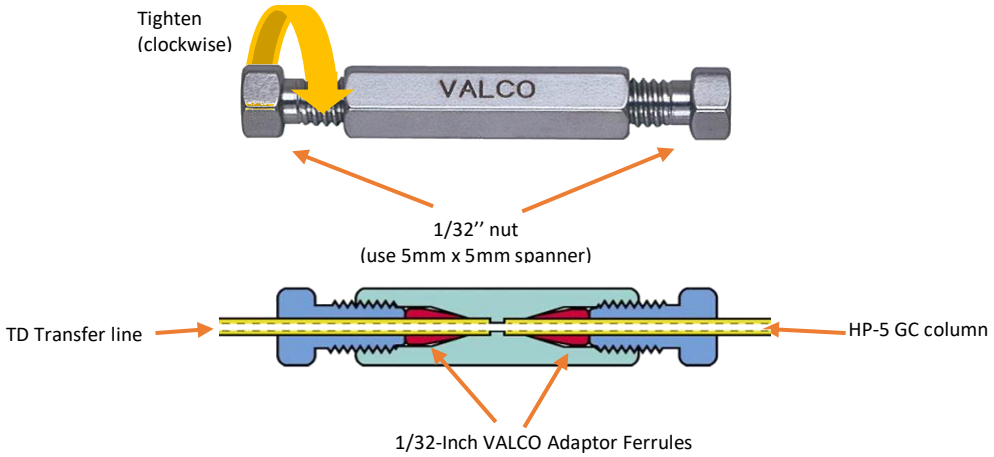


Figure 9. Diagram showing the different parts of the Siltek MXT connector kit. The 1/32'' nuts are tightened clockwise and loosened anticlockwise.

5.2.2 Finger-loose the male nut and remove the 1/32-Inch VALCO adaptor ferrule that goes inside. **Keep the 1/32'' male nut as it will be reused**. The adaptor can be re-used as long as the same column model is installed, and the union has not been excessively overtightened.

5.2.3 Using the 7/16'' spanners, untighten the 1/8'' Swagelok nut connecting the GC-LNS transfer line, located on the right side of the GC oven (Figure 10). Unscrew the male 1/8'' Swagelok nut. Fingers can be used if the nut is not hot.

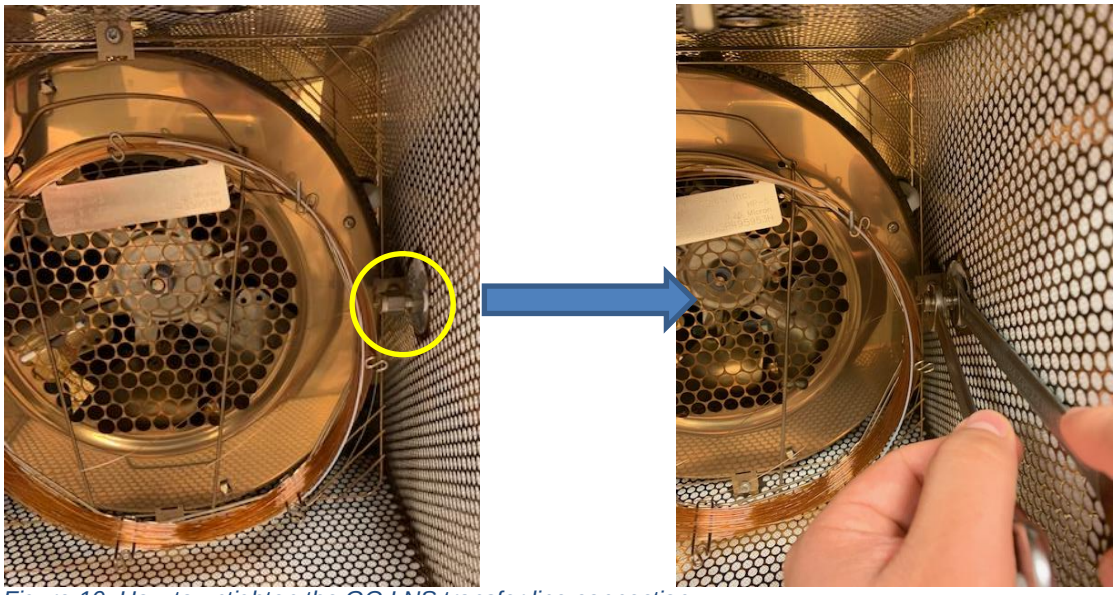


Figure 10. How to untighten the GC-LNS transfer line connection.

5.2.4 Remove the 21cm of GC column that goes through the GC-LNS transfer line (Figure 11).

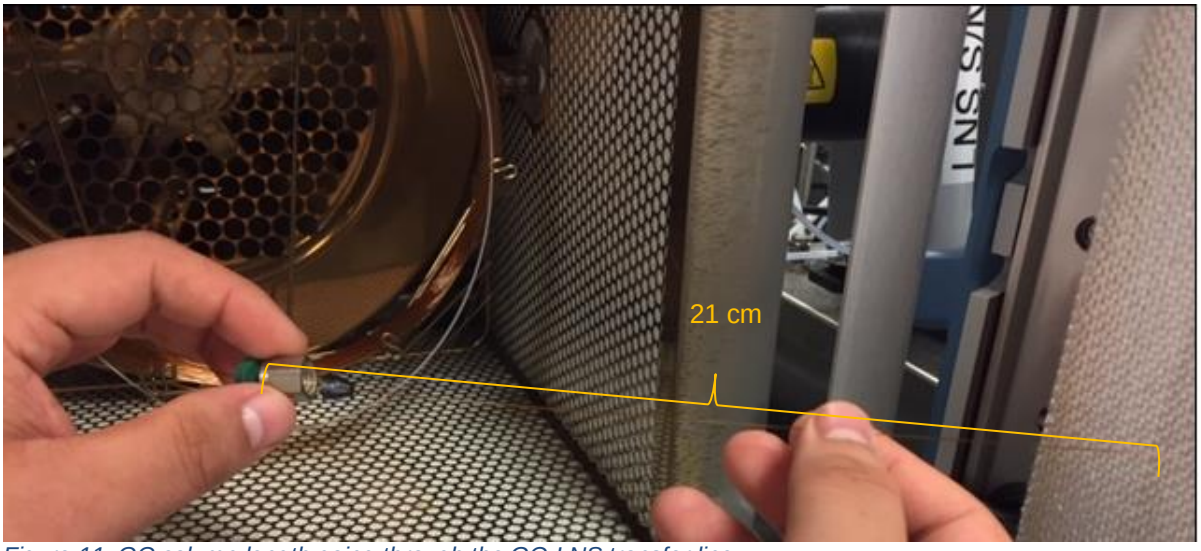


Figure 11. GC column length going through the GC-LNS transfer line.

5.2.5 Remove the Vespel Graphite ferrule and the 1/8'' Swagelok nut. ***Keep the 1/8'' nut as it will be used when installing the new column***. Unlike the 1/32-Inch VALCO adaptor ferrule, a new Vespel Graphite ferrule must be used whenever a column is replaced.

5.2.6 Remove the old column from the column rack. It is recommended to cap both ends with silicone caps, especially when the column needs to be shipped to OML or stored for long periods. The silicone caps which the column was manufactured and shipped with can be used. Alternately, these caps can be purchased (see section 3).

5.3 LNS Pressure test.

5.3.1 Before connecting the GC column to the LNS system, it is recommended to perform a pressure test on the TD. This is to demonstrate the LNS is leak free, so that any leak detected on the GC-LNS transfer line at the GC side can be attributed to loosen fittings, bad column trimming techniques or column inserted inappropriately.

The GC column must be disconnected from the GC-LNS inlet. During this pressure test, the LNS system plots the pressure versus time showing the pressure test monitoring for 10 minutes and fails if the pressure drop is more than 0.06 bar (60 mbar).

5.3.2 Pressurise the LNS by re-opening, temporarily, the pneumatic ATLAS box (see Figure 5)

5.3.3 From the LNS interface, go to "*Plugins*" >" Pressure *test*".

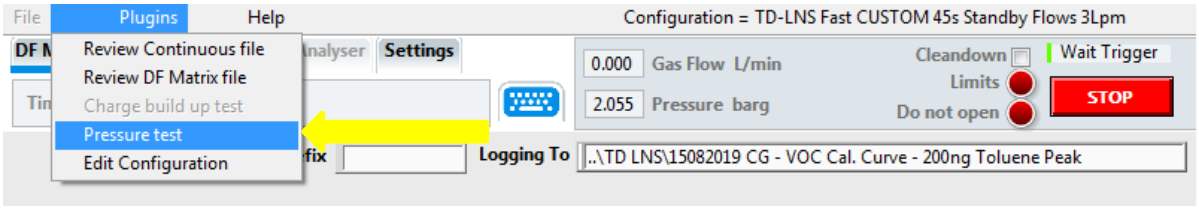


Figure 12. LNS pressure test function.

5.3.4 The LNS will display the screen shown on Figure 13, which provides the guidelines needed to complete the pressure test (these will be explained below).

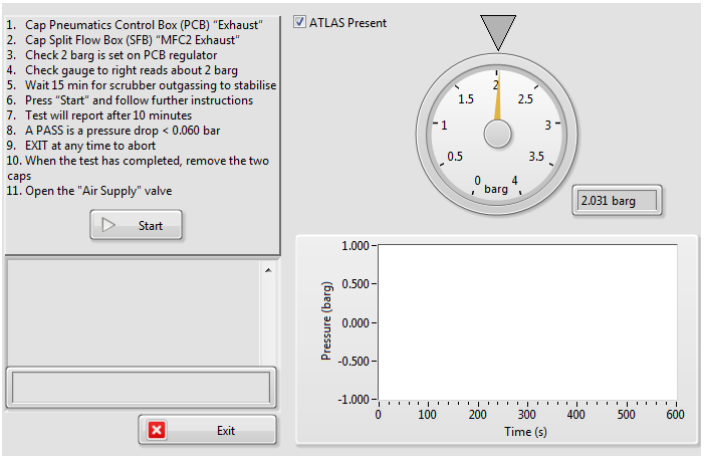


Figure 13. How to run a pressure test on the LNS system.

5.3.5 Cap the ATLAS pneumatic box exhaust located on the back-left corner of the box.

Figure 14. 1/8" Swagelok blanking nut capping the ATLAS pneumatic box exhaust.

5.3.6 Cap the ATLAS Split Flow Box exhaust located at the back of the Split Flow Box.

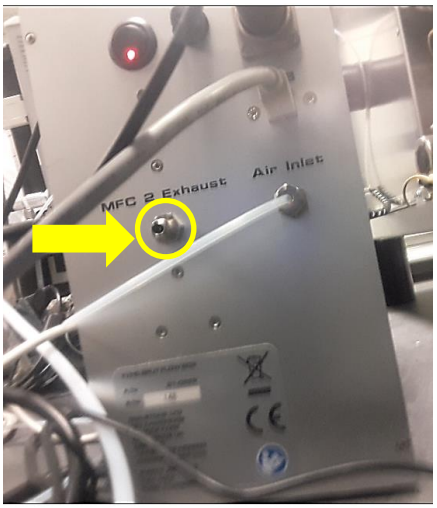


Figure 15. 1/8" Swagelok blanking nut capping the ATLAS Split Flow Box exhaust.

5.3.7 Cap the “MFC1” out and “MFC3 out”, located at the front of the Split Flow Box, if they are not connected to the TD-GC-LNS system. The same applies to any disconnected line coming from the bottle.

Figure 16. Split Flow Box front view. MFC1 out and MFC3 out required to be capped with a 1/8" blanking nut if they are not connected to the TD-GC-LNS system.

5.3.8 Cap with a 1/8'' Swagelok blanking nut the column inlet connecting the GC-LNS transfer line, located on the right side of the GC oven (same connection from where 21cm of column has been removed, as shown in Figure 10).

5.3.9 Check the pressure shown on the LNS Pressure Test screen is stable and around 2 barg and wait 15 minutes as per the instructions the instrument displays.

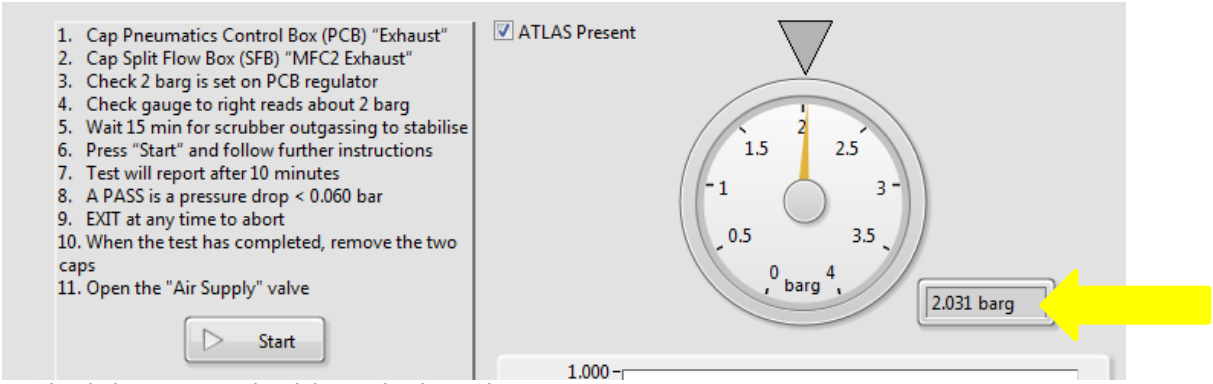


Figure 17. Check the gauge to the right reads about 2barg.

5.3.10 Press the "Start" button and let the pressure to stabilize for 30 seconds. A box indicating to close the pneumatic box air supply will pop up. Follow the instructions, close the air supply from the ATLAS pneumatic box and then press "OK".

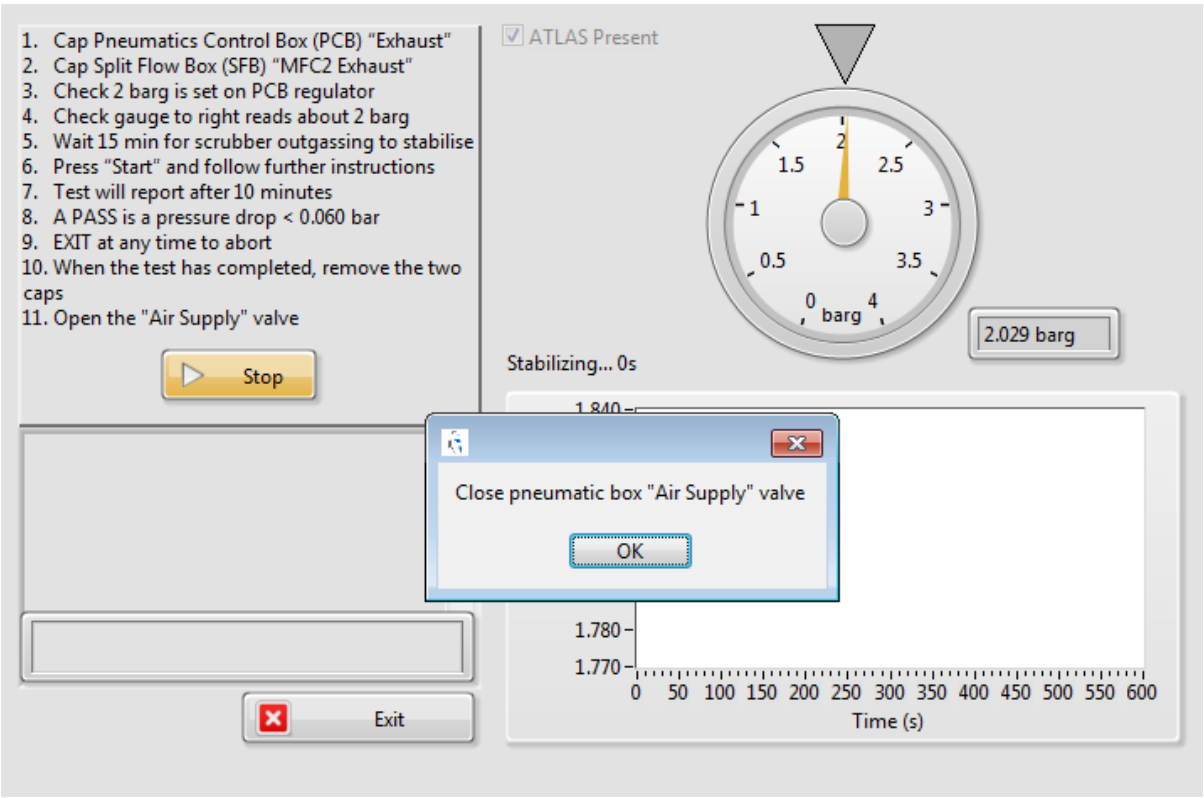


Figure 18. Close the pneumatic box after the pressure has stabilized. Once done, press "OK".

5.3.11 The LNS Pressure Test will then begin and the instrument will start plotting the pressure readings over time.

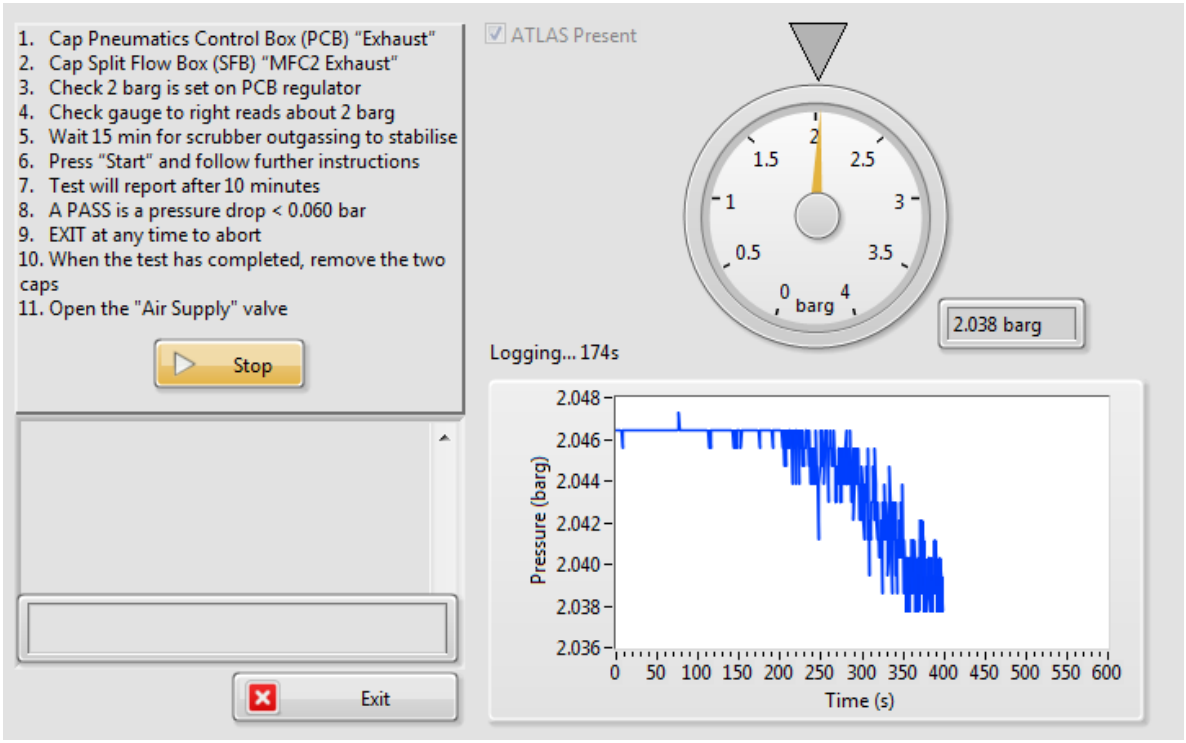


Figure 19. LNS pressure test in progress.

5.3.12 The test will be passed if the pressure drop is less than 0.060bar. Press “OK” to finish exit the test screen.

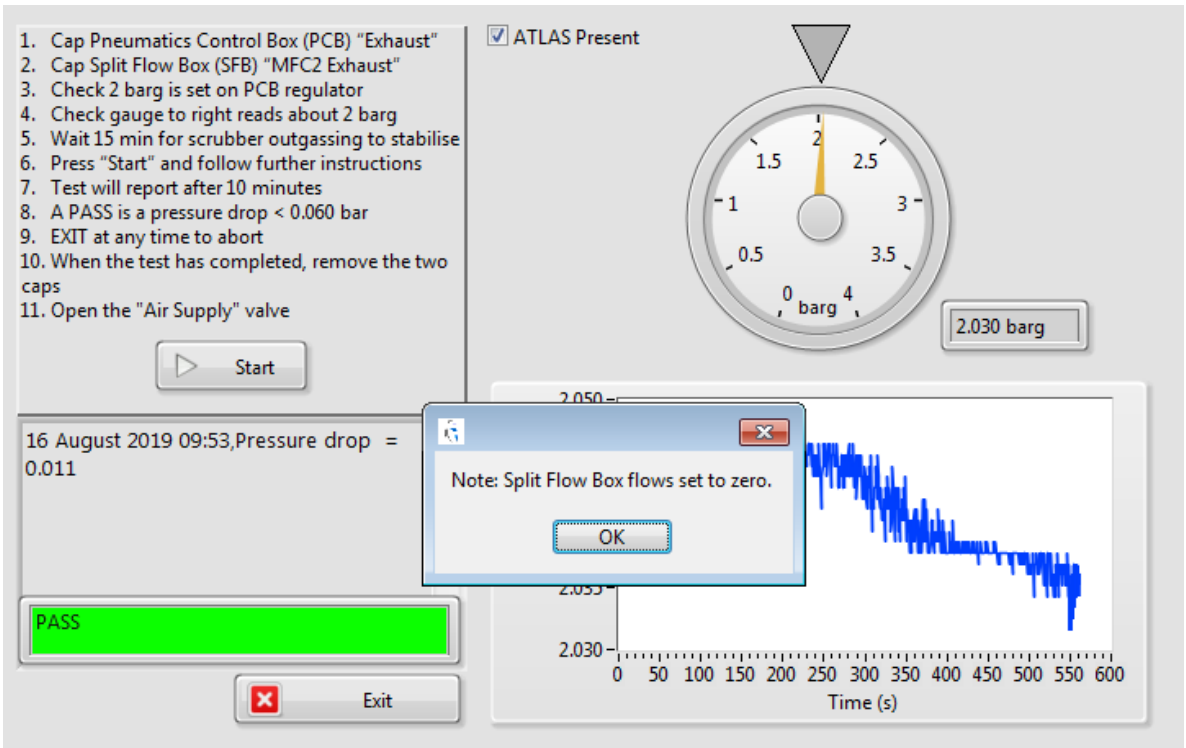


Figure 20. LNS Pressure test – Pass result screen.

5.3.13 Remove the blanking caps. Leave the ATLAS pneumatic box closed during the installation of the column.

NOTE: to restore the LNS gas flow (L/min) and pressure (barg) parameters on the LNS, just press STOP and then START.

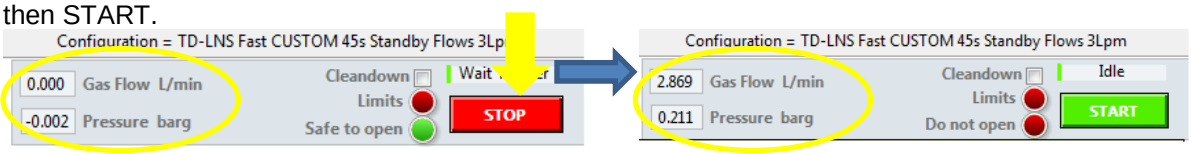


Figure 21. How to restore gas flow and pressure parameters on the LNS.

5.3.14 If the LNS pressure test fails, ensure that the blanking caps are not loose. Check systematically all the Swagelok fittings and gently tighten them if required. Repeat the LNS pressure test. If the test continues failing, request technical support. For additional support, please visit www.owlstonemedical.com and click on Support or send an email to support@owlstone.co.uk.

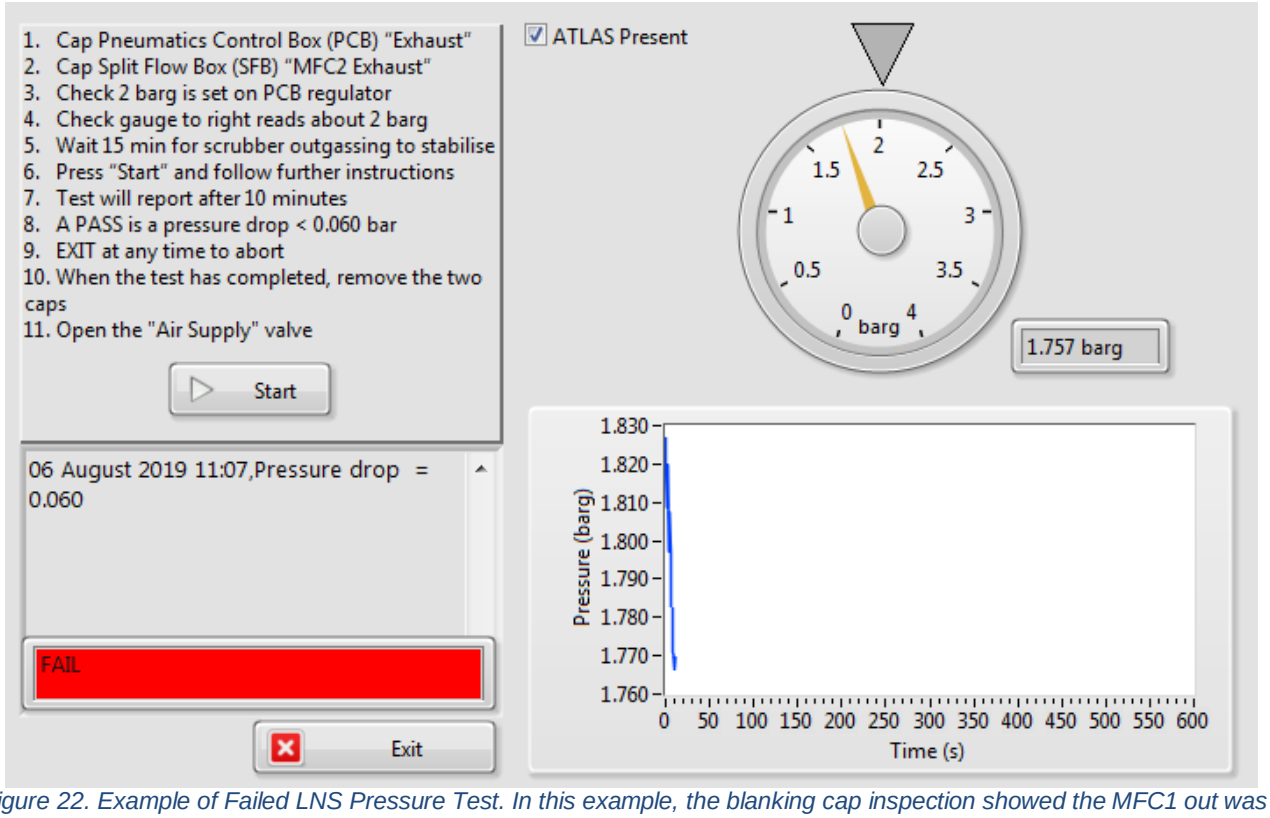


Figure 22. Example of Failed LNS Pressure Test. In this example, the blanking cap inspection showed the MFC1 out was not capped. The test passed after resolving it.

5.4 How to connect a new column to the TD-GC transfer line.

5.4.1 Unpack the new HP-5 column and place it in the GC oven column rack.

5.4.2 Unwind one column end. This end will connect with the TD transfer line through the VALCO union.

5.4.3 Insert the VALCO male nut kept from the step described in section 5.2.2, followed by a 1/32-Inch VALCO adaptor ferrule. Leave around 6 cm of GC column from the end of the ferrule (Figure 23).

Thermostable GC septa can be used to provide a support where the nuts and ferrules can rest. Make sure to hold the column as close as possible to the end when perforating the septa.

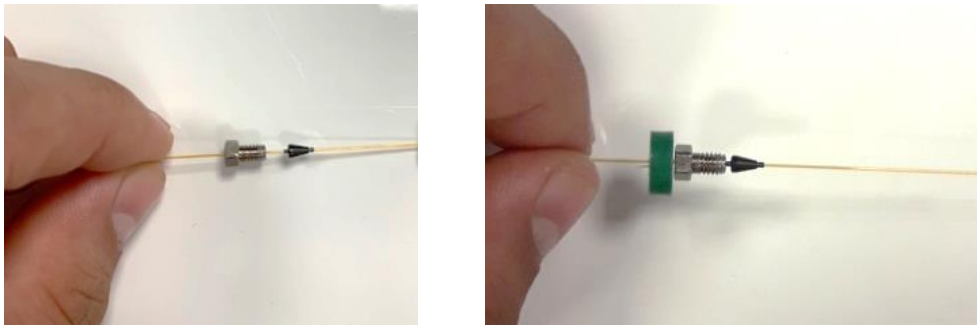


Figure 23. How to insert the Siltek MXT VALCO union male nut and the 1/32'' adaptor ferrules in a GC column. A thermostable GC septum can be used, but it is not essential.

5.4.4 Cut 2-3 cm of column. This step is critical and can affect negatively the system performance. Bad cuts and sharp GC column ends can cause peak broadening or carrier gas leaks, altering in last instance the results obtained. The cutting/trimming technique is described and depicted in sections 5.4.4.1 to 5.4.4.3.

5.4.4.1 Hold the column as shown in Figure 23.

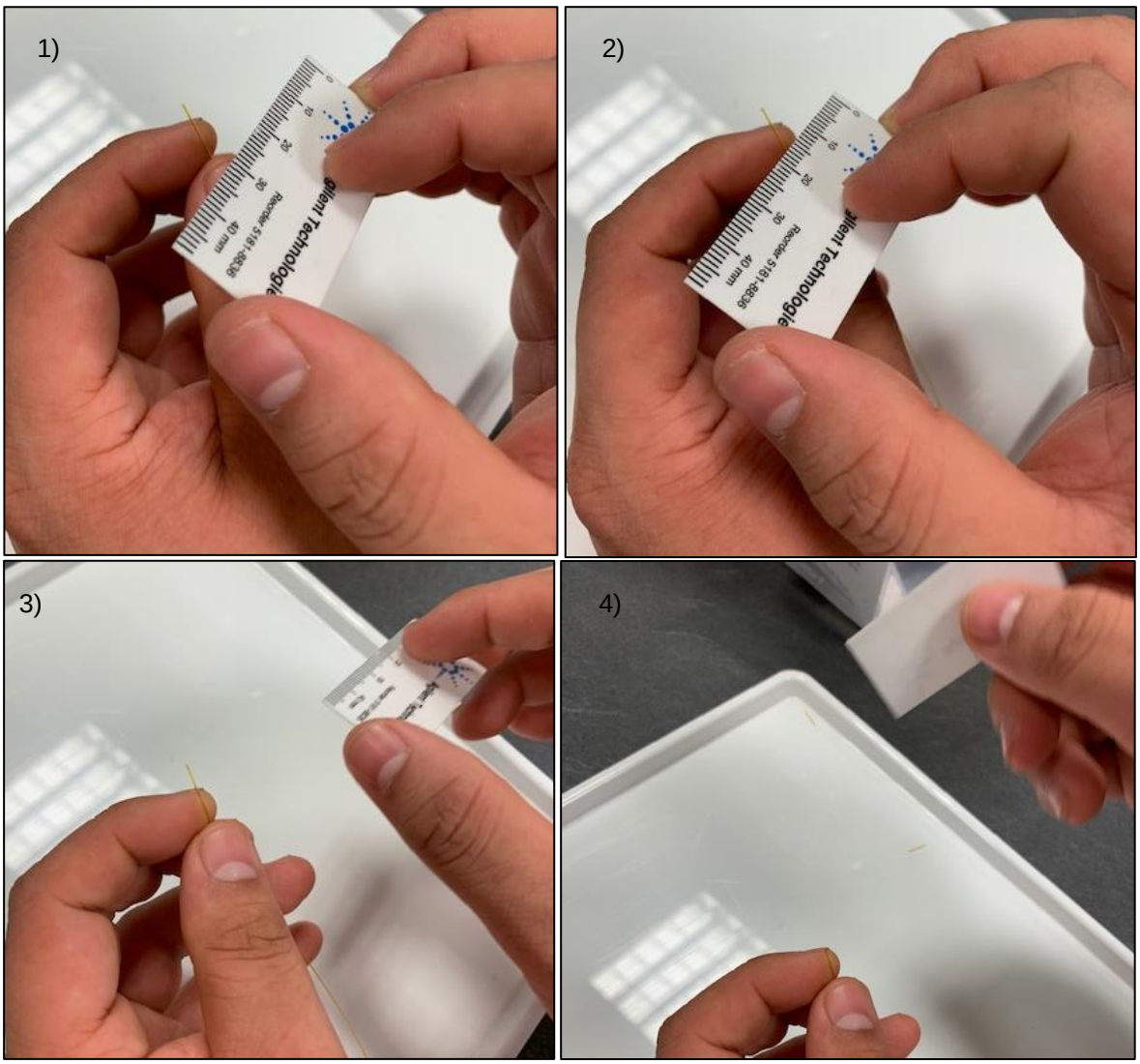


Figure 24. GC Column trimming technique. 1) Hold the end of the GC column. The cut is done in the GC section rest in the index finger. 2) Place the smooth side of the ceramic wafer perpendicularly to the capillary. 3) Gently, move the ceramic wafer alongside the finger to make a scratch on the capillary surface. 4) Flick the column to cut the column. If done properly, the cut should be straight and clean.

5.4.4.2 The ceramic wafer has a rough and a smooth side. Use the smooth side to make a superficial and horizontal scratch on the column. The aim is to cut the external coating evenly without damaging the inside of the column.

5.4.4.3 Flick the column end to break it.

5.4.4.4 Use a magnifier, check that the cut is clean and repeat if it is not (bad GC column cuts affects chemical elution and can lead to carrier gas leak). Figure 25 provides examples of good and bad cuts.

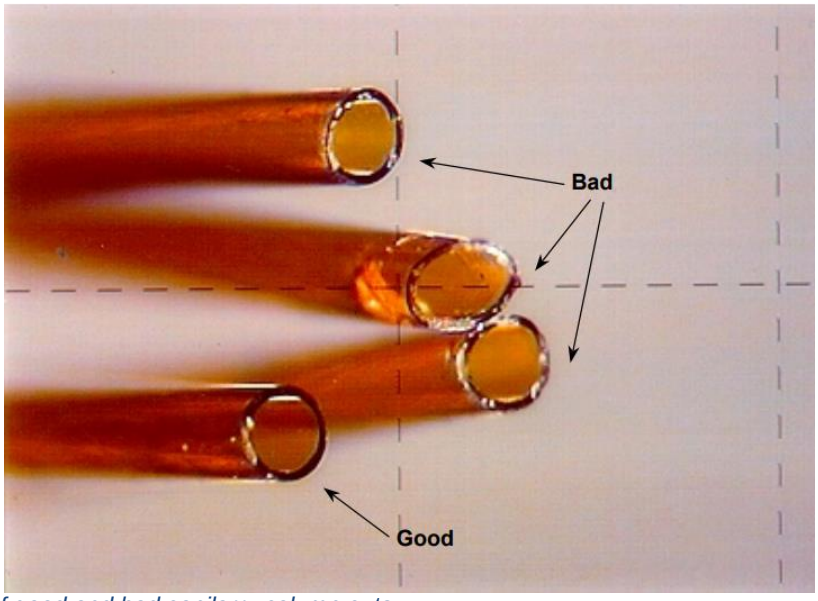


Figure 25. Examples of good and bad capilarry column cuts.

5.4.5 Once the cut is done, insert the column end in the VALCO union and pull the VALCO male nut. Finger tighten to hold in place.

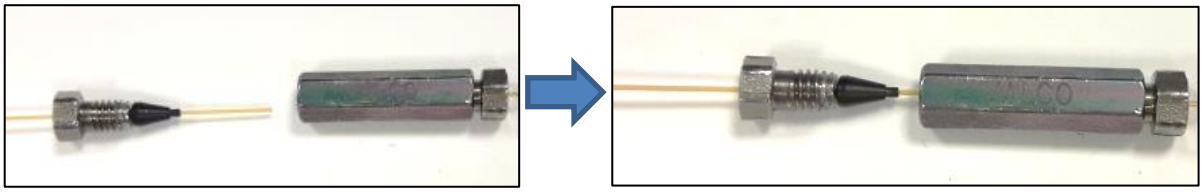


Figure 26. How to connect the GC Column to the Siltek MXT VALCO union. Leave a couple of cm from the GC column end and insert it into the union body. Pull then nut and finger tighten making sure the column end is fully inserted.

5.4.6 Use the Bahco (5mm x 5mm) spanners to tighten the 1/32'' nut. ¼ turn should be enough. A correct installation would be that in where the GC column is *fully* inserted, cannot be pulled out using the fingers and pass the leak check explained in section 5.6. If this is not achieved, tighten ¼ turn again and leak check. However, be careful not to overtighten the union as this will crack the end of the column, causing a permanent leak that cannot be solved by tightening more.

5.5 How to connect the GC column to the LNS transfer line.

5.5.1 Unwind the other column end. 21cm of this end will be inserted in the GC-LNS transfer line.

5.5.2 Insert the 1/8'' Swagelok nut, followed by a new Vespel Graphite ferrule.

5.5.3 Cut around 2-3 cm of the end of the GC column (repeating steps described from 5.4.4.1 to 5.4.4.3).

5.5.4 Mark the column 21cm from the cut end (liquid white Tippex can be used to mark the column), ensuring the nut and ferrule are less than this length down the column, so the mark is not erased when installing the column. Alternatively, a thermostable GC septum can be used.

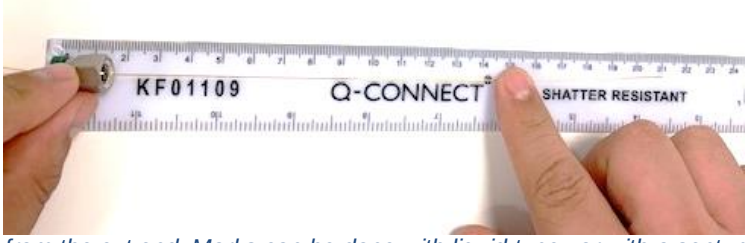


Figure 27. Measure 21cm from the cut end. Marks can be done with liquid typex or with a septum.

5.5.5 Pass the GC column through the GC-LNS Transfer line, loosely threading the nut and ferrule onto the 1/8'' Swagelok fitting so their weight is not being supported by the GC column.

5.5.6 When the 21cm mark is only visible after the rear of the 1/8'' Swagelok nut, tighten the nut carefully using two 7/16'' spanners so that the GC column does not move.

5.5.7 Open the carrier gas line again. This is essential when leak checking the installation and purging the column.

⚠	***Do not run the instrument with the carrier gas switched off. Heating a column without carrier gas flow results on permanent column damage***

5.6 How to perform a leak test and to purge the GC column.

5.6.1 Make sure the column is not touching the GC oven walls. Unwind more column if necessary, but DO NOT bend excessively the column.

5.6.2 Perform a leak test on the unions to confirm they are leak free. Tighten more if a leak is spotted (usually there is no need to tighten further than ½ turn. The presence of a leak after ½ turn or a full turn usually means the column end has been broken due to overnighting. If this occurs, cut the column again and repeat the process. The guidelines on how to perform a leak test are:

5.6.2.1 Take the Agilent G3388B leak detector and switch it on by holding the “Func/Power” button.

5.6.2.2 Let the leak detector to warm up away from the TD-GC-LNS system.

5.6.2.3 Press the “Zero/Enter” button to zero the signal.

5.6.2.4 Place the end of the needle close to the unions for around 10-15 seconds. Presence of green bars means the connection is leak-free (Figure 28). Presence of red bars in the leak detector correlates to the carrier gas leak, i.e., the greater number of ed coloured bars appear, the leakier the connection is. Resolve by tightening slightly more and repeat the leak check. Re-install the union if the leak persists after a couple of ¼ turns.

Note: it is important to leak check the unions after the first GC cycles once the column has been installed, as the unions may require re-tightening.

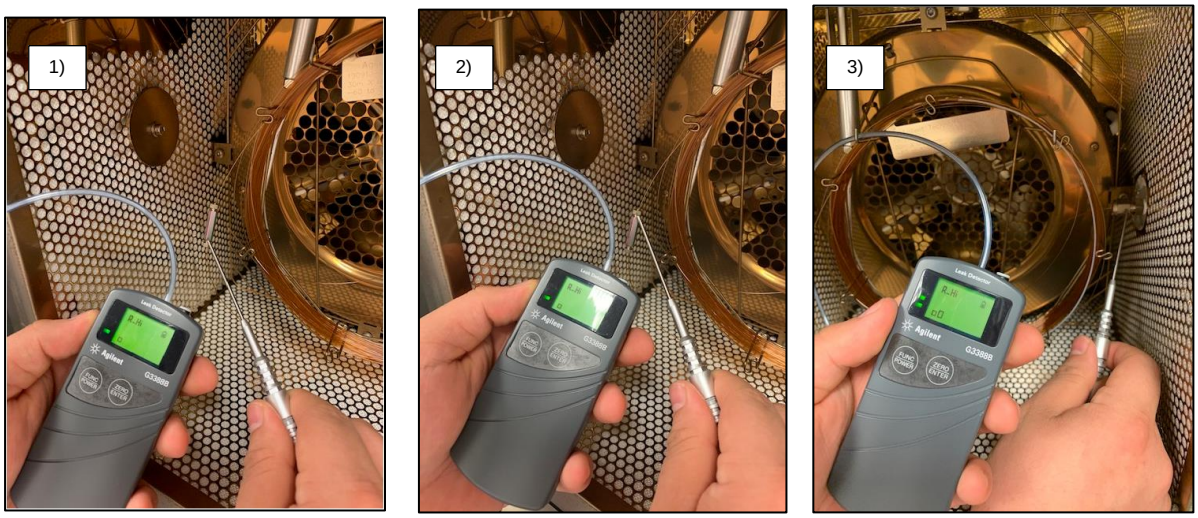


Figure 28. Pictures showing where to leak check. 1) Siltek MXT VALCO union, GC column side, 2) Siltek MXT VALCO union, TD transfer line side, 2) 1/8’’ Swagelok nut, connecting the GC column with the LNS transfer line. These connections must be tightened if the leak check fails.

5.6.3 Once the installation has been demonstrated to be leak-free, open the carrier gas valve and allow the gas to flow for a minimum of 20 minutes. This is to purge and remove residual oxygen and moisture that may have entered the column.

⚠	***The GC column must be purged before being heated in the oven to prevent stationary phase damage.***

⚠	***If a brand-new GC column has been installed, a column conditioning step is required prior running column blanks and samples.***

⚠	***If the GC column installed has already been conditioned, run a column blank after leak-checking and purging the column. This is to assess column performance, to enable baseline stabilization. Do not run any standard/sample.***

5.7 How to condition a GC column.

5.7.1 Make sure the GC system has been leak checked and the column purged after the installation.

⚠	***Heating the column with leaks in the GC column installation and/or without purging the column leads to permanent damage on the column.***

5.7.2 Ramp settings can be adjusted by navigating in the Thermo Trace 1310 GC, using the "*Instrument Control" > "Oven"* options. This process is detailed in *95-0012 User Guide: TD-GC-Lonestar: How to run a sample tube*

5.7.3 Conditioning settings varies depending on the GC column and the analytical method. Program the oven either to 20°C above the highest temperature in the instrumental method or to the column's maximum ISOTHERMAL temperature, whichever is lower.

Note: The HP-5 column isothermal is 325°C (maximum temperature that a column can be kept at for long periods without damaging the stationary phase). The HP-5 maximum programmable temperature is 350°C. This temperature can be held for a few minutes without causing permanent damage to the column.

⚠	***Do not condition the HP-5 over its isothermal max temperature of 325°C.***

5.7.4 An example of GC oven ramp rates settings for conditioning a HP-5 column, run at a final temperature of 240°C, are provided in Table 1. Same ramp rates of 10°C/min and initial and final times could be used HP-5 conditioning.

Temperature (°C)	40
Initial Time (min)	30
Ramp 1 (°C/min)	10
Final Temperature, depends on the method (°C)	260
Final time (min)	300
Ramp 2 (°C/min)	Off

Table 1. Example of HP-5 column conditioning settings.

5.8 How to run column blanks.

5.8.1 The TD does not require any preparation as there is no sampling during the GC column blank. However, the LNS must be set for data logging.

5.8.2 Ensure that the carrier (He) and purge (N2) gas cylinders are not empty and that the installation has been leak checked. Replace the cylinder if the pressure is less than 200PSI. Both gases should be 5.5 grade (i.e. 99.9995%). Check that the gas line toggle and GAS01 valves are opened. If a gas generator is feeding the system, ensure it is switched on and perform pertinent checks. The primary and secondary regulator pressures to be set are listed in Table 2

	Cylinder (primary) regulator	GAS01 (secondary) regulator
Carrier gas pressure	50psi	20psi
Purge gas pressure	50-60psi	50psi

Table 2. Gas line pressure settings.

5.8.3 Check the LNS and ensure the ATLAS pneumatic and heat boxes are on. The gas flow and pressure parameters, displayed at the top of the LNS interface, should be oscillating around 2800mL/min and the pressure 0.25bar respectively.

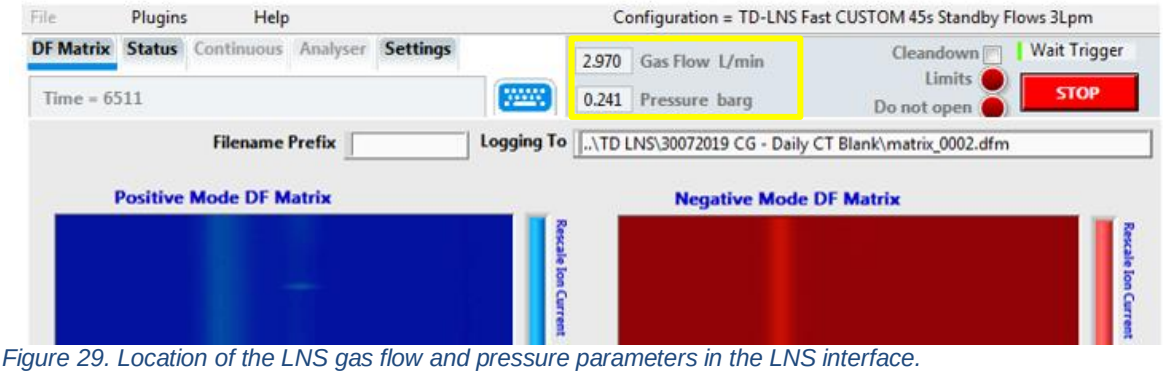


Figure 29. Location of the LNS gas flow and pressure parameters in the LNS interface.

5.8.4 If gas flow and pressure parameters are both zero, go to “Settings” and press the “COMM port” and “SFB settings” buttons, so that both display the message “Connect”, as shown in Figure 30.

Request technical support if the problem persists. For additional support, please visit www.owlstonemedical.com and click on Support or send an email to support@owlstone.co.uk.

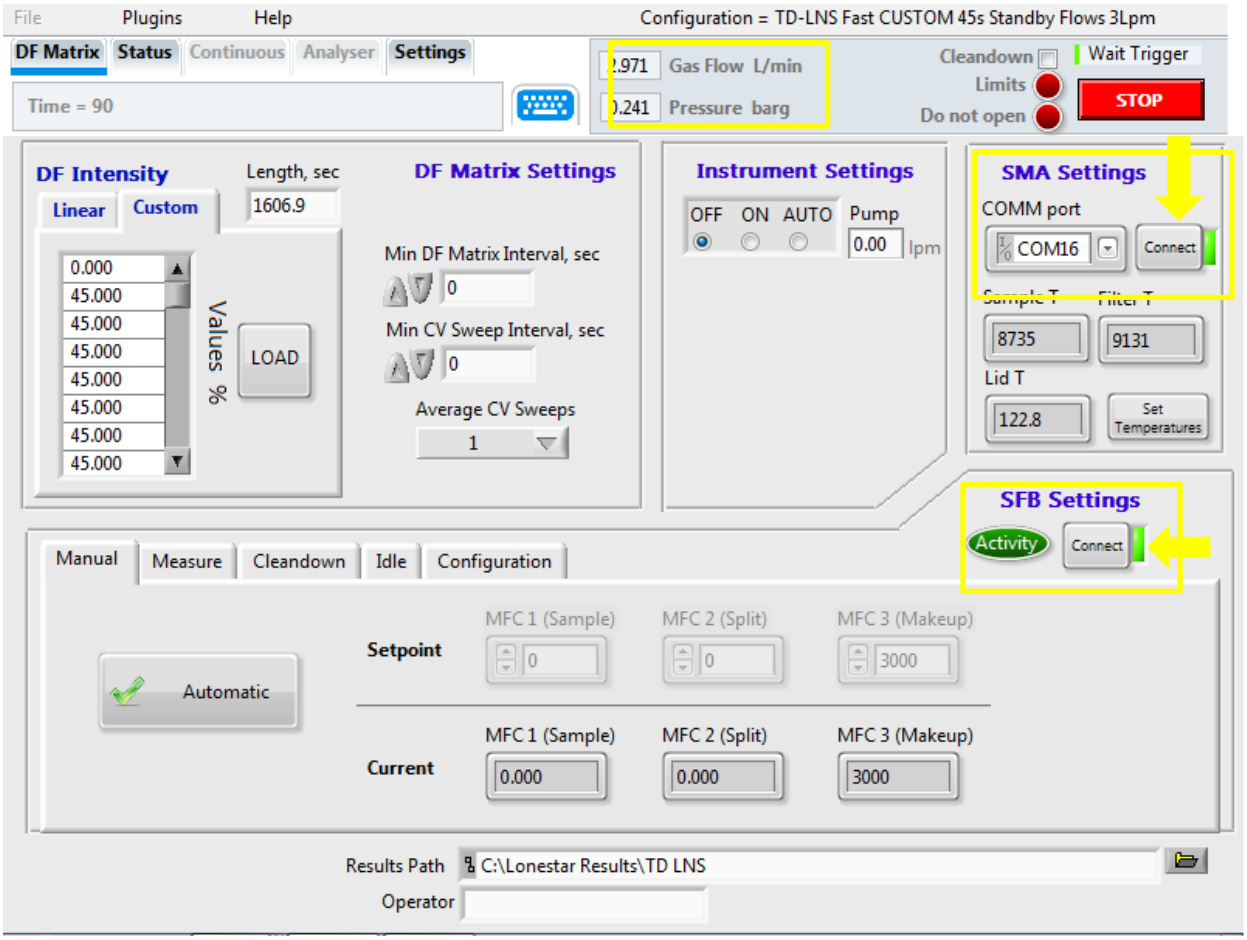


Figure 30. LNS interface screen. The yellow arrows point at the buttons that should be displaying “Connect”, press them if they are not.

5.8.5 Log the data on the LNS system. For guidance on this see *95-0012 User Guide: TD-GC-Lonestar: How to run a sample tube.*

5.8.6 Check the GC oven ramps settings. The GC column blank is to be done under same sample analytical conditions. Ramp settings can be adjusted by navigating in the Thermo Trace 1310 GC, using the "*Instrument Control" > "Oven"* options. This process is detailed in *95-0012 User Guide: TD-GC-Lonestar: How to run a sample tube.*

5.8.7 Press "Status" in the main menu interface. Wait until the oven has reached the correct temperature. The green Start Button will appear (Figure 31) once the GC is ready to inject. Press the Start Button to begin the GC column blank.

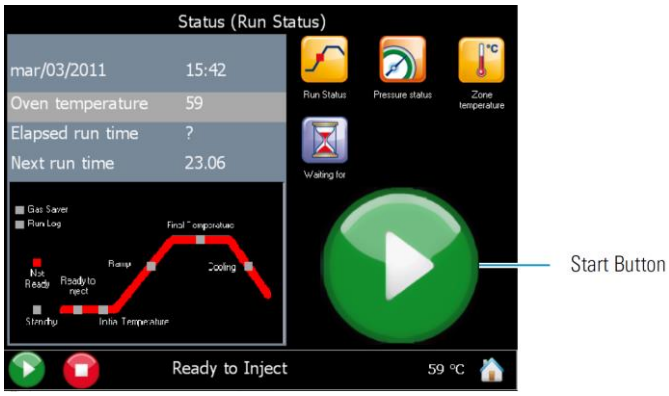


Figure 31. How to run a GC column blank: wait until the GC's status is "Ready to Inject" and then press the start button.

5.8.8 Once the GC cycle and the DF matrix have been completed, load and process the data generated on FAIMS Viewer. For guidance on this see *95-0011 User Guide- TD-GC-Lonestar: Offline data processing using the FAIMS Viewer Lonestar GC software*.

5.8.9 Demonstrate the GC column is blank – all background peaks should be <2pA with no upwards baseline drift (Figure 32)

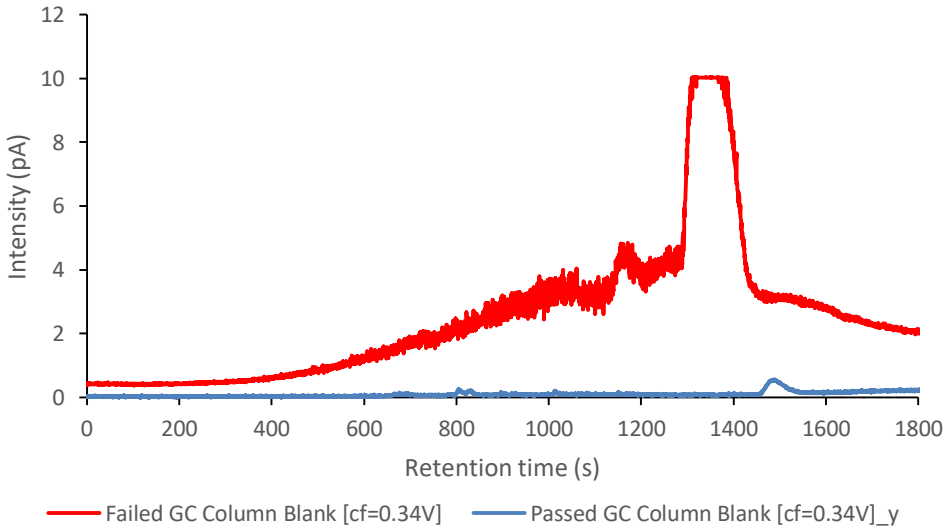


Figure 32. Comparison between and acceptable blank and a failed blank.

5.8.10 Repeat the GC column blank is failed. If the intensity drops, continue until the signal stabilizes. If the intensity does not vary, leak check, resolve and rerun the column blank.

Consider conditioning the column or installing a new conditioned column if the blank keeps failing. See Figure 33 (appendix).

6 Appendix

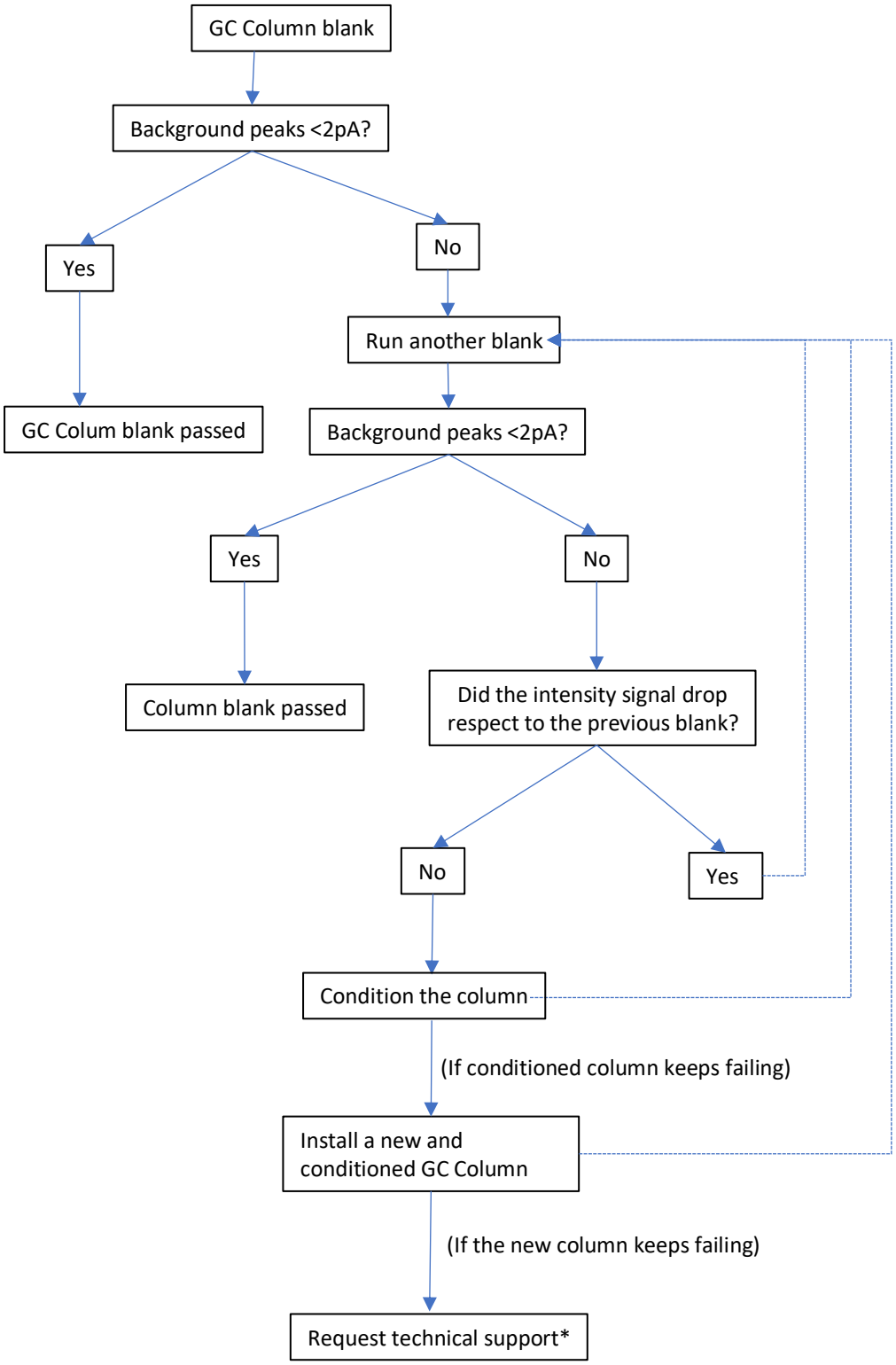


Figure 33. Steps in GC column blanking.

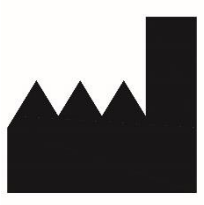
*For additional support, please visit www.owlstonemedical.com and click on Support or send an email to support@owlstone.co.uk.

7 Contacts and support

The Owlstone Medical Ltd team is dedicated to providing excellent support. For all technical and safe use question relating to this manual, contact as at:

Owlstone Medical Ltd.
183 Cambridge Science Park
Milton Road
Cambridge CB4 0GJ
United Kingdom

Tel: +44 (0) 1223 428200



Or email support at **support@owlstone.co.uk**