Lavigne Laboratories, LLC

Syringeless Injector Manual

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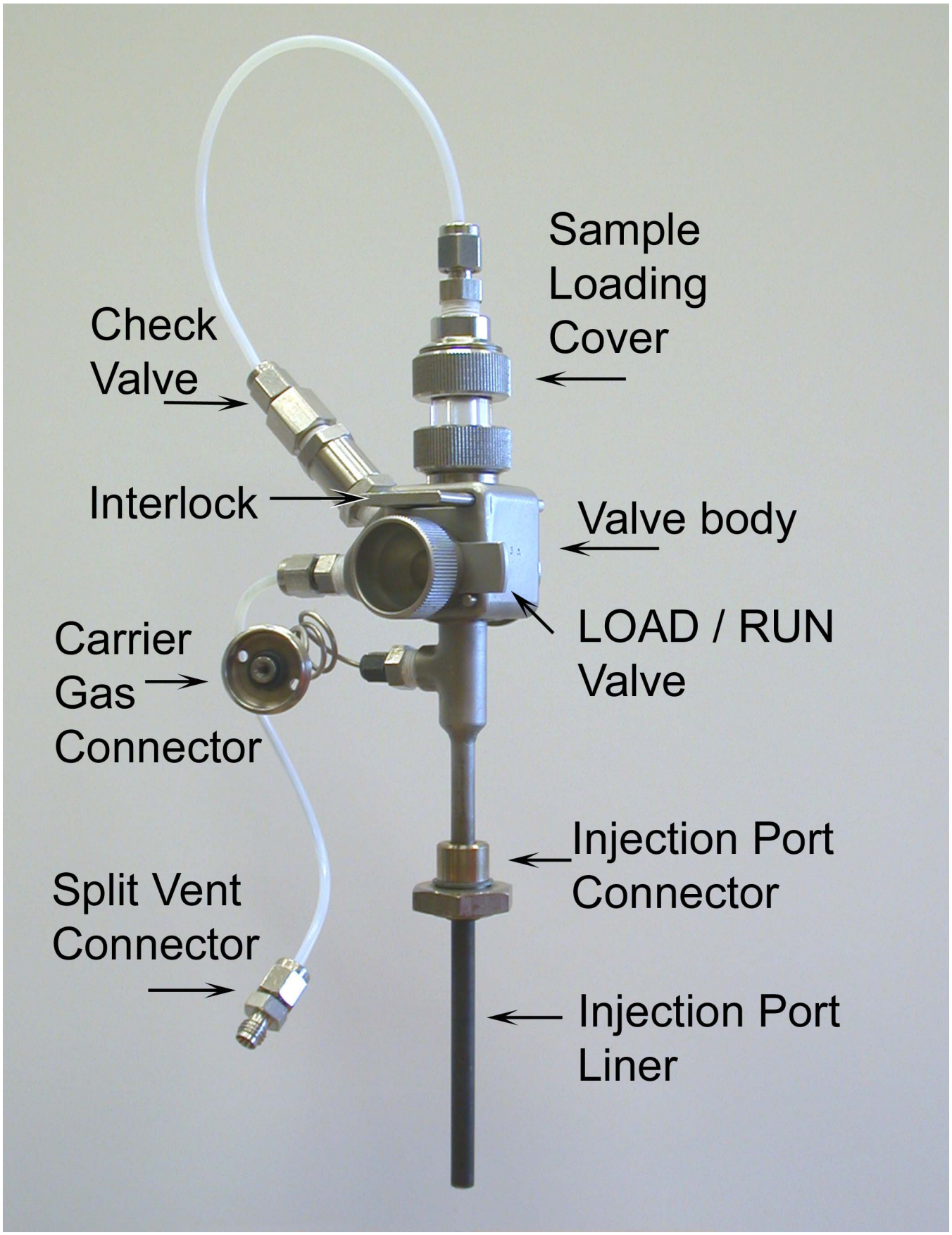
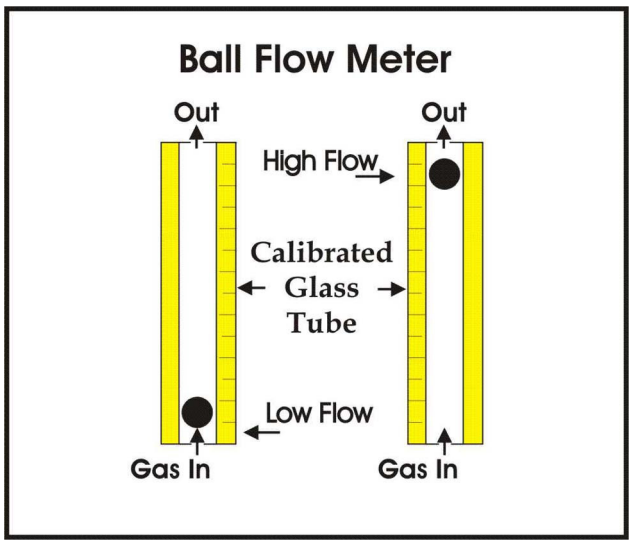
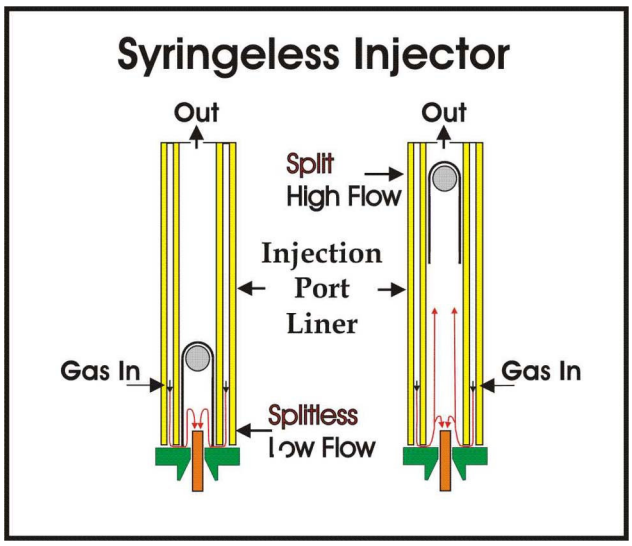




Theory of Operation

What moves the sample vial into and out of the injection port?

The Syringeless Injector operates like a Ball Flow Meter. The ball (sample vial) is suspended within a tube surrounded by a flowing stream of gas. As you increase the flow rate of the gas, the height of the ball increases.



The Syringeless Injector operates by switching the carrier gas flow within the injection port between Split / Splitless mode of operation.

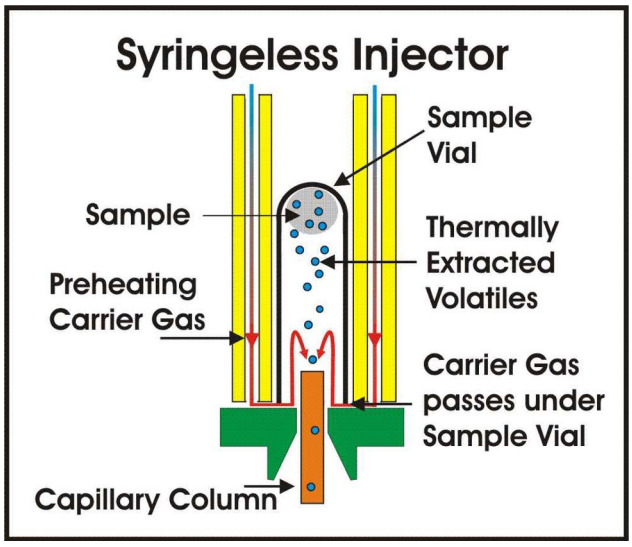
Splitless mode has a low flow rate of carrier gas within the Injection port which is insufficient to support the sample vial allowing it to fall into the injection port for sample collection. Split mode has a high flow rate of carrier gas which can expels the sample vial up and out of the injection port.

Splitless Operation

Low flow rate / Equal to Column flow rate
Used to sweep volatiles into the capillary column.

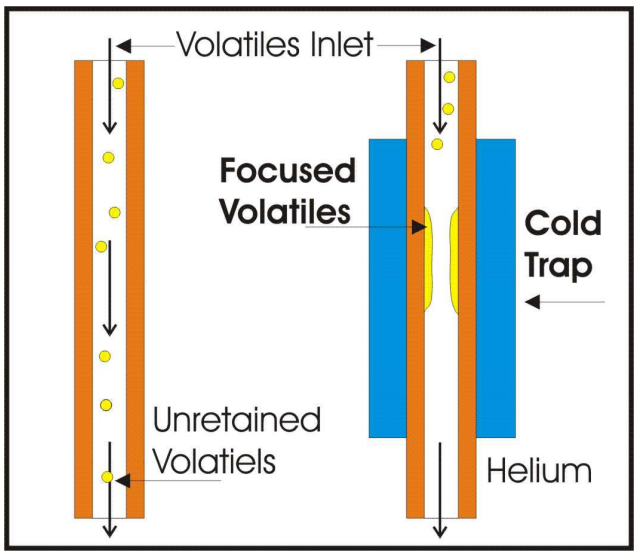
Split Operation

High flow rate / Total Injection Port Flow
Used to expel and or keep the sample vial out of the injection port.



Thermal Extraction of sample

Carrier gas is heated as it passes down through the Syringeless Injector's double walled injection port liner. The preheated gas enters at the bottom of the injection port. In order to reach the capillary column the carrier gas must squeeze under the wall of the sample vial. The velocity of the gas is increased at as this location helping to prevent thermally extracted volatiles from escaping. As the carrier gas flows into the sample vial it directs the expanding thermally extracted volatile gases into the capillary column for collection and separation.



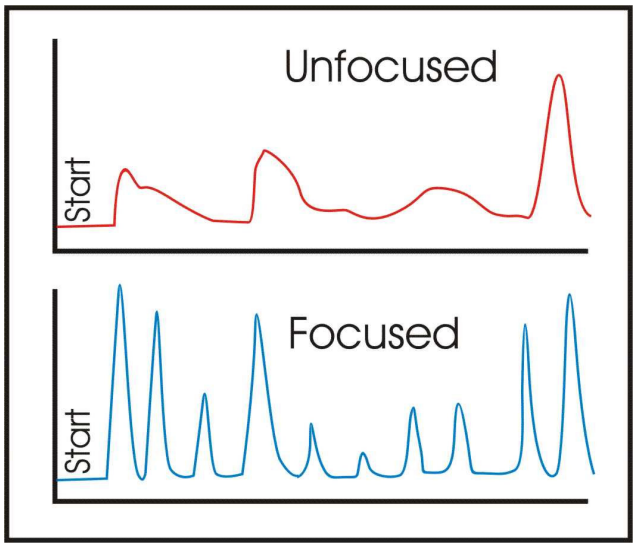
Cryofocusing volatiles

Sharp peak shape is the result of condensing volatilized organics from the vaporized sample into a narrow band at the beginning of the capillary column.

This is usually necessary for the highly volatile organics that will not condense at room temperate without cryogenics.

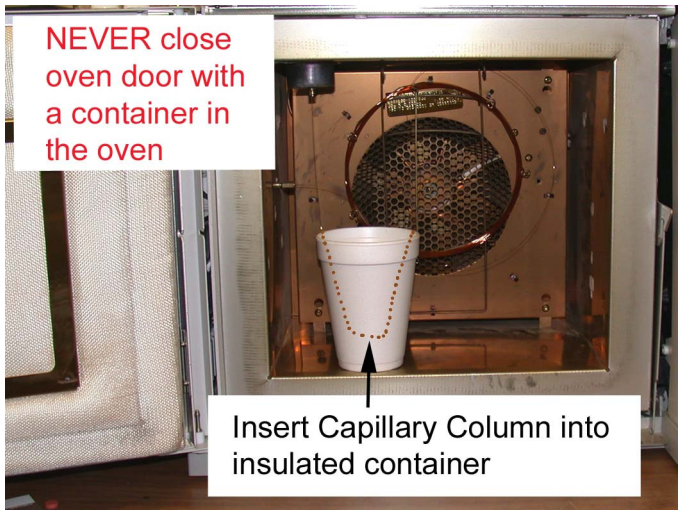
Warning

Condensing excessive amounts of volatiles from a sample can overload the capillary GC column and form a plug of frozen sample within the GC column. This will prevent the carrier from flowing though the column.



Removing the cryogenic trap allows the capillary column to quickly warm due to exposure to ambient room temperature. The room temperature volatile portion of the collected sample can now vaporize and start moving through the capillary column.

On the left is an example of what can be achieved by properly focusing the volatiles prior to chromatographic separation. This is most noticeable at the beginning of the run.



A Simple Cryofocusing Method

Place an insulated container filled with Ice water, crushed Carbon Dioxide or Liquid Nitrogen into the GC oven.

KEEP OVEN DOOR OPEN and OVEN OFF

Insert the beginning of the Capillary GC column into the container to form a U shaped trap. Remove the trap when the collection process is completed and close the oven door.

Danger

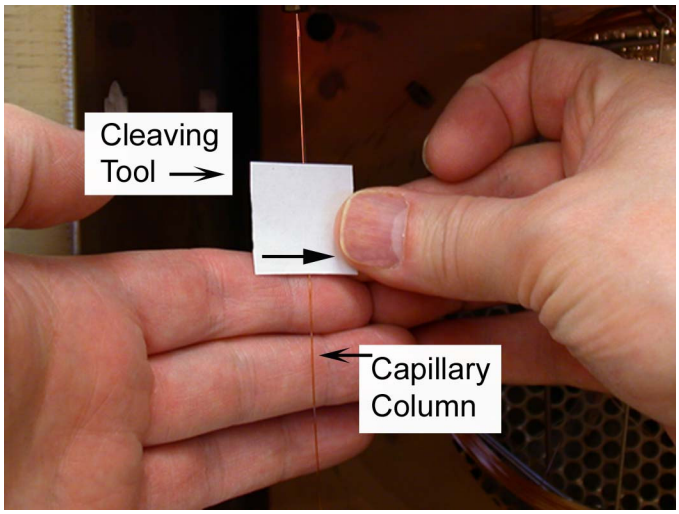
Remove the container before closing the oven door and turning on the GC oven.

Disconnecting your column

Refer to you Gas Chromatograph manual for direction on changing septa, injection port liners and columns, be sure to read and follow all manufactures directions.

- Shut off GC Oven
- Shut off Detector
- Shut off Injection Port

Allow all heated zones to cool to room temperature before starting.



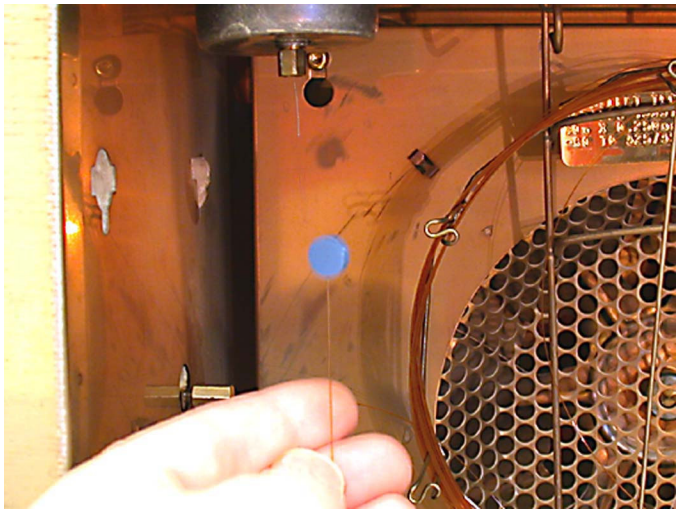
Step #1

How to cut a capillary column

Support the capillary column with one hand and hold the cutting tool with the other. (as shown)

Scratch the column in one direction, by dragging the cleaving tool across the surface of the capillary column. Just a scratch will do. Do not try to cut completely through the column.

Now bend the column, and it will break at the scratch.



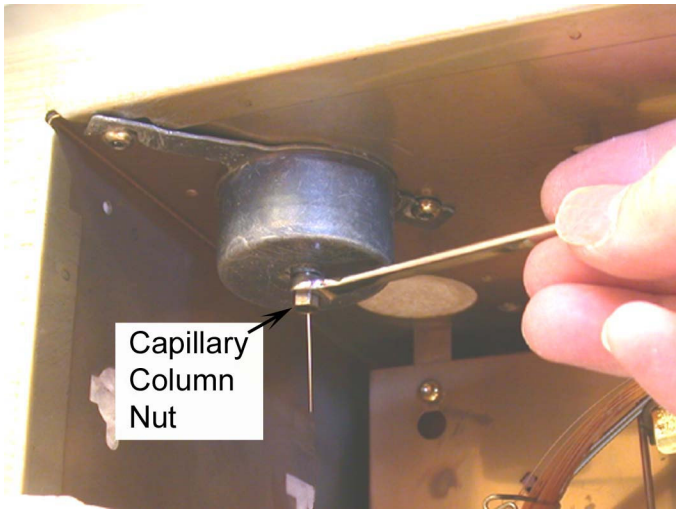
Step #2

Plug the capillary column with a septum.

GC/MS users

Plug the column **immediately** after cutting.

Plugging the column prevents room air from continually being drawn into the Mass Spectrometer by the vacuum system



Step #3

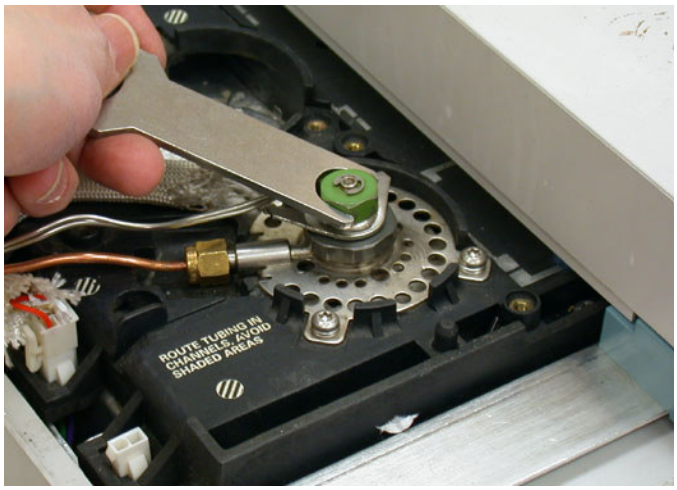
Remove the capillary column nut, along with the remaining short section of capillary column.

Proceed to Syringeless Injector Installation

Syringeless Injector Installation

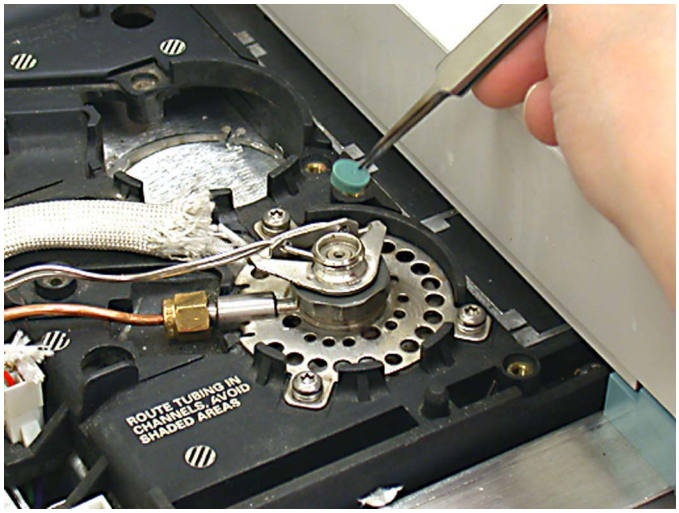
Refer to your Gas Chromatograph manual for changing septa, injection port liners and columns, be sure to read and follow all manufactures directions.

Allow all heated zones to cool to room temperature before starting.



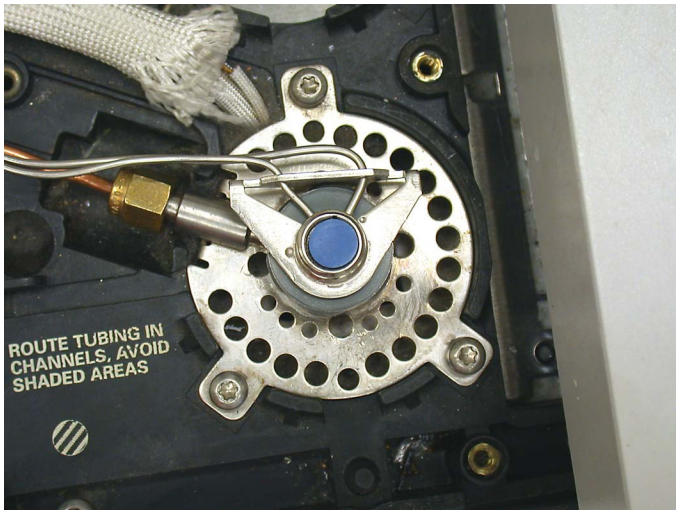
Step #1

Remove septum cover



Step #2

Remove septum using tweezers



Step #3

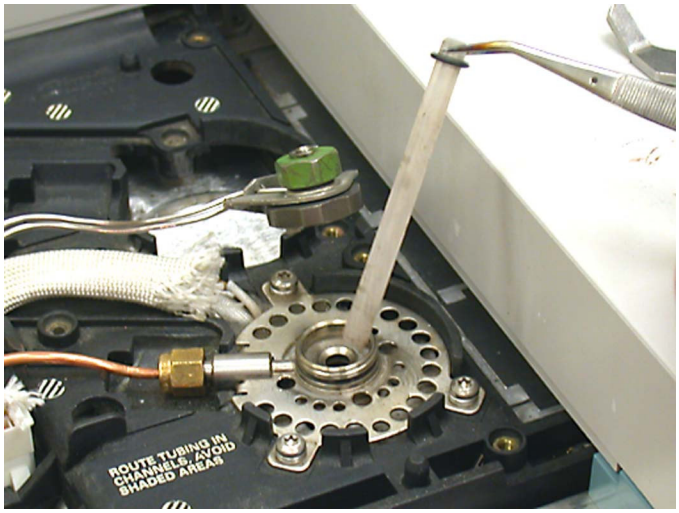
Insert new septum
Replace septum cover

Do not use predrilled septa

Do not over tighten septum cover

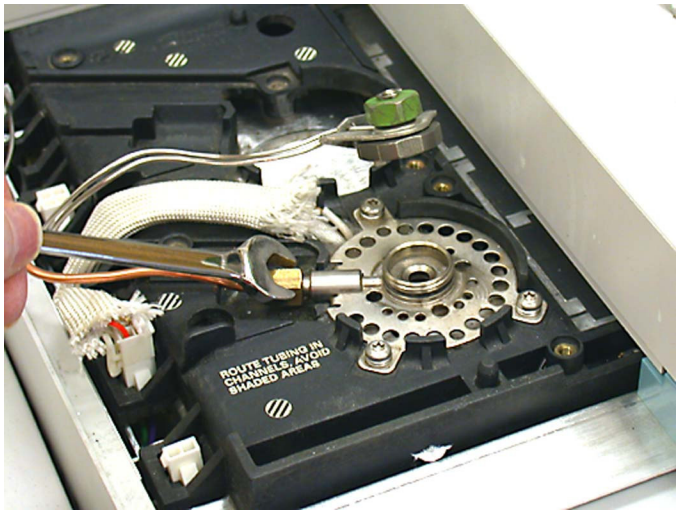
Step #4

Unscrew injection port nut



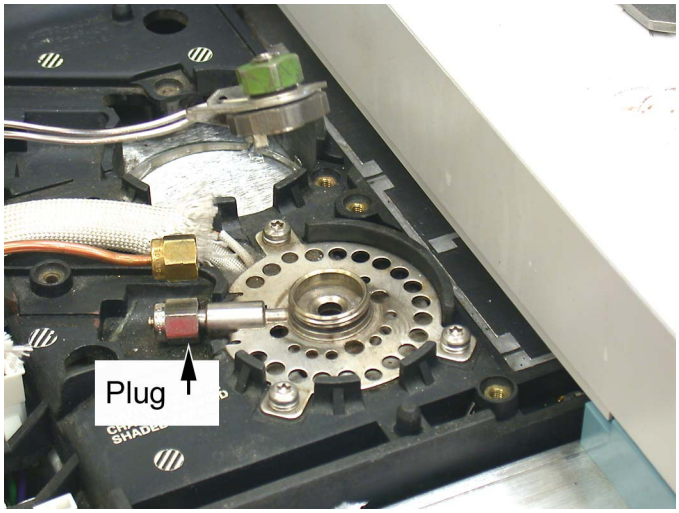
Step #5

Remove injection port liner



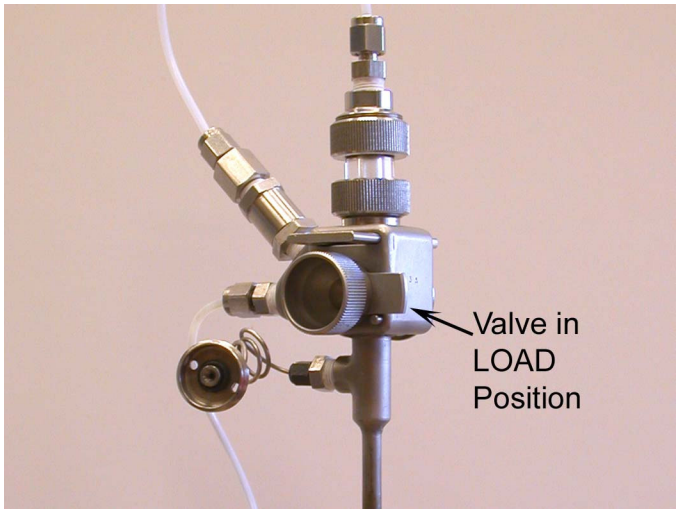
Step #6

Disconnect split vent line from injection port



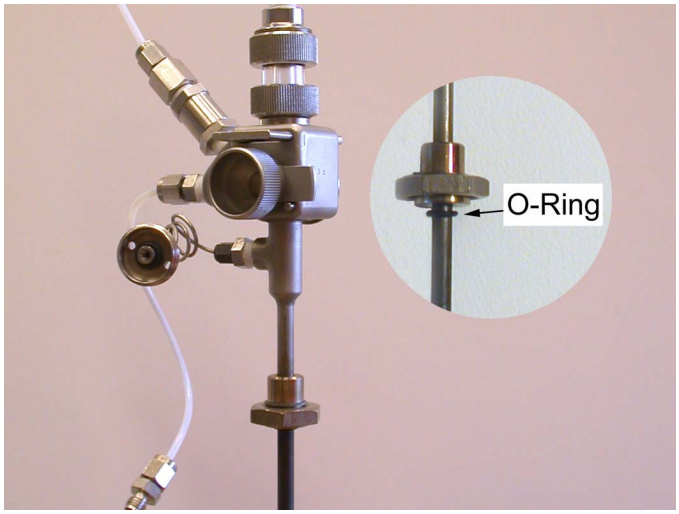
Step #7

Plug injection port split vent, with compression fitting.
(Supplied)



Check position of Load / Run valve before installing

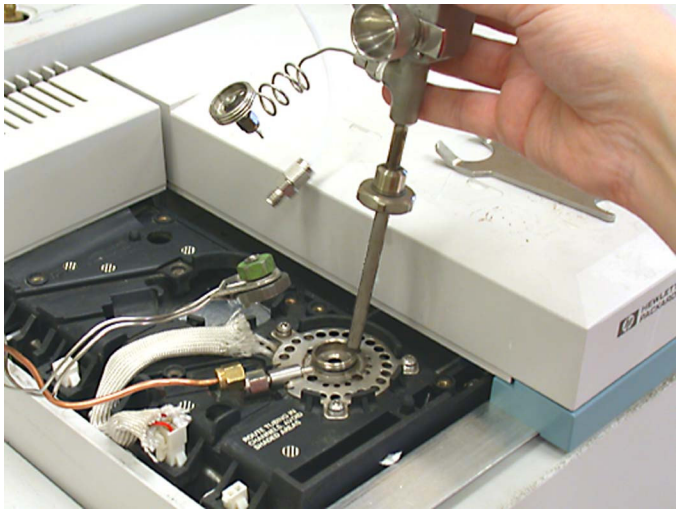
Move Valve to <u>LOAD</u> position



Check before installing

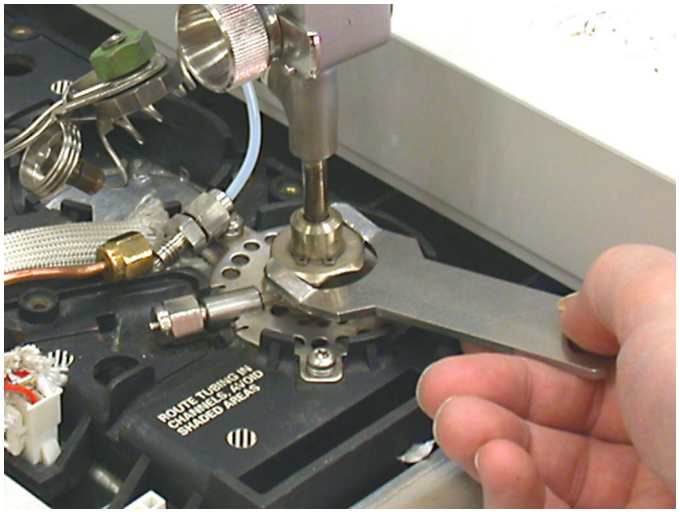
Select proper O-Ring materials

Injection port Temperature
Ambient to 300C –Viton
300C to 400C - Graphite



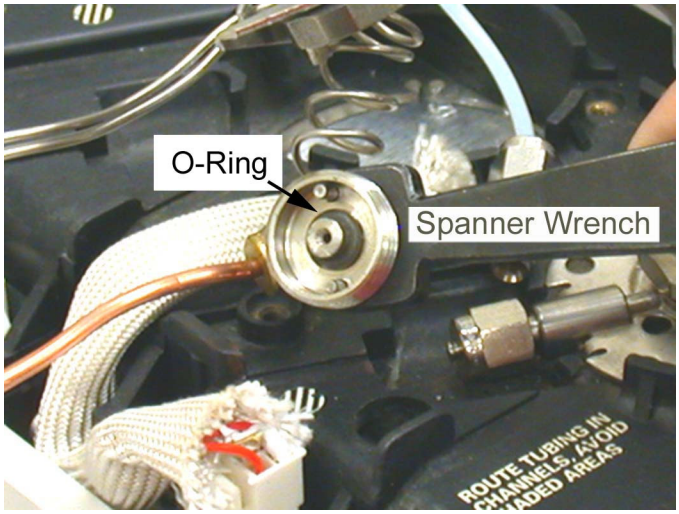
Step #8

Insert Syringeless Injector
into injection port



Step #9

Connect Syringeless Injector
Tighten Injection port connector

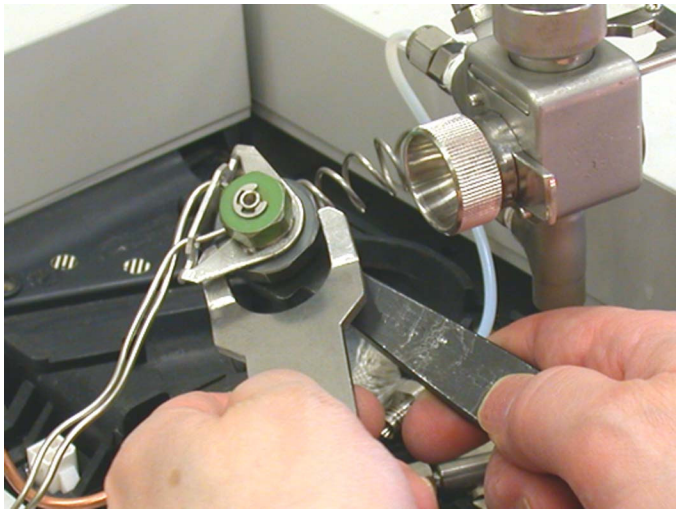


Step #10

Place Spanner Wrench (supplied) behind Carrier Gas Connector

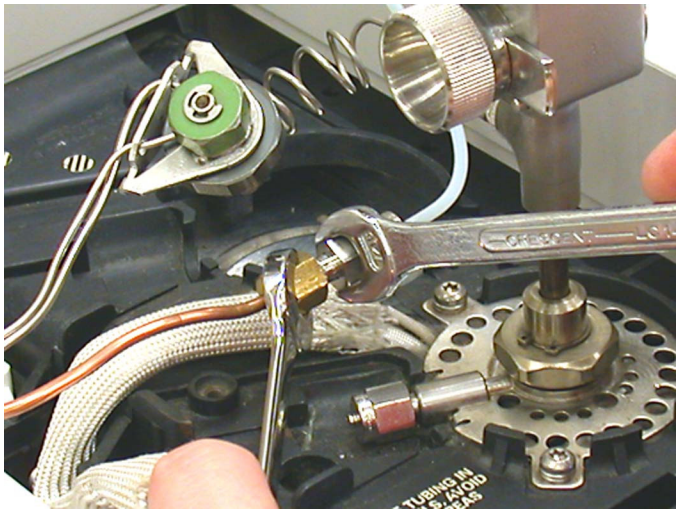
Check for O-Ring

Make sure O-ring is present on Carrier inlet connector.



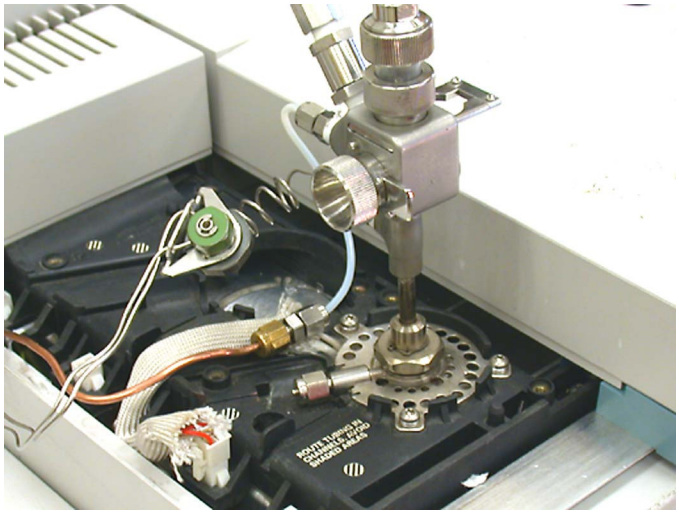
Step #11

Tighten injection port nut to carrier gas connector



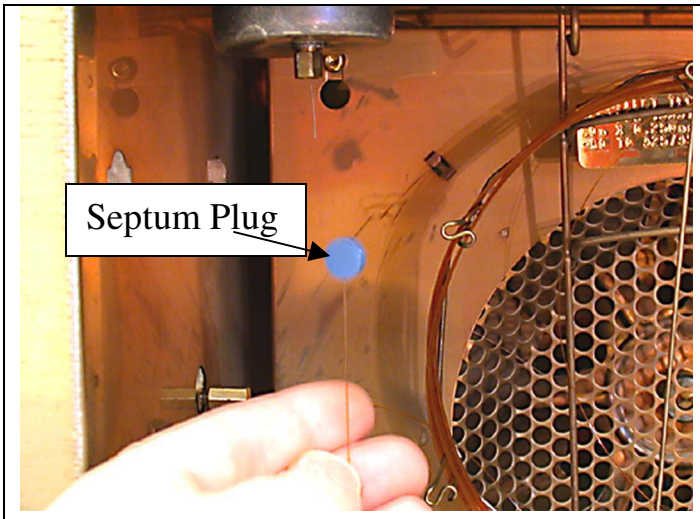
Step #12

Connect Split vent line to Syringeless Injector



Completed installation of Syringeless Injector

Proceed to Capillary Column Installation



Capillary Column Installation

Step #1

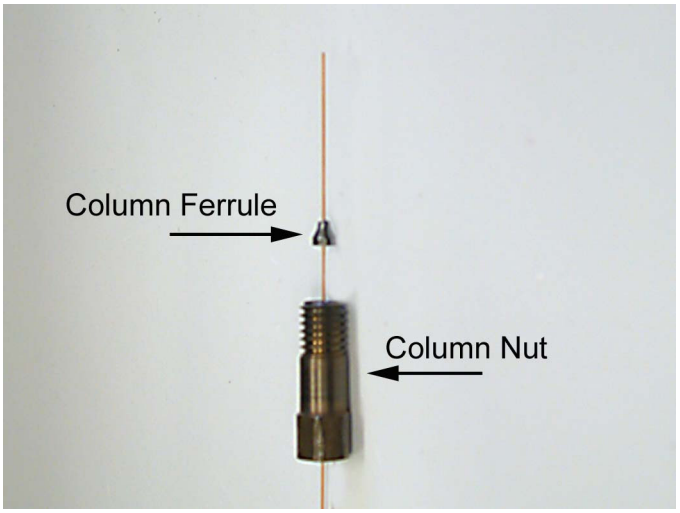
Turn on Carrier Gas

Set total flow to 350ml/min

Step #2

Remove the septum plug

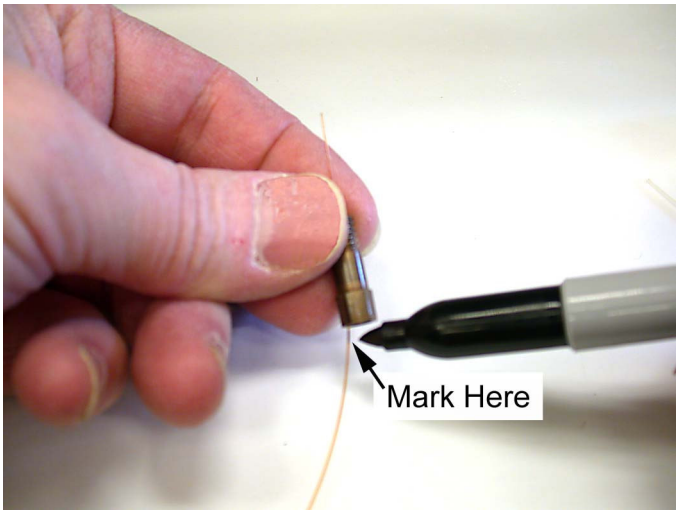
Usually a small piece a septa will remain inside of the column, it will keep the column plugged and prevent ferrule particles from entering the column on step #3



Step #3

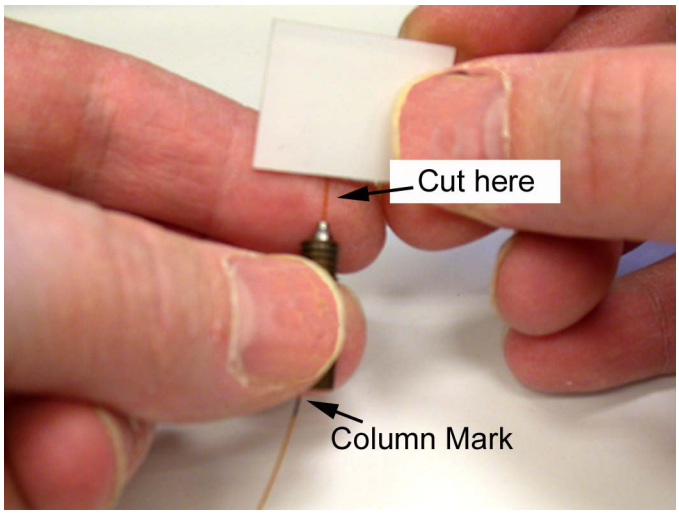
Slide Column Nut then
Push Ferrule onto column.

Pushing the column through the graphite ferrule will cause particles to build up at the inlet of the capillary column. The next two steps will remove the plugged end of the column and any graphite particles.
Do not skip steps #4 and #5



Step #4

Mark column at bottom of nut



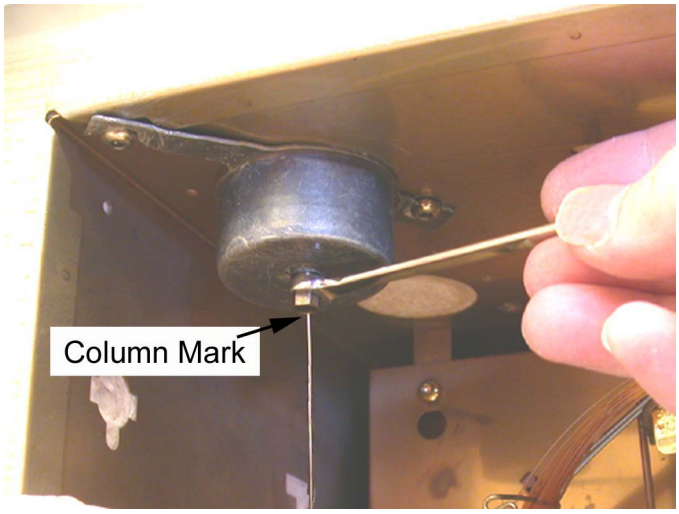
Step #5

Cut column approximately 5 mm above ferrule

Your column is now ready for installation to the injection port.

Warning

Before proceeding to step #6 make sure your carrier gas is on and set to 350 ml/min total flow.



Step #6

Insert column nut into injection port and tighten.

The column mark should be visible at bottom of column nut. Adjust the column up or down if necessary to make the mark visible.

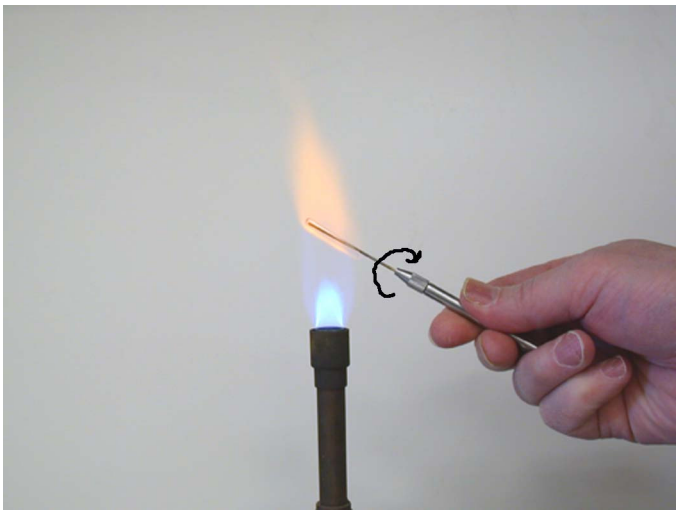
As soon as the column nut is tightened, the injection port pressure should increase immediately.

No pressure check trouble shooting section.

Congratulations

Your installation is complete

Continue to Sample Preparation



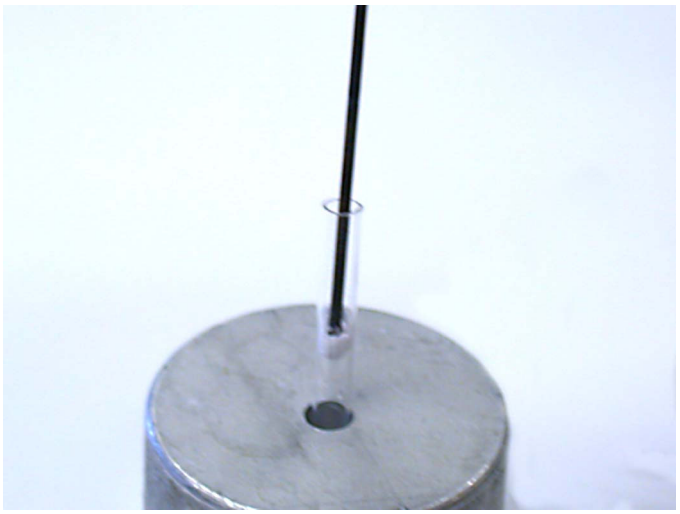
Sample Preparation

Step #1
Cleaning Sample Vials

Hold the probe with sample vial at an angle (as shown)
Rotate in flame for 5 to 6 seconds
or
Place in vials in 500C furnace until clean.

Do not distort shape of tube.

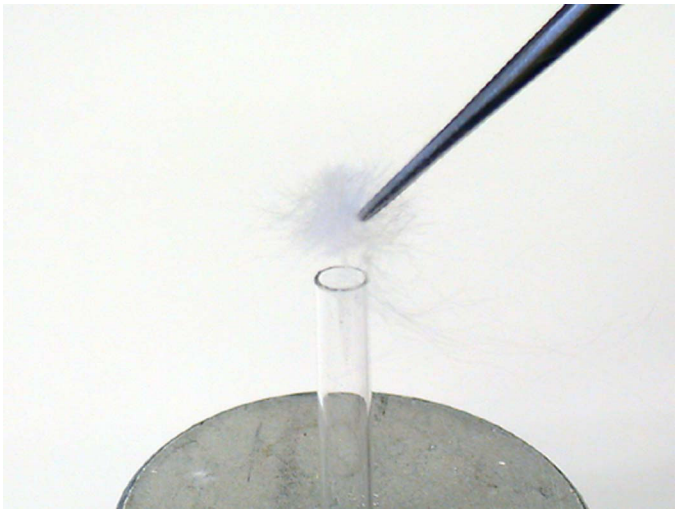
Allow sample vial to cool completely before weighing



Step #2

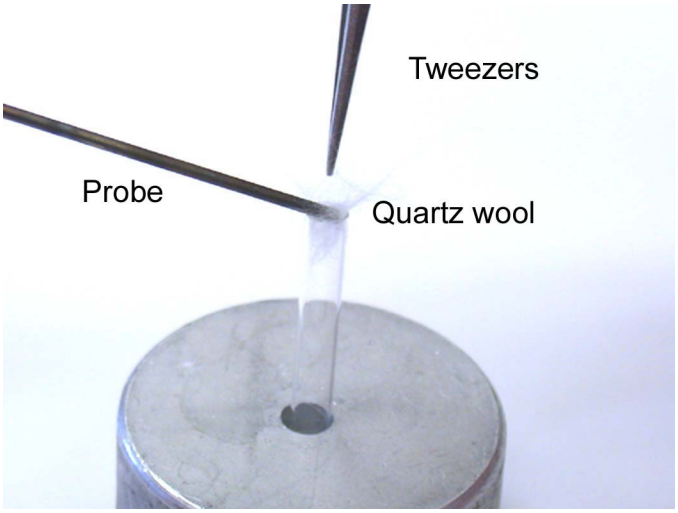
Push sample into bottom of Sample Vial

Weigh sample for Quantitative analysis.



Step #3

Flame quartz wool to clean.
Allow to cool
Insert clean quartz wool
into Sample Vial

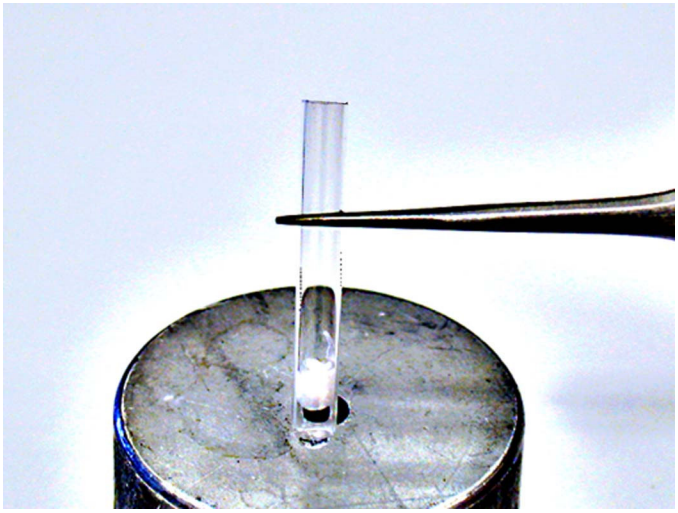


TIP: Probe and tweezers work together to insert quartz wool into vial.

Push quartz wool into beginning of vial with tweezers.

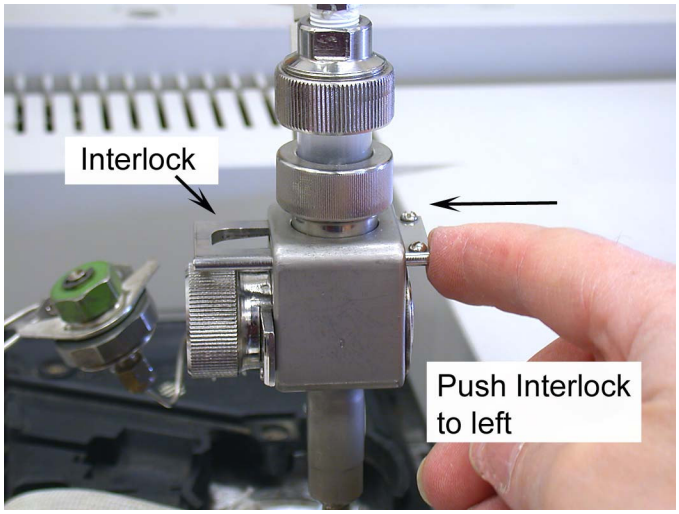
Hold quartz wool in place using the probe when removing tweezers (see photo).

Once quartz wool is partially within sample vial, push the quartz wool down onto the sample with the probe.



Sample Preparation Completed

Proceed to
Syringeless Injector Operation

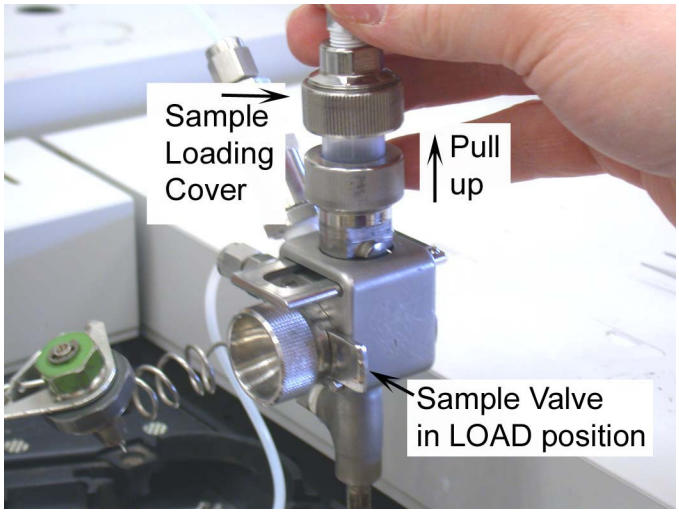


Syringeless Injector Operation

Step #1

To unlock Sample Loading Area push interlock to left.

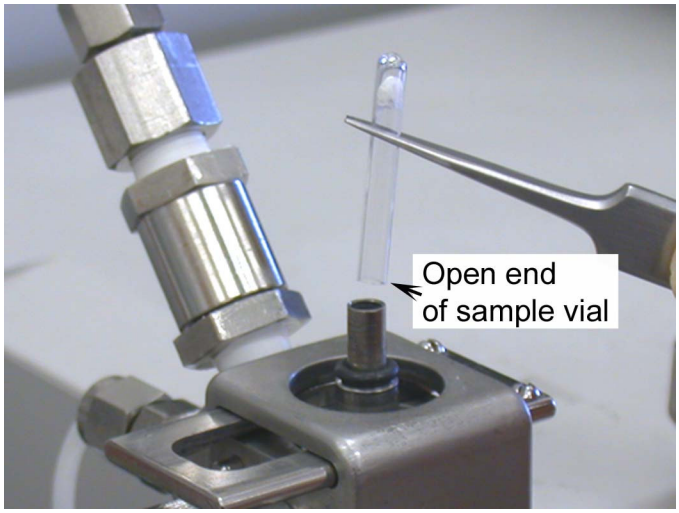
Sample valve needs to be in Load position



Step #2

Remove Sample Loading Cover
Do not force!
Make sure interlock is all the way to the left.

Caution
Never move the Sample Valve to RUN position without Sample Loading Cover in place.

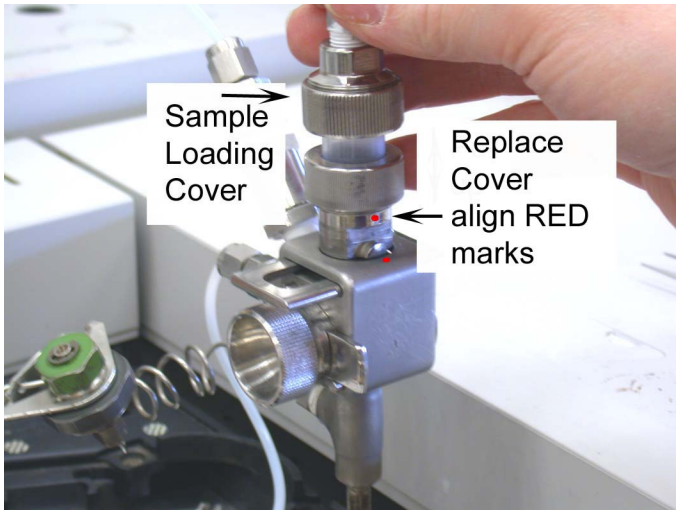


Step #3

Insert Sample Vial into Syringeless Injector with tweezers.

Open end at bottom

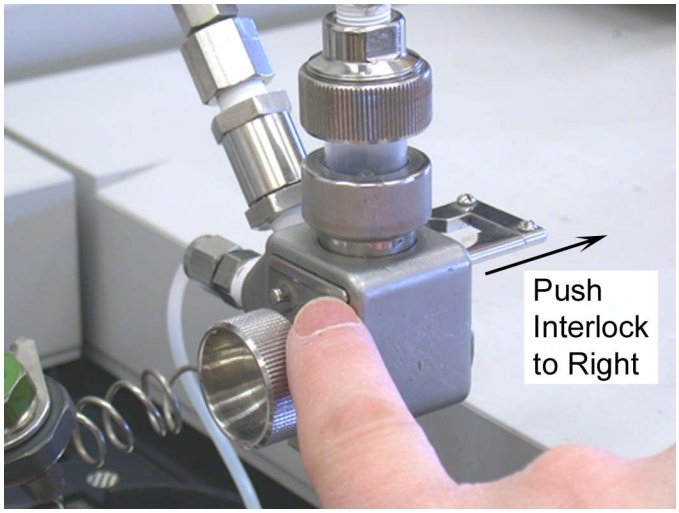
Run a Blank run (empty sample vial) to check for contamination in system.



Step #4

Replace Sample Loading Cover

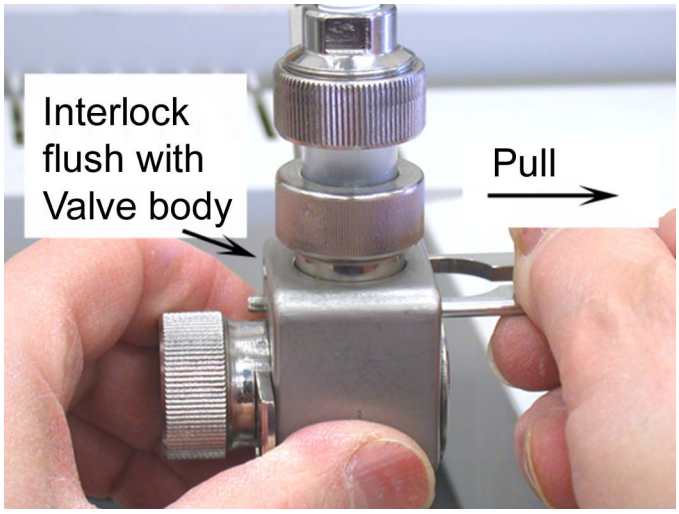
Use red marks to align cover.



Step #5

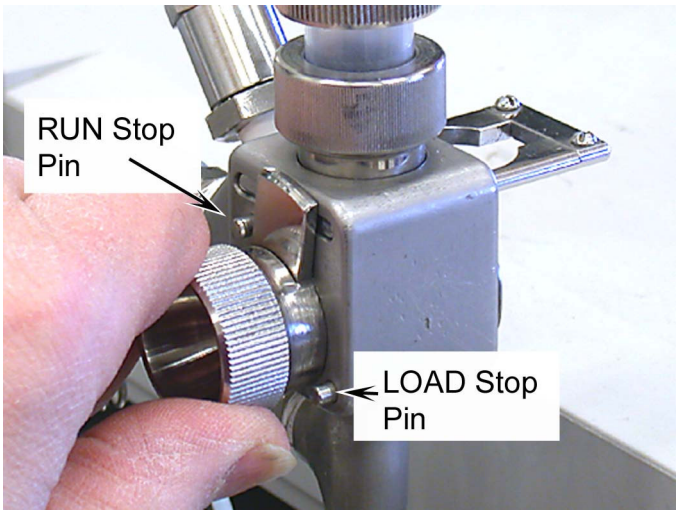
Push Interlock to Right

Interlock should slide freely.



Step #6

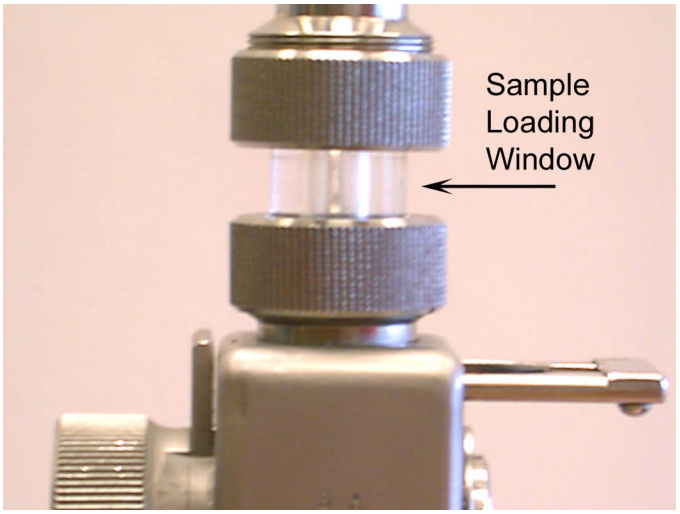
Holding the Valve body with your left hand, gently pull the Interlock all the way to the right using your right hand. The interlock should be flush with Valve Body to seal Sample Loading Cover and allow rotation of the valve to RUN position



Step #7

Rotate Valve from
LOAD to RUN position

Valve must contact
RUN stop pin.



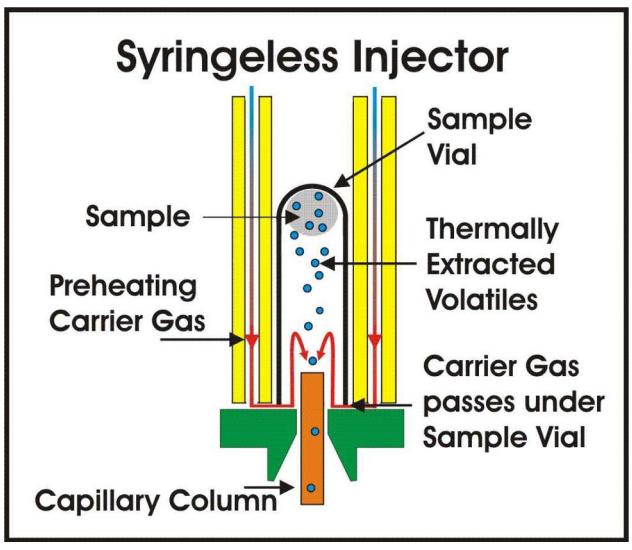
Sample tube should be visible, floating in a stream of carrier gas, through the Sample Loading Window

Allow carrier gas to displace air in sample vial for 15 seconds before injection.

If the sample vial falls into the injection port during rotation of the sample valve you have insufficient flow within you injection port or your split vent is OFF

Total injection port flow = 350ml/min

Initial Injection port mode = Split mode.



Sample vial moves into injection port by switching from split to splitless operation.

Example for a 2 minute Thermal Extraction

Split valve initially ON

Splitless Control

ON TIME = 0.01 minutes

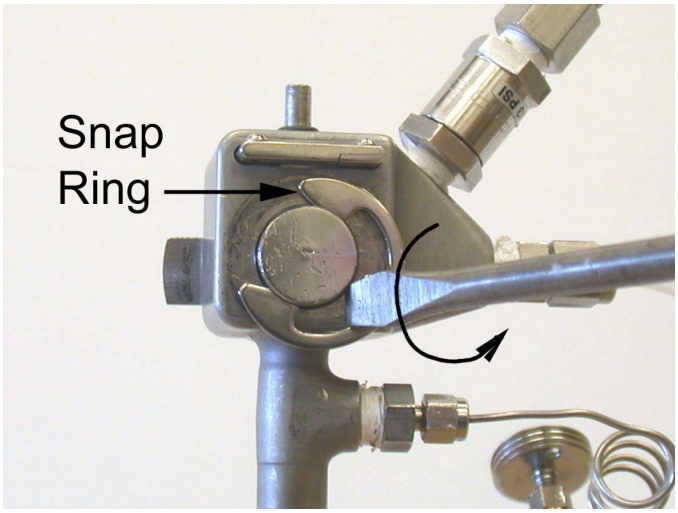
Sample falls in injection port when STARTrun button on GC is pushed.

OFF TIME = 2.00 minutes

Sample is expelled from injection port

Refer to your GC manual for
Split / Splitless injection operation.

Syringeless Injector Cleaning

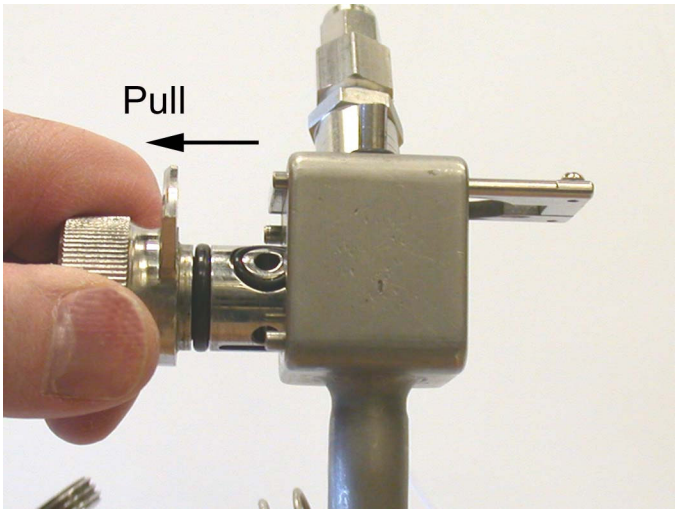


Remove Syringeless Injector from Injection port

Step #1

Remove Snap Ring

To remove place flat blade screw driver between Valve and Snap Ring and twist screw drive. (as shown)

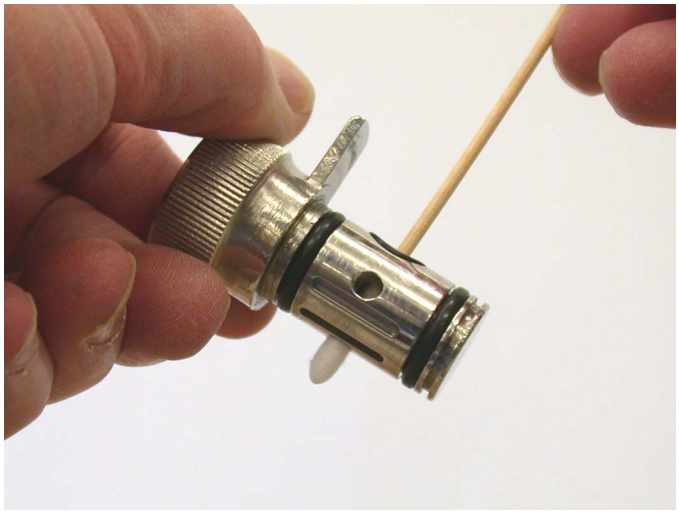


Step #2

Pull Valve straight out

Step #3

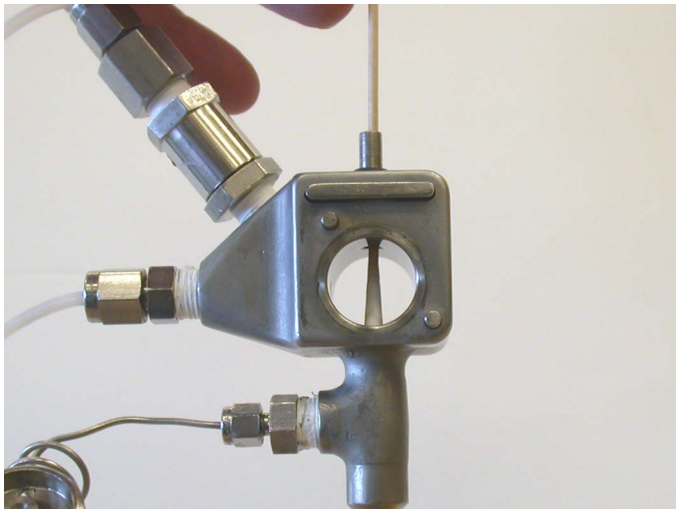
Remove O-Rings



Step #4

Use a Cotton Swab
saturated with Methanol

Clean all areas of valve
Inner and outer surfaces



Step #5

Also Clean down through
Syringeless Injector body

Push Cotton Swab through from one end to the other.
Repeat starting at the other end.
Continue until swab comes out clean

Proper method to apply silicone grease to O-Ring

Object is to apply coating of silicone grease as thin as possible a to O-Ring.

Apply a small amount of silicone grease to thumb and forefinger and rub together to thin out grease. Then place O-Ring between fingers and smear over surface. Surface should appear shinny and smooth not sticky.
More in this case is definitely not better.

Correct

Wrong

Step #6

Apply Silicone Grease to O-Ring

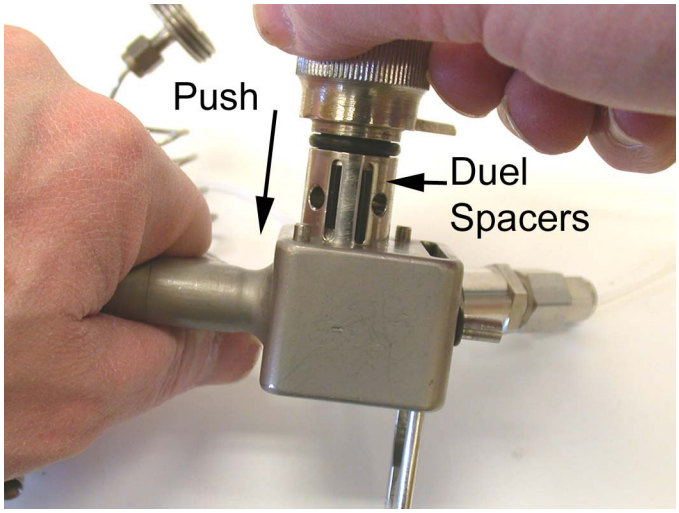
Correct

O-Ring should have a shinny appearance.

Wrong

Globs of Silicone

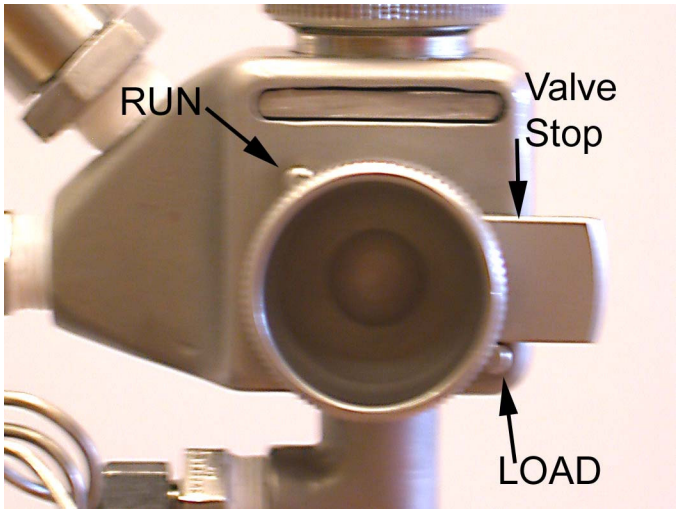
Sticky feeling to Surface



Step #7

Replace O-Ring on valve and insert valve into valve body

Duel rubber spacers should be facing up to prevent then from falling out when pushing valve into valve body.
Valve should slide in smoothly.



Step #8

Make sure Valve Stop is between Load and RUN positions before replacing snap ring

Slide the Snap Ring onto valve and tap in place with the screw driver handle. Ring should snap to place

Trouble Shooting

Symptom	Cause	Solution
No column pressure	Major leak in Injection port	✓ Rotate Valve to LOAD position ✓ Sample Loading Cover in place ✓ Make sure plug is in Injection port ✓ Check O-Rings Liner and Carrier ✓ Split Vent line connect to Syringeless Injector ✓ Replace Capillary column ferrule
Loss of Column pressure when removing Sample Loading Cover.	Depressurization of Syringeless Injector	✓ Valve must be in LOAD position when removing Sample Loading Cover ✓ Malfunctioning Check Valve (Replace)
Sample Vial falls into injection port immediately after turning Valve from LOAD to RUN position.	Insufficient Total Injection port Flow	✓ Split mode initially ON ✓ Total Flow set to 350ml/min
Sample Vial will not fall into injection port	Obstruction or High flow within Syringeless Injector	✓ Valve in RUN Position?. ✓ Switch Injection port controls from Split to Splitless operation
Sample Vial will not come out of injection port.	Insufficient High Flow within Syringeless Injector	✓ Total Flow set to 350ml/min ✓ Increase Total Flow > 350 ✓ Leak in injection port septum ✓ Split vent line clogged after Syringeless Injector
Broken sample vial	2 Sample vials loaded into Syringeless Injector at once or Rotation of Valve with vial partially expelled or injected sample vial	✓ Remove Syringeless Injector from Injection port and clean both the Injection port and disassemble the Syringeless injector ✓ Remove bottom metal seal on injection port and replace
Contamination peaks from previous injection	Contaminated injection port	✓ Sample has fallen into injection port. Insufficient amount of quartz wool ✓ Remove bottom metal seal on injection port and replace ✓ Clean Syringeless Injector