

Troubleshooting Tips & Tricks for your GC Analyzer & CFT Application

7890A/7890B GC

Overview

Definitions

Carrier Gas

• Pressurized gas used to transport the sample through the system.

Detector Support Gases

• Gases needed by specific detectors to operate, i.e. hydrogen and air.

Sample Introduction

• Method of "placing" the sample into the carrier gas stream.

Inlet

• Device that controls the carrier gas flow and sample introduction onto the column.

Column

• Device that separation of the components in the sample.

Detector

• Device that responds to sample components as they elute from the column.

Data Acquisition

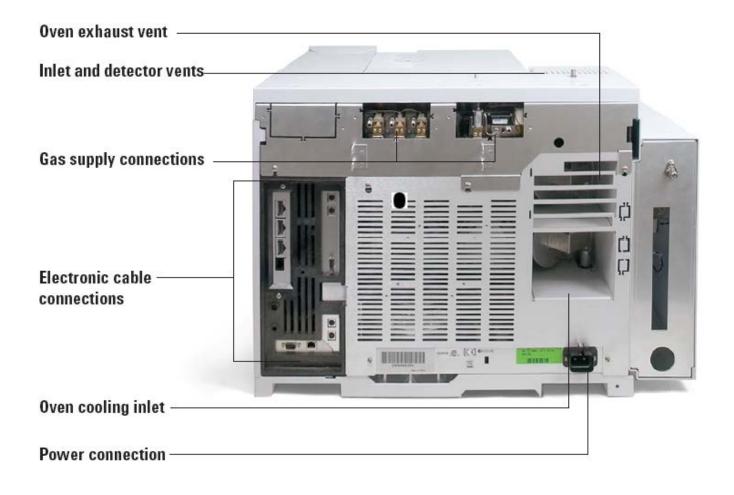
• Collection, conversion, and storage of detector signal used for integration and generation of chromatogram and reports.

Agilent 7890B & 7890 Front View

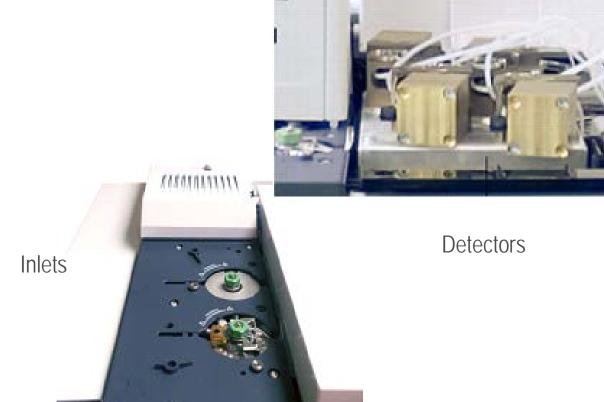




Agilent 7890A & 7890B Rear View



Agilent 7890 Top View



Valve Box, Actuators and Valves



Gas Supply Characteristics

Gases must be chosen with consideration of the type of detector used:

Inert.

Dry.

Pure.

99.9995% pure.

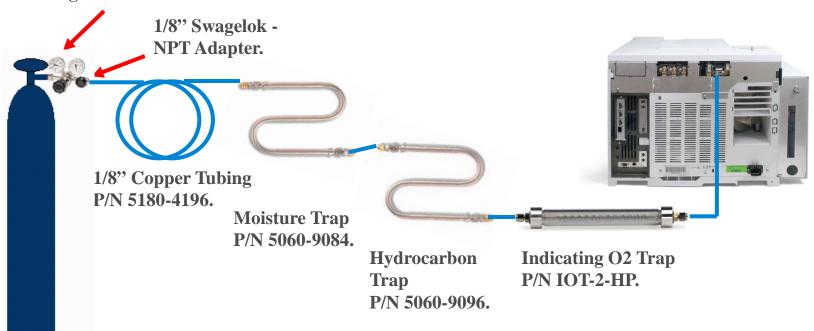
Using Compressed Gases Safely

Obtain safety information from your company's safety department or from your local gas supplier.

Carrier Gas Plumbing



Stainless Steel Diaphragm Tank Regulator.



Gas System Maintenance

Filters/Traps

- Re-condition or replace at manufacture's recommended interval.
- Default replacement interval is every 3 cylinders.

Leaks

- Every 4 to 6 months check all external tubing and fittings for leaks.
- Electronic leak detector recommended for internal and external leak detection.

Liquid leak detection (soap solution) is NOT recommended.





Troubleshooting Process

- 1. Know operation fundamentals.
 - 2. Gather information about problem.
 - 3. Verify symptoms.
 - 4. Verify operational parameters.
 - 5. Isolate the problem.
 - 6. Fix the problem.
 - 7. Verify fix.
 - 8. Document fix.
 - 9. Create PM task?

Isolate the Problem

Inspect parts/assemblies.

Review common recurring fixes:

- Instrument Log book.
- ChemStation Logbook
- Instrument Utilities Logs if configured

Check site/environmental conditions.

7890 Keypad

GC & GC/MS User Manuals and Tools

Theorize a solution.

Isolate problem to assembly/functional area:

- Flow.
- Chemical.
- Electrical.
- Mechanical.
- Operational.



Fix the Problem

Only try one "fix" at a time.

Try quick fixes and easy solutions first.

Perform related maintenance tasks:

- Make sure to reset EMF resource counters on GC.
- Instrument Utilities Software



Verify the Fix

Verify that system performance is restored:

- ChemStation method.
- 7890 Keypad method
- Instrument Utilities

Verify that parts replaced are bad.

Restore system to operational configuration.



Document the Fix

Review troubleshooting and fix actions.

Update instrument logbook.

Update Instrument Utilities Software.



Make a Preventive Maintenance Task

Has the problem occurred before?

Did consumables repair problem?

Should the repair action be made part of scheduled preventive maintenance?

Instrument Utilities Software:

• For scheduled maintenance.

Service Mode:

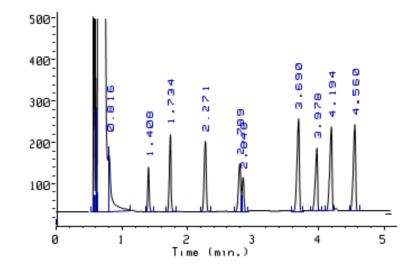
• EMF Resource counters.



Chromatographic Troubleshooting

Use Chromatogram to:

- Evaluate system performance:
 - Detector response.
 - Baseline.
 - Peak resolution.
 - Retention times.
- Evaluate maintenance/repair steps.
- Verify "fix" actions.





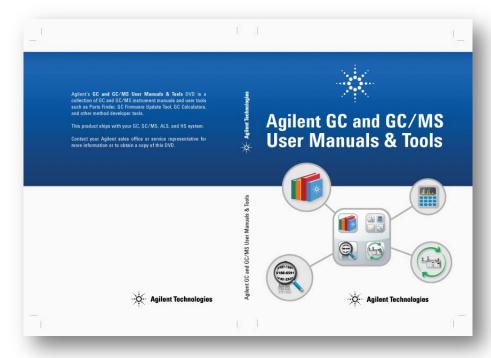
Troubleshooting Tips & Tricks for your GC Analyzer & CFT Application

Diagnostics

Reference Documentation

- GC and GC/MS User Manuals & Tools DVD
 - Documentation
 - Videos
 - Calculator
 - Parts finder
 - Firmware update
 - Instrument utilities
- www.chem.agilent.com









Chromatographic Troubleshooting Tools

- Reference points
 - Chromatogram and report of "standard"
 - Copy of method parameters
- Known good standard samples
 - TCD Checkout PN: 18710-60170
 - FID Checkout PN: 5188-5372
 - ECD Checkout PN: 18713-60040
 - FPD Checkout PN: 5188-5953
 - NPD Checkout PN: 18789-60060
- New syringe
- Spare column
- Spare consumables and frequently replaced parts
 - Ferrules
 - Septa
 - Inlet liners
 - O-rings

Agilent website: www.chem.agilent.com



Common Tools

- Torx drivers (T10 and T20)
- Open-end wrenches (1/4", 1/2", 3/8", 5/8", 5/16", 7/16", 9/16")
- 1/4" hex nut driver
- 7 mm hex nut driver
- Tweezers
- Flashlight
- Volt-ohm meter
- Needle-nose pliers
- Spare fuses (check the instrument manual)

Instrument Tools and Supplies

- Electrostatic discharge (ESD) protective wrist strap
- FID cleaning kit (PN 9301-0985)
- FID flow meter adapter (PN 19231-60660)
- Column cutter
- Electronic leak detector
- Electronic flow meter or soap film bubble meter
- Solvents for cleaning parts
- Light weight cotton gloves

Error Messages and Logbooks

Keypad and Display

GC & GC/MS User Manuals and Tools

Instrument Utilities

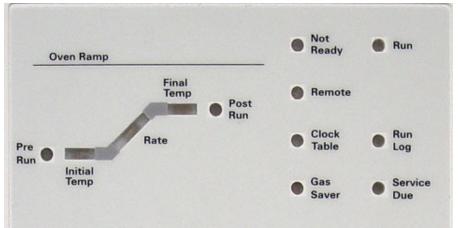
Open Lab ChemStation



Agilent 7890 Keypad







7890B Features – Early Maintenance Feedback

EMF (Counters)

- Injection based
- Time based
- Track usage
- Replace parts before degradation impacts chromatographic results.
- Two Warning Thresholds
 - Service Due
 - Service indicator lights
 - Maintenance log created
 - Service Warning
 - Reminder that the component needs











7890B Features – Resource Conservation

Resource Conservation, the system provides an instrument schedule to conserve resources such as electricity and gases.

- Sleep Method
 - Sets low flows and temperatures
- Wake Method
 - Sets new flows and conditions, typically to restore operating conditions
- Condition Method
 - Sets flows and temperatures high enough to clean out contamination

```
INST SCHEDULE

Go to sleep now.

Edit the Instrument Schedule?<
Edit clock table?

Actual Sepoke
```

```
INST SCHEDULE ( 1 of 2)

Monday: 17:30-c

Go to Sleep.

Actual Support
```



GC and GC/MS User Manuals & Tools

- Parts Finder
 - Interactive software
 - Locate replacements parts for your Agilent instrument
 - Add parts to Parts List and print
 - Order parts
- Firmware Update
 - Easily update firmware to your instrument
- Instrument Utilities
 - Monitor instruments
 - View all the instruments' information
 - Tests and diagnostics
 - Status of instruments on the network



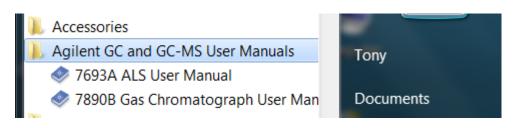


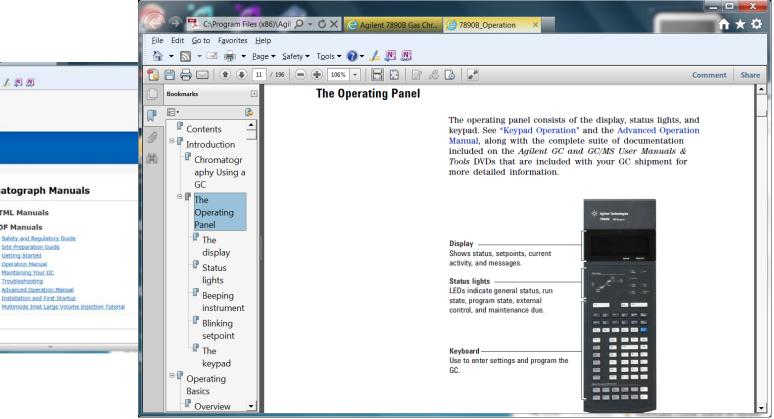


User Manuals









Parts Finder

Easily find parts through search

Selective to specific instrument

 7693A, 7650A, 7890A, 7890B, 5975 MSD, 5977 MSD

Search by component area

Interactive image

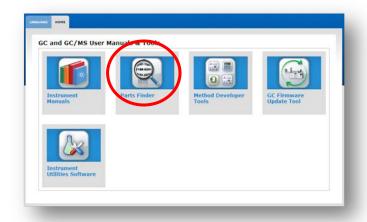
Interactive

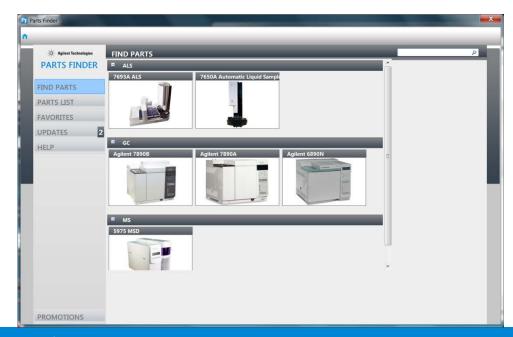
Can access Agilent Store to place an order

Can add to parts list

Can add parts to favorite folder

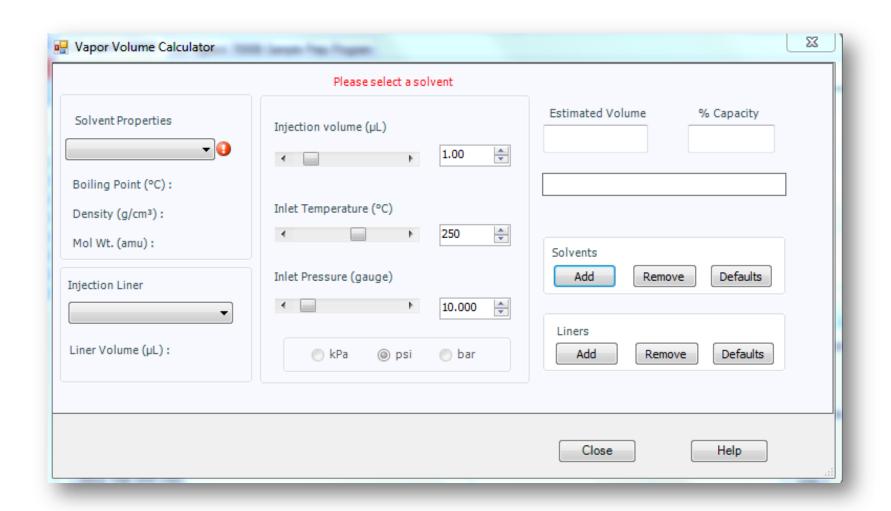
Component breakdown by areas



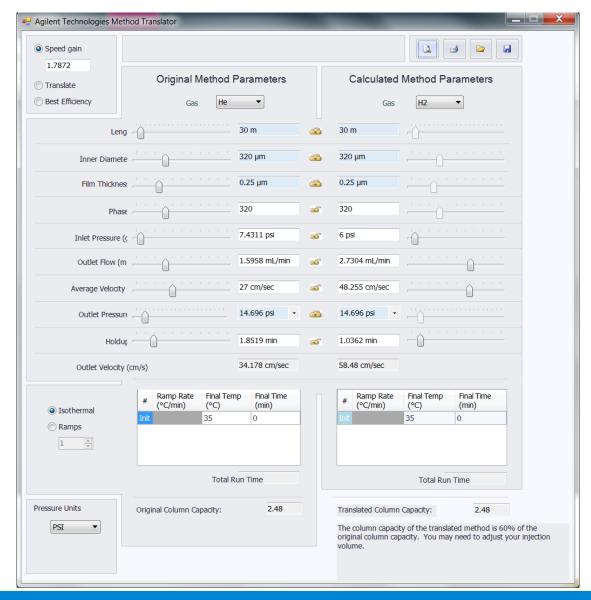




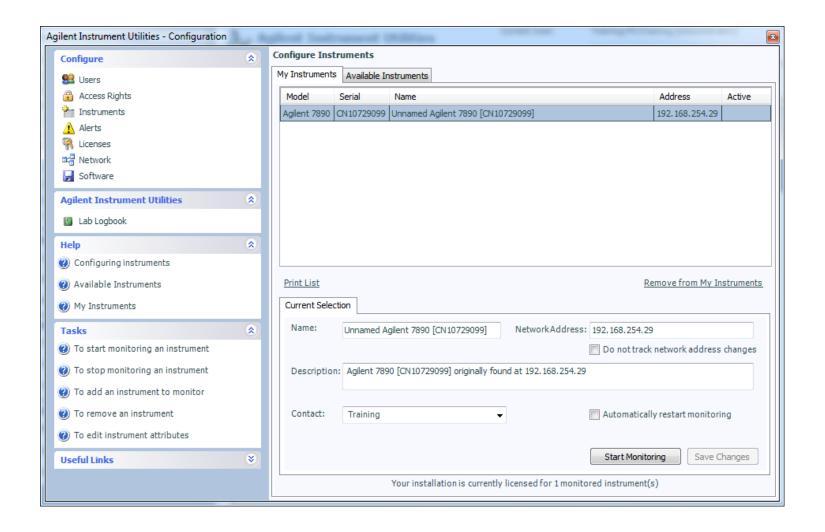
Method Developer Tools – The Vapor Volume Calculator from OpenLAB C01.05



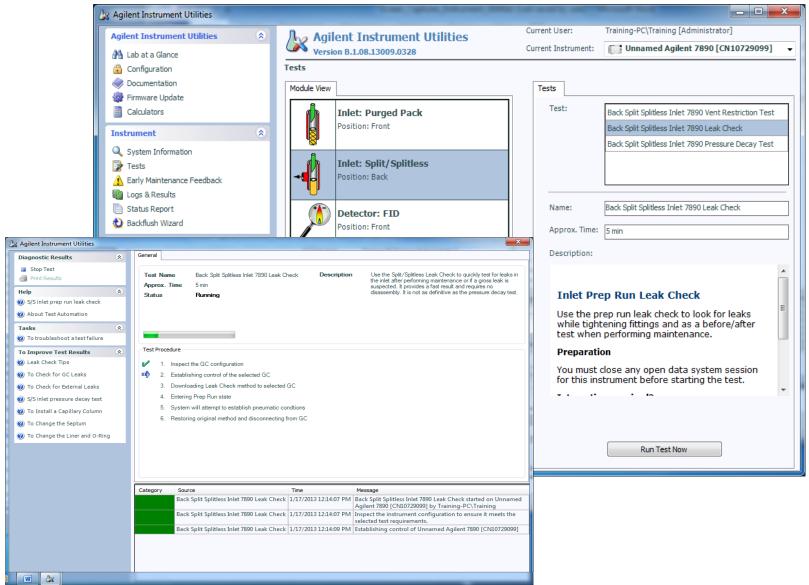
Method Developer Tools – The GC Method Translator from OpenLAB C01.05



Instrument Utilities – Configure

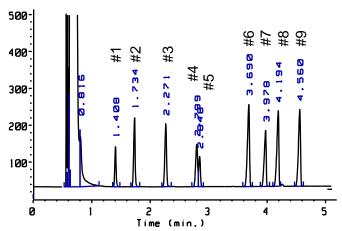


Instrument Utilities – Tests

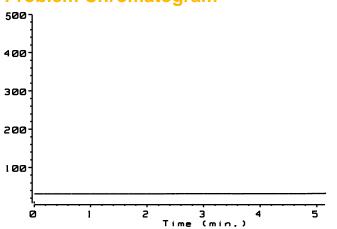


Problem Chromatogram

Typical Chromatogram



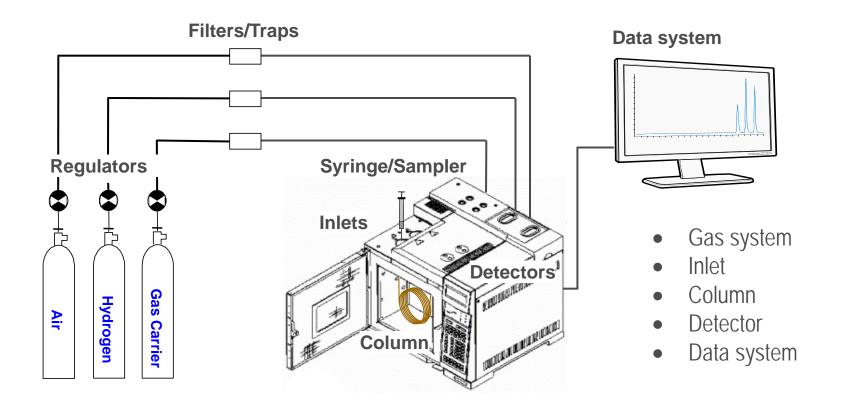
Problem Chromatogram



| Peak | Peak | | |
|------|-------|------|------|
| No. | Width | Area | Type |
| #1 | 0.023 | 1639 | ВВ |
| #2 | 0.027 | 3291 | BB |
| #3 | 0.032 | 3522 | ВВ |
| #4 | 0.037 | 2750 | BV |
| #5 | 0.03 | 1590 | VB |
| #6 | 0.037 | 5473 | BB |
| #7 | 0.034 | 3465 | BB |
| #8 | 0.037 | 4932 | BV |
| #9 | 0.036 | 5020 | BB |
| | | | |



Isolate the Problem to a Functional Area



Know the purpose and function of the major component areas.

Functional Areas

Flow

- improper supply pressures
- leaks

Chemical

- contaminated or "aged" samples/standards
- contaminated supply gases/traps
- contaminated inlet, column, detector

Electrical

cable connections

Mechanical

- auto sampler syringe condition or installation
- vial fill level and sampling depth settings

Operational

- incorrect method loaded
- method parameters modified but not saved

Solve the Problem

Fix the problem.

Verify the fix.

Document fix.

Add to PM schedule if consumable solve problem.

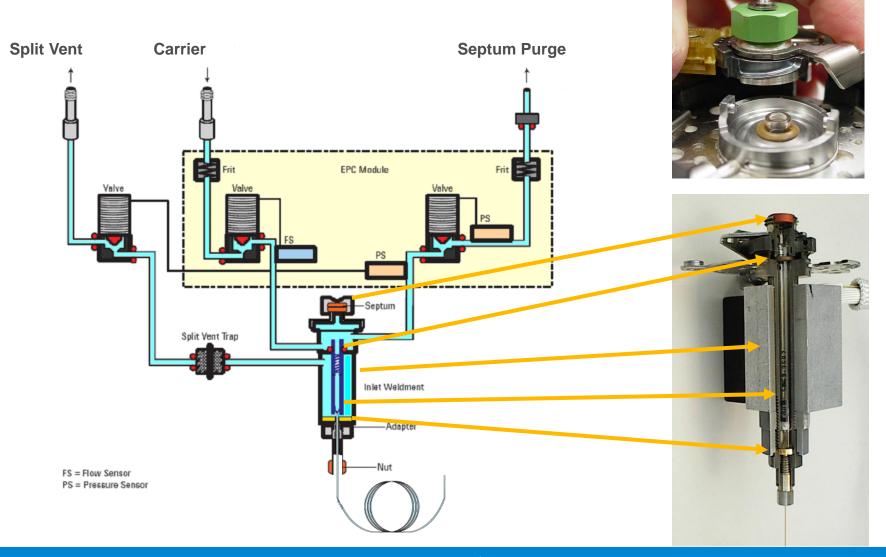
Do not forget

- Reset counters or EMF
 - Open Lab ChemStation
 - 7890B or 7890A Keypad
 - Instrument Utilities
- Schedule maintenance.

SSI Injection Modes

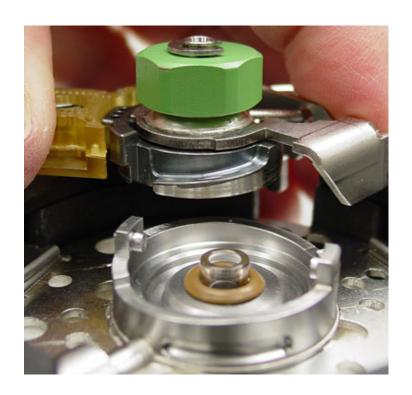
| Injection Mode | Purpose | |
|------------------|--|--|
| Split | Major component analysis. | |
| Pulsed-split | Allows larger injection volume without liner overload and faster transfer to column. | |
| Splitless | Trace component analysis. | |
| Pulsed-splitless | Larger injection volume without liner overload and faster transfer to column. | |

Agilent 7890 Split/Splitless Inlet

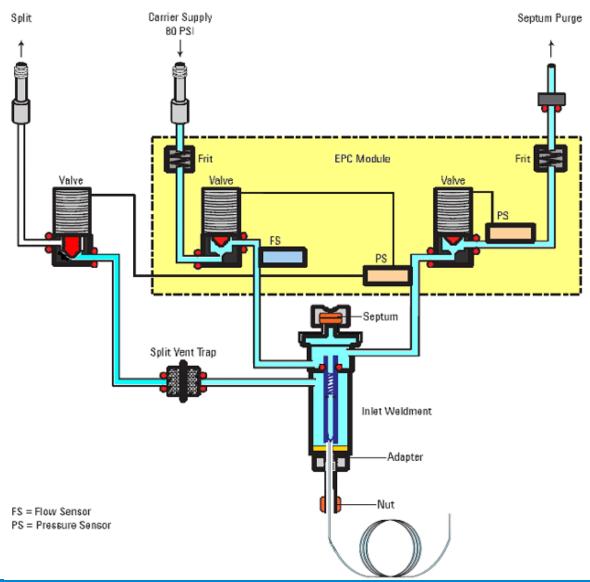


SSI Splitless Mode

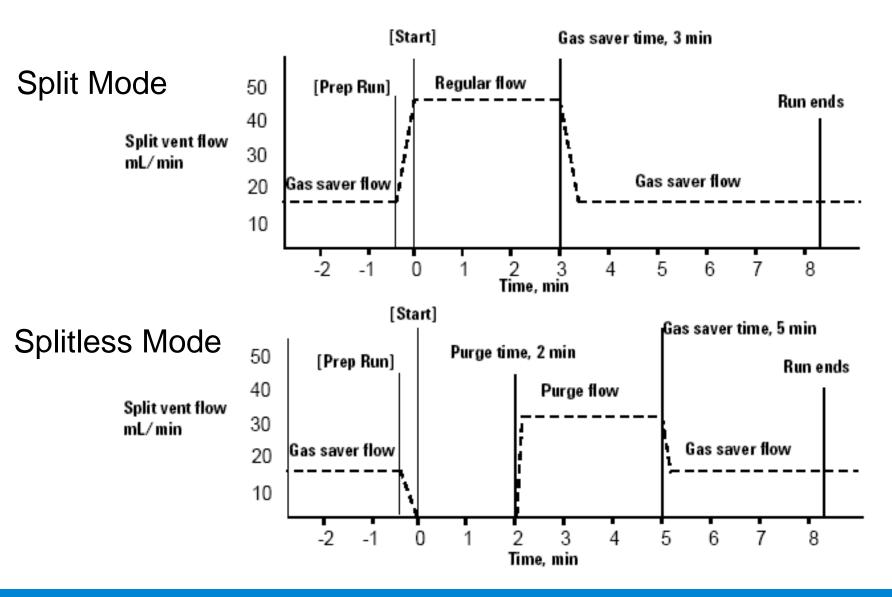
- Trace analysis
- Nearly all of the analytes and solvent enter the column.

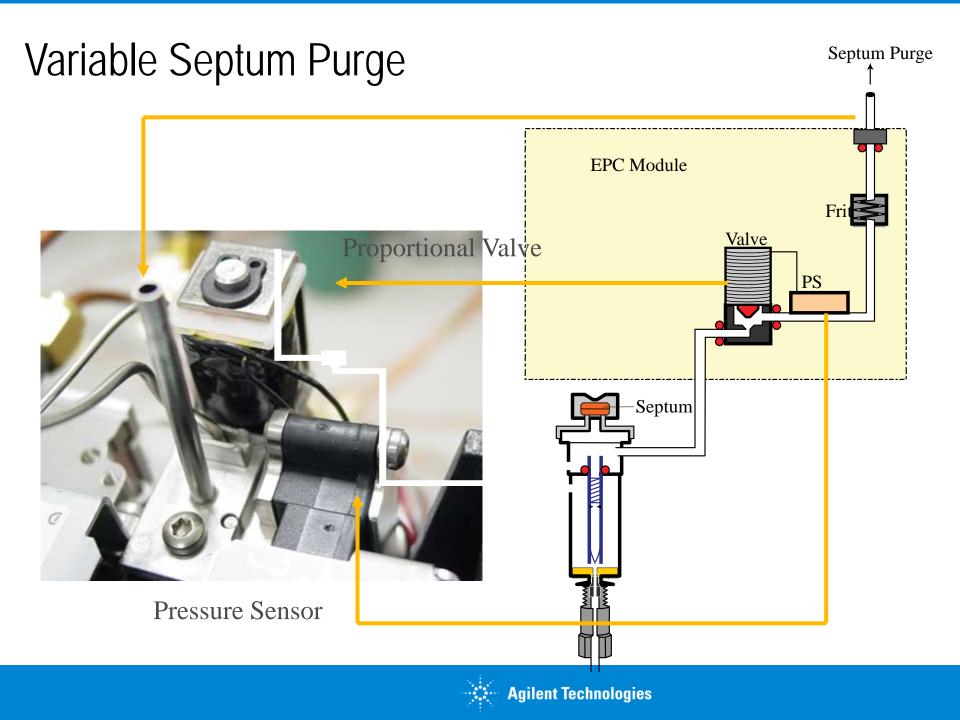


SSI Splitless Mode Flow Diagram

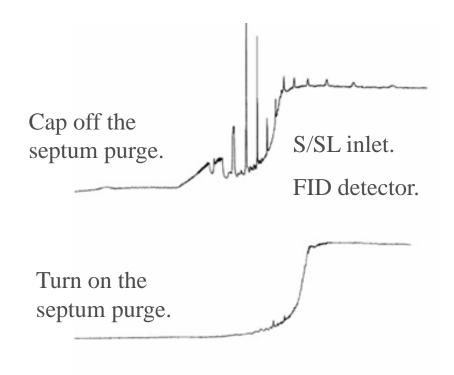


Gas Saver





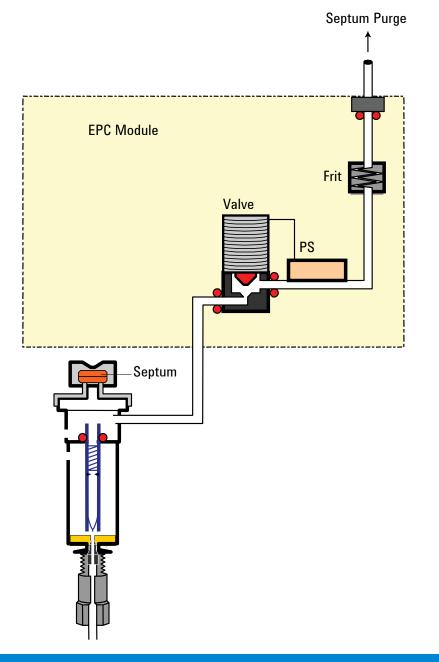
Effects of Septum Purging



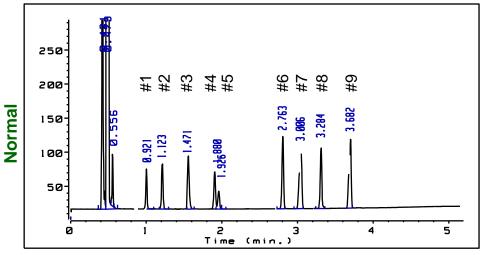
Septum purge prevents septa volatiles from getting into the column.

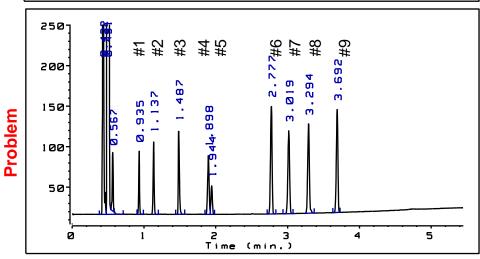
Used in four inlets: SSL_PP_COC

Used in four inlets: SSL, PP, COC, and PTV.



SSI Problem #1

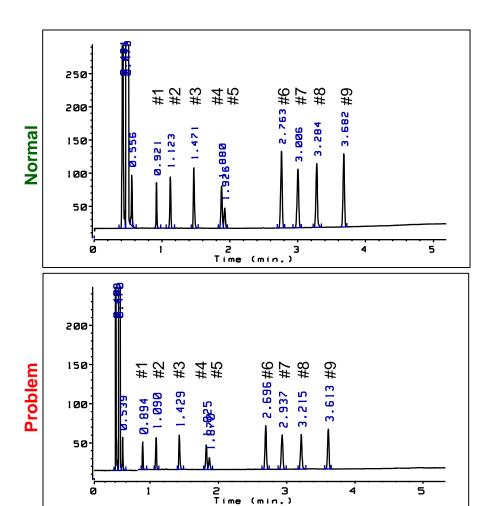




| Peak
No. | Before
Peak
Width | After
Peak
Width | Before
Area | After
Area | Type |
|-------------|-------------------------|------------------------|----------------|---------------|------|
| #1 | 0.024 | 0.025 | 780 | 1099 | BB |
| #2 | 0.024 | 0.026 | 1310 | 1630 | BB |
| #3 | 0.018 | 0.035 | 1820 | 2140 | BB |
| #4 | 0.022 | 0.022 | 1390 | 1899 | BV |
| #5 | 0.022 | 0.022 | 705 | 889 | VB |
| #6 | 0.025 | 0.024 | 2890 | 3680 | BB |
| #7 | 0.027 | 0.027 | 2420 | 3088 | BB |
| #8 | 0.025 | 0.025 | 2430 | 3092 | BB |
| #9 | 0.024 | 0.024 | 2615 | 3256 | BB |

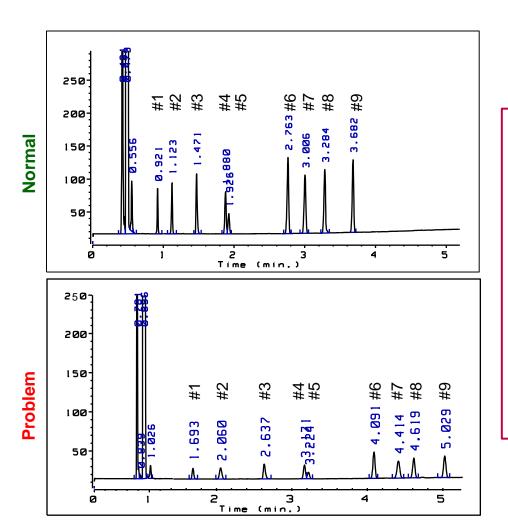
- Manual split injection
- Narrow-bore capillary column
- FID.

SSI Problem #2 – Which Flow Was Adjusted?



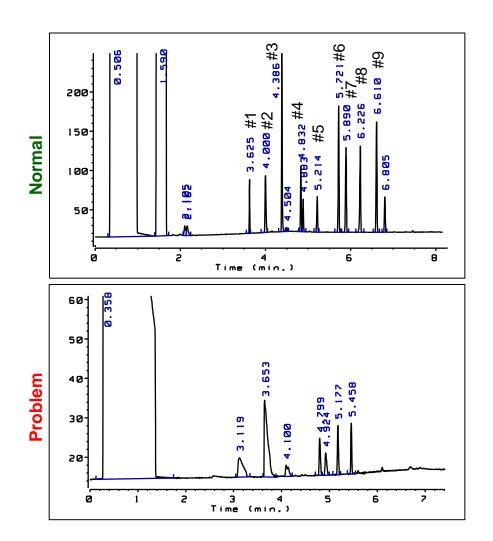
| Peak
No. | Before
Peak
Width | After
Peak
Width | Before
Area | After
Area | Туре |
|-------------|-------------------------|------------------------|----------------|---------------|------|
| #1 | 0.024 | 0.023 | 530 | 242 | BB |
| #2 | 0.024 | 0.023 | 804 | 371 | BB |
| #3 | 0.018 | 0.017 | 1068 | 453 | BB |
| #4 | 0.022 | 0.021 | 909 | 400 | BV |
| #5 | 0.022 | 0.021 | 435 | 195 | VB |
| #6 | 0.025 | 0.023 | 1826 | 790 | BB |
| #7 | 0.027 | 0.026 | 1535 | 698 | BB |
| #8 | 0.025 | 0.024 | 1538 | 657 | BB |
| #9 | 0.024 | 0.023 | 1687 | 713 | BB |

SSI Problem #3 – Which Flow Was Adjusted?



| Peak
No. | Before
Peak
Width | After
Peak
Width | Before
Area | After
Area | Туре |
|-------------|-------------------------|------------------------|----------------|---------------|------|
| #1 | 0.024 | | 530 | | BB |
| #2 | 0.024 | | 804 | | BB |
| #3 | 0.018 | 0.031 | 1068 | 332 | BB |
| #4 | 0.022 | 0.028 | 909 | 238 | BV |
| #5 | 0.022 | | 435 | | VB |
| #6 | 0.025 | 0.031 | 1826 | 585 | BB |
| #7 | 0.027 | 0.040 | 1535 | 505 | BB |
| #8 | 0.025 | 0.033 | 1538 | 465 | BB |
| #9 | 0.024 | 0.034 | 1687 | 531 | BB |

SSI Problem #4



| Peak
No. | Peak
Width | Area | Type |
|-------------|---------------|------|------|
| #1 | 0.021 | 551 | BV |
| #2 | 0.026 | 1214 | BB |
| #3 | 0.018 | 2521 | BV |
| #4 | 0.020 | 1037 | BV |
| #5 | 0.019 | 511 | VB |
| #6 | 0.022 | 2253 | BB |
| #7 | 0.025 | 1737 | BB |
| #8 | 0.024 | 1703 | BB |
| #9 | 0.023 | 2104 | BB |

SSI Septa and Merlin Microseal



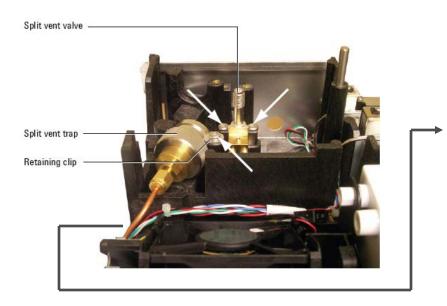
- High Temp Analysis.
- Low Bleed.
- Lower Coring.
- Packaged in glass to maintain purity.
- 11-mm diameter.

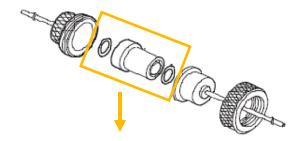


- Longer life alternative.
- Duckbill design.
- Inlet pressures: 3-100 psi.
- Inlet temperatures: 100-300°C.

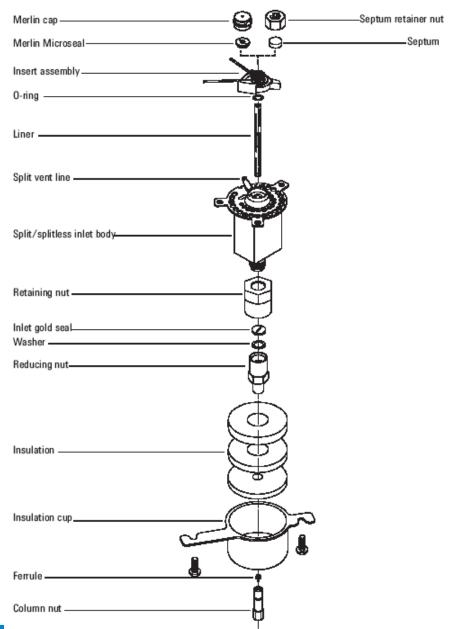
Merlin microseal requires 23 gauge syringe needle tapered tip.

SSI Assembly



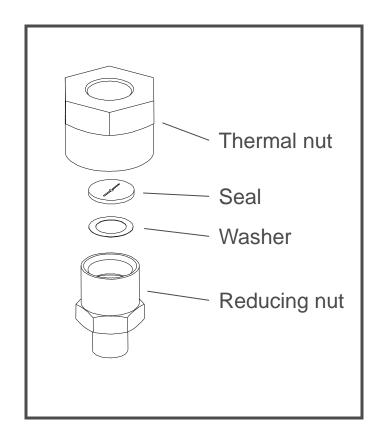


Replacement Filter Cartridge Kit P/N G1544-80530





SSI Seals





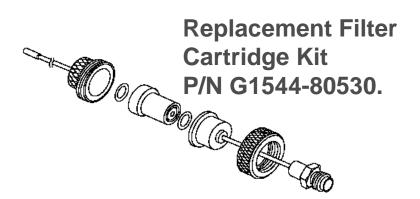
Gold seal (original)



Gold seal (cross)

| 18740-20885 | Gold-plated seal |
|-------------|--------------------------|
| 5182-9652 | Gold-plated (cross) seal |
| 18740-20880 | Stainless steel seal |
| 5061 - 5869 | Washer |

Agilent 7890 – Split/Splitless Capillary Inlet





GLASS POSITIONING BEAD



Deactivated Split Liner P/N 5183-4647



High Split Flow Gold Seal. P/N 5182-9652 - Use with liners that introduce restriction.

Split Liner

| Liner | Description | Volume | Mode | Deactivated | Part Number |
|-------|---|--------|---------------------------|-------------|-------------|
| | Low Pressure Drop
– Positioning Bead | 870 μL | Split – Fast
Injection | Yes | 5183-4647 |
| | 4mm ID, Glass Wool | 990 µL | Split – Fast
Injection | No | 19251-60540 |
| | Empty Pin & Cup | 800 µL | Split – Manual
Only | No | 18740-80190 |
| | Packed Pin & Cup | 800 µL | Split – Manual
Only | No | 18740-60840 |

Splitless Liner

| Liner | Description | Volume | Mode | Deactivated | Part Number |
|----------------------------|----------------------------|-----------------|--|-------------|-------------|
| | Single Taper Glass
Wool | 900 uL | Splitless | Yes | 5062-3587 |
| \leftarrow | Single Taper | 900 uL | Splitless | Yes | 5181-3316 |
| \leftarrow \rightarrow | Dual Taper | 800 uL | Splitless | Yes | 5181-3315 |
| | 2 mm Quartz | 250 uL | Splitless | No | 18740-80220 |
| | 2 mm Quartz | 250 uL | Splitless | Yes | 5181-8818 |
| | 1.5 mm | 140 uL | Direct
Inject, Purge
&Trap,
Headspace | No | 18740-80200 |
| | Single Taper Glass
Wool | 900 uL | Splitless | Yes | 5062-3587 |
| \vdash | Single Taper | 900 uL | Splitless | Yes | 5181-3316 |
| \vdash | 4 mm Single Taper | Direct column c | onnect | Yes | G1544-80700 |
| \leftarrow | 4 mm Dual Taper | Direct column c | onnect | Yes | G1544-80730 |

SSI Liner Volume versus Sample Volume

| Solvent | Density | Mol. Wt. | Exp. Ratio |
|--------------------|---------|----------|------------|
| Isooctane | 0.69 | 114 | 138:1 |
| Hexane | 0.66 | 86 | 174:1 |
| Pentane | 0.62 | 72 | 198:1 |
| Ethyl acetate | 0.90 | 88 | 233:1 |
| Chloroform | 1.48 | 119 | 284:1 |
| Methylene Chloride | 1.33 | 85 | 356:1 |
| Methanol | 0.79 | 32 | 563:1 |
| Water | 1.00 | 18 | 1261:1 |

Injection temperature = 250° C. Column head pressure = 13 psi (90 kPa).

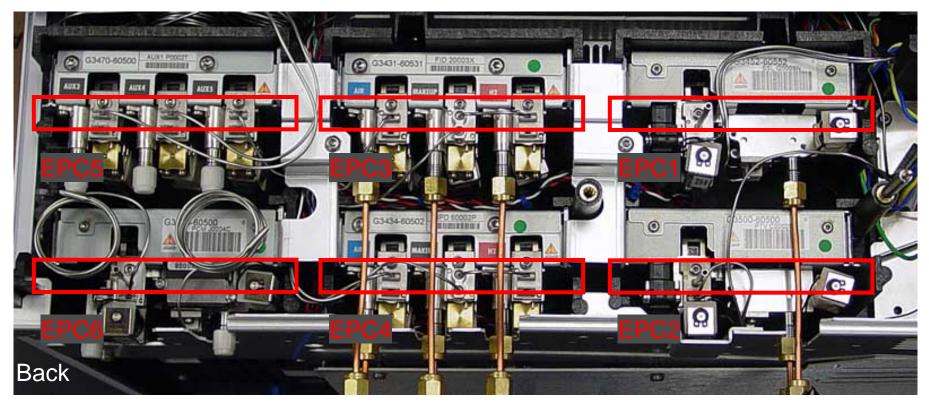


EPC Modules

Auxiliary

Front Detector

Front Inlet



PCM

Back Detector

Back Inlet

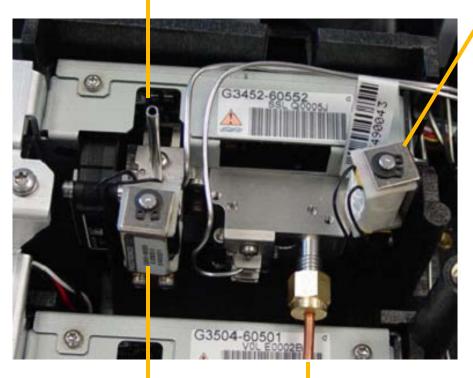


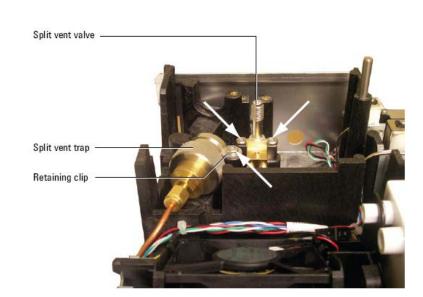
SSI EPC Module

Septum Purge Vent





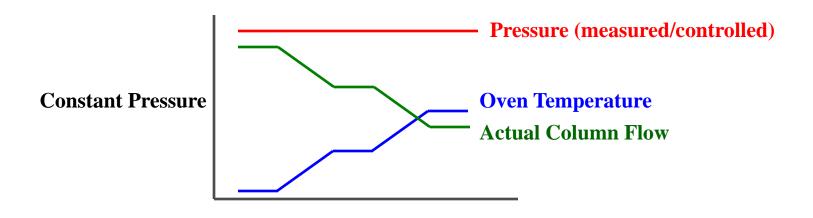


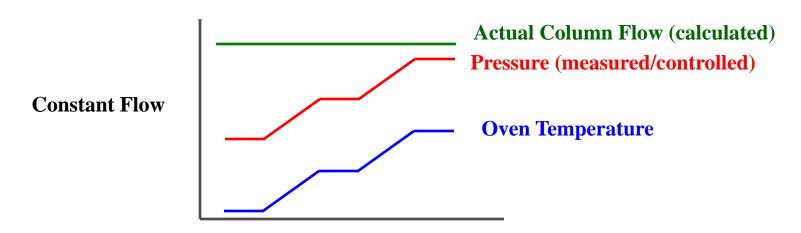


Carrier Gas Inlet

Septum Purge Valve

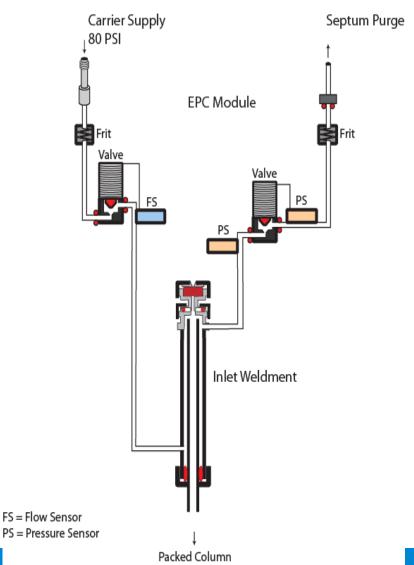
EPC Module Constant Pressure versus Constant Flow





PPI Flow-Controlled Flow Diagram

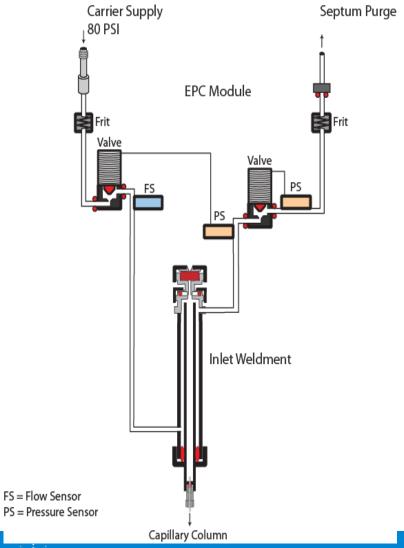
Flow-controlled mode recommended for packed columns.



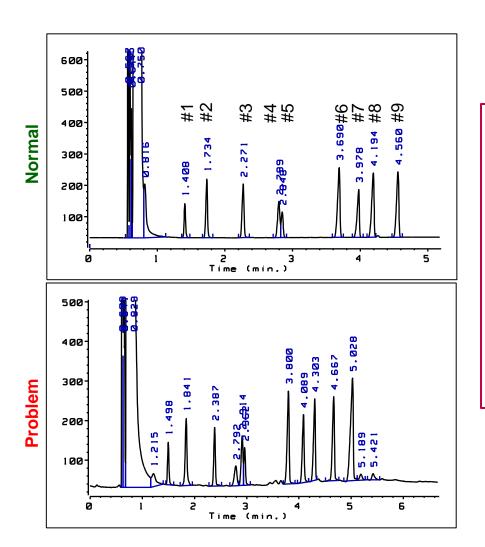
PPI Pressure-Controlled Flow Diagram

Pressure-controlled mode recommended for capillary columns.

Packed Inlet

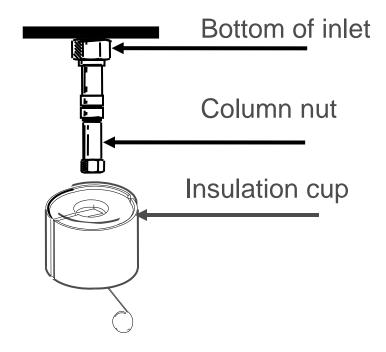


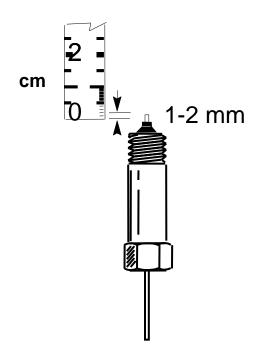
PPI Problem #1



| Peak
No. | Before
Peak
Width | After
Peak
Width | Before
Area | After
Area | Туре |
|-------------|-------------------------|------------------------|----------------|---------------|------|
| #1 | 0.023 | 0.029 | 1639 | 2009 | BB |
| #2 | 0.027 | 0.039 | 3291 | 4394 | BB |
| #3 | 0.032 | 0.035 | 3522 | 3363 | BB |
| #4 | 0.037 | 0.040 | 2750 | 3252 | BV |
| #5 | 0.03 | 0.033 | 1590 | 2141 | VB |
| #6 | 0.037 | 0.039 | 5473 | 6168 | BB |
| #7 | 0.034 | 0.040 | 3465 | 4516 | BB |
| #8 | 0.037 | 0.039 | 4932 | 5568 | BV |
| #9 | 0.036 | 0.044 | 5020 | 6049 | BB |

PPI Column Installation





Typical Injection Port Problems

- Discrimination
 - Needle
 - Inlet
- Sample decomposition
- Incorrect temperature
- Contamination
- Blockage
- Leaks
- Flashback

Detecting Leaks

- EPC "chirp" alarm and shutdown
- Electronic leak detector
- Pressure test
- Chromatographic changes
 - Retention times
 - Area counts
- Methanol

Soap-film leak detection fluids

Routine Injection Port Maintenance

- Change septum.
- Clean or replace syringe.
- Perform pressure decay or leak test and repair any leaks.
- Clean or replace liners/inserts.
- Replace O-rings.
- Clean or replace seal and washer (SSI).

