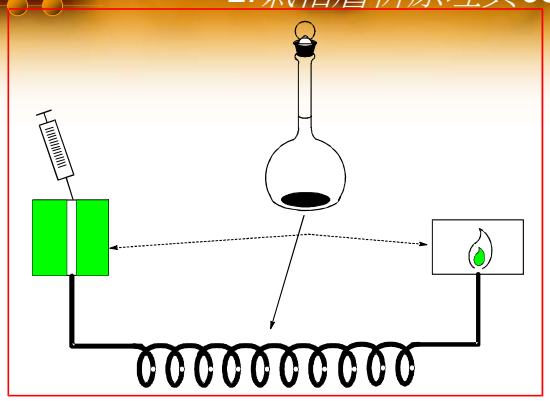
6820 GC 氣相層析儀教育訓練

教育訓練內容

- 1.氣相層析原理與6820GC簡介
- 2.注射口(INLET)
- 2-1分流/非分流注射口(split/splitless inlet)
- 2-2填充式注射口(purged packed inlet)
- 3.分析管柱
- 4.偵測器
- 4-1火焰離子偵測器(FID)
- 4-2電子捕捉偵測器(u-ECD)
- 5.儀器操作
- 6.日常維護與保養

1. 氣相層析原理與6820GC簡介



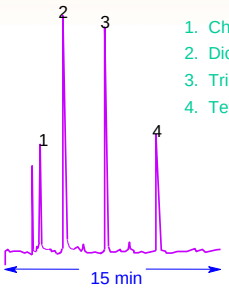
The sample determines the instrument configuration, i.e.:

- ***Type of Carrier Gas***
- ***Type of Sample Inlet***
- ***Type of Column***
- ***Type of Detector***
- ***Type of Data Acquisition***

6820 GC 外觀

Applications

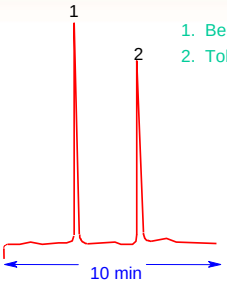
Chlorinated Hydrocarbons in Water by Headspace Analysis



1. Chloromethane
2. Dichloromethane
3. Trichloromethane
4. Tetrachloromethane

Column: HP-1 (Cross-Linked Methyl Silicone)
30 m x 0.53 mm x 2.65 µm film
(Part No. 19095Z-123)
Carrier: Helium, 5.2 psi
Oven: 40 C °
Injection: 1cc, splitless
Detector: FID

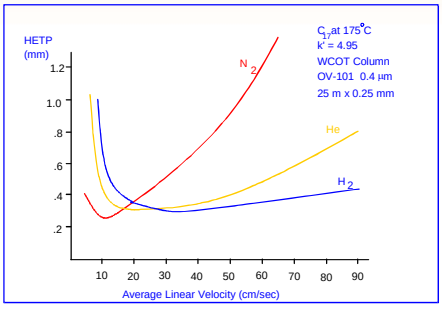
Aromatic Hydrocarbons in Water by Headspace Analysis



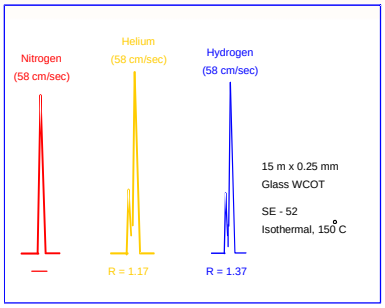
1. Benzene
2. Toluene

Column: HP-1 (Cross-Linked Methyl Silicone)
30 m x 0.53 mm x 2.65 µm film
(Part No. 19095Z-123)
Carrier: Helium, 5.2 psi
Oven: 60 C °
Injection: 1 cc, split 5:1
Detector: FID

Type of Carrier Gas Effect on Efficiency and Resolution



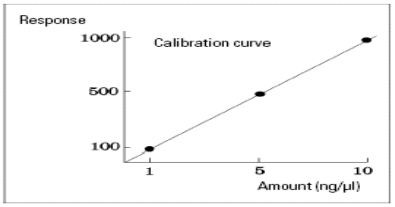
Efficiency curves for a 25 m x 0.25 mm id WCOT column with 0.4 um of OV-101.



Effect of carrier gas on the resolution of n-heptadecane and pristane.

calibration

Multilevel Calibration Curve With Three Levels

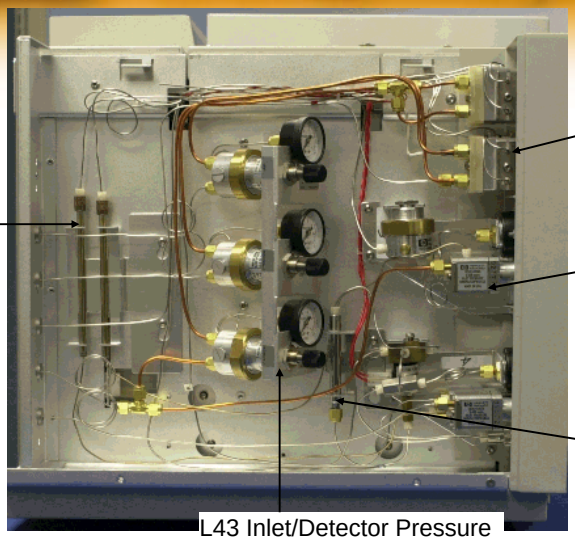


The corresponding calibration table, which is the tabulation of the information used to generate this curve, might look similar to the one shown in Table 9.

Calibration Table

Level	Amount (ng/ml)	Response (area counts)
2	5	500
3	10	1000

6820 流量控制系統



Carrier Chemical Filters

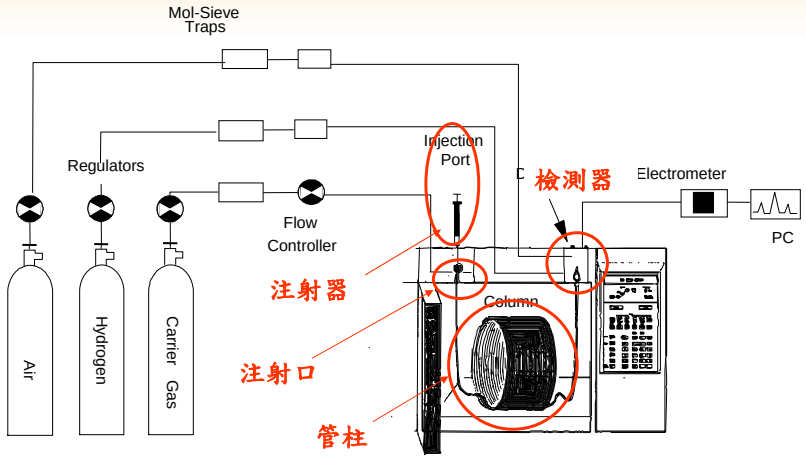
Detector Flow Blocks

Inlet Flow/Pressure Controls

New Split Vent Trap

L43 Inlet/Detector Pressure Controls - Standard

氣相層析系統組成



儀器狀態

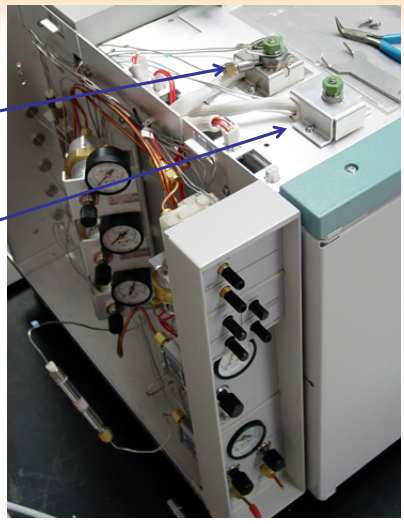
- *6890 style electronics*
- *New keypad and display*

2. INLET 注射口

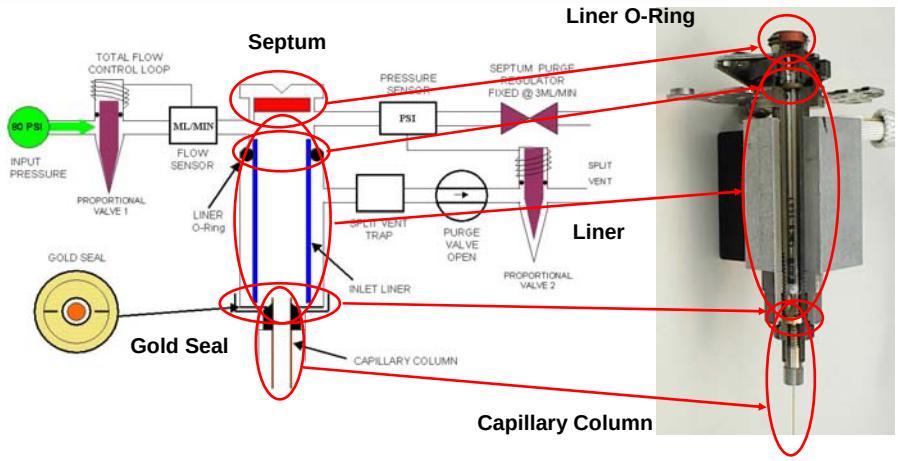
- *導入樣品*
- ***sample*** *控制進樣量*
- *保持* ***sample*** *原貌,不使降解或反應*
- *充分使樣品汽化*

6820 Inlets type

- ✓ *Inlet Type*
  - – Capillary Split/Splitless
  - – Purged Packed
- ✓ *Manual pneumatics*
- ✓ *6890 style heater/ sensor*

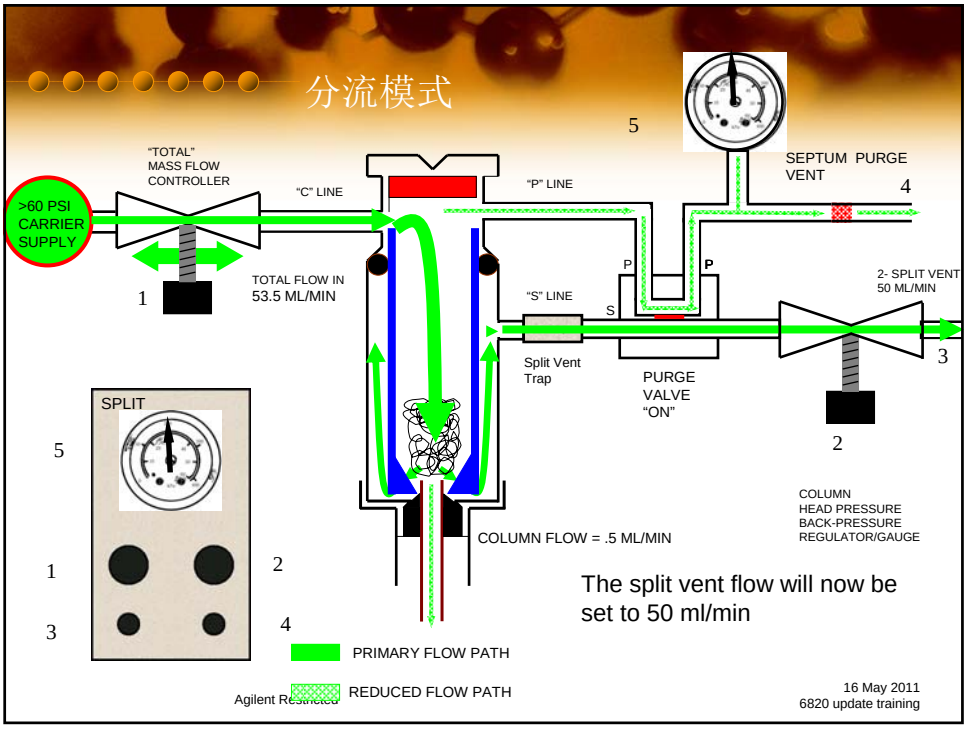


2-1 分流非分流注射口(Split/Splitless Inlet)



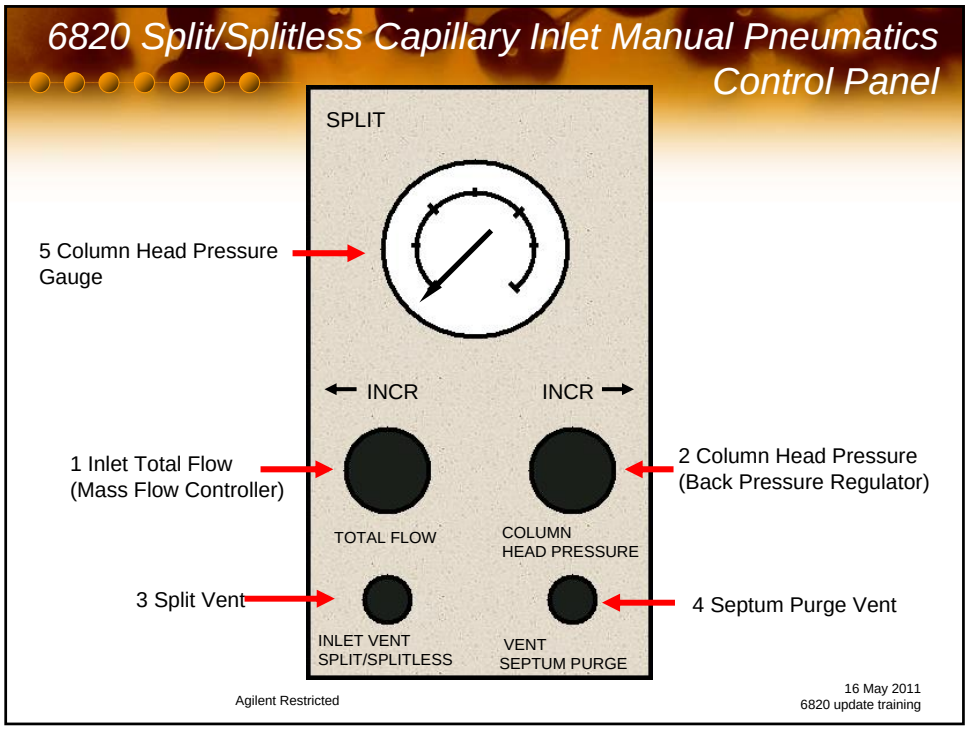
5/4/00

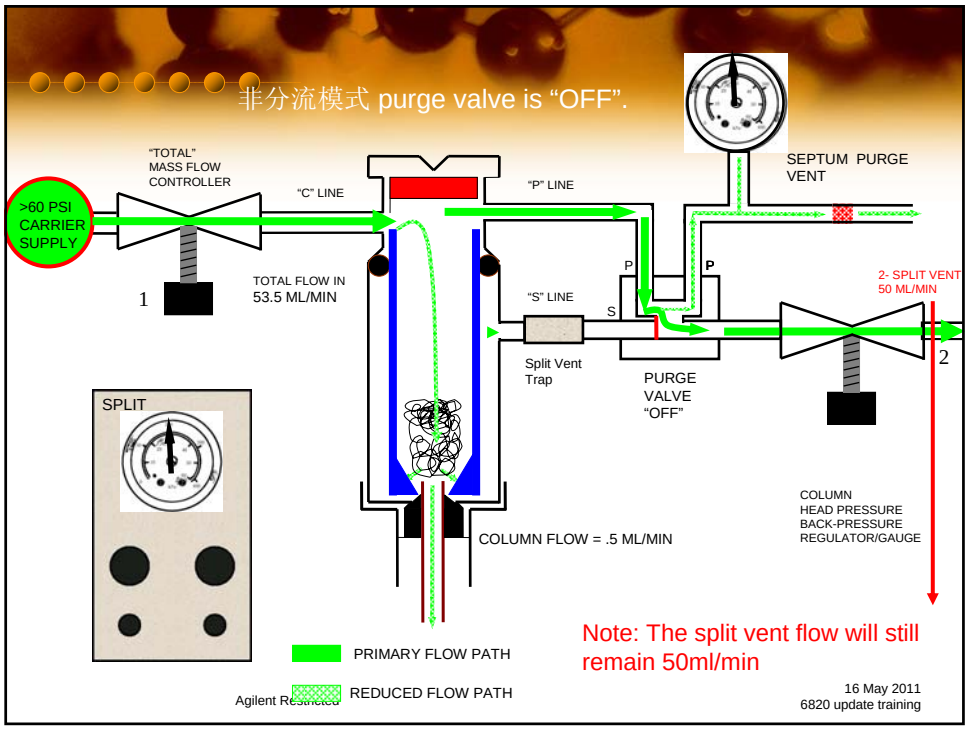
分流模式

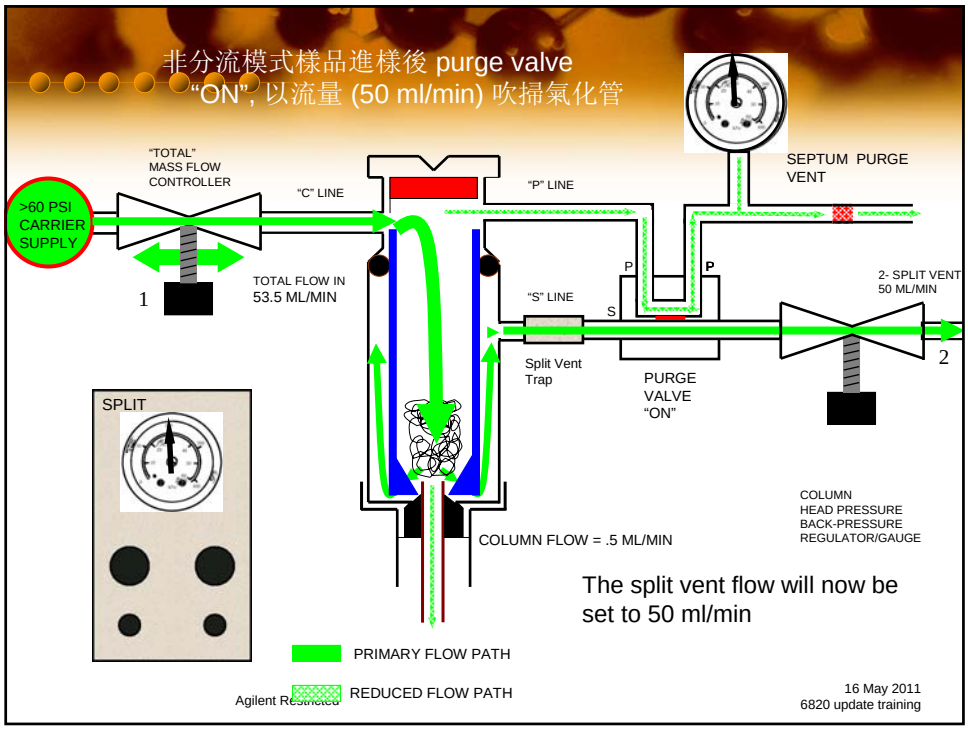


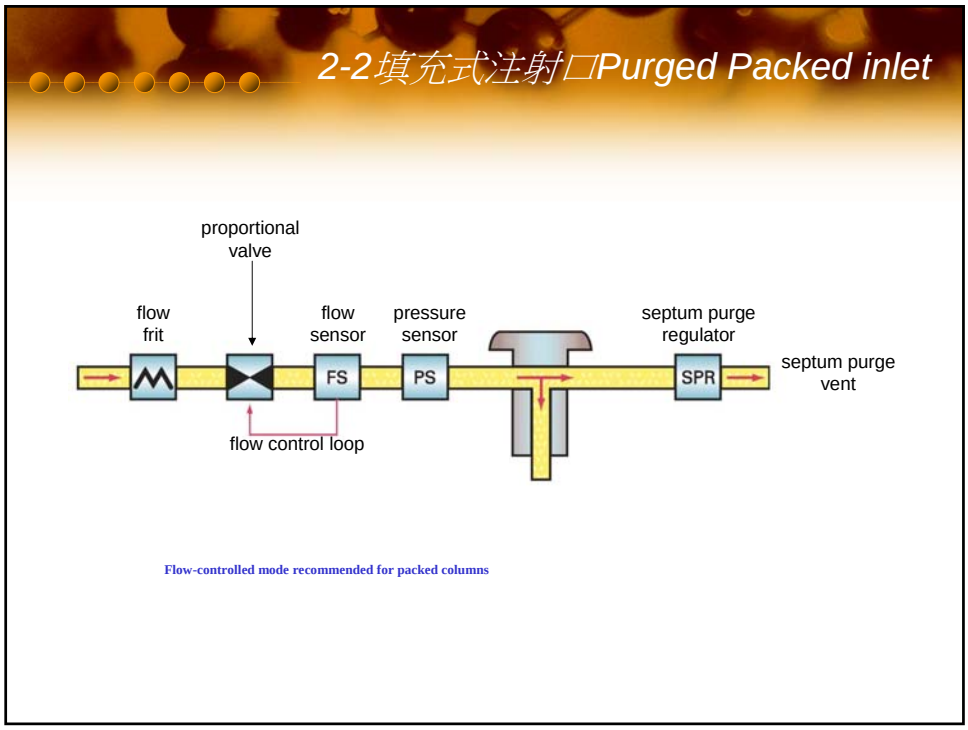
Agilent Restricted

16 May 2011
6820 update training

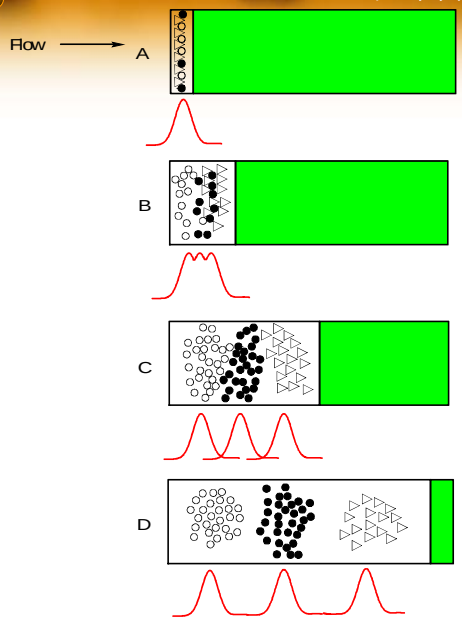

6820 Split/Splitless Capillary Inlet Manual Pneumatics Control Panel
SPLIT
5 Column Head Pressure Gauge
INCR
INCR
1 Inlet Total Flow (Mass Flow Controller)
2 Column Head Pressure (Back Pressure Regulator)
TOTAL FLOW
COLUMN HEAD PRESSURE
3 Split Vent
4 Septum Purge Vent
INLET VENT SPLIT/SPLITLESS
VENT SEPTUM PURGE
Agilent Restricted
16 May 2011
6820 update training


非分流模式 purge valve is “OFF”.
“TOTAL” MASS FLOW CONTROLLER
>60 PSI CARRIER SUPPLY
“C” LINE
“P” LINE
SEPTUM PURGE VENT
1
TOTAL FLOW IN 53.5 ML/MIN
“S” LINE
P
S
2- SPLIT VENT 50 ML/MIN
Split Vent Trap
PURGE VALVE “OFF”
2
SPLIT
COLUMN FLOW = .5 ML/MIN
COLUMN HEAD PRESSURE BACK-PRESSURE REGULATOR/GAUGE
PRIMARY FLOW PATH
REDUCED FLOW PATH
Note: The split vent flow will still remain 50ml/min
16 May 2011
6820 update training

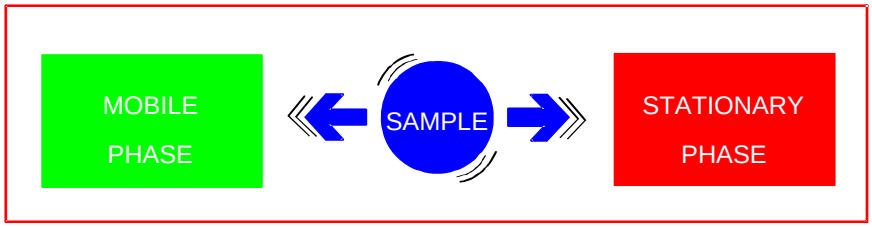

非分流模式樣品進樣後 purge valve
“ON”, 以流量 (50 ml/min) 吹掃氣化管
“TOTAL”
MASS FLOW
CONTROLLER
>60 PSI
CARRIER
SUPPLY
“C” LINE
“P” LINE
SEPTUM PURGE
VENT
TOTAL FLOW IN
53.5 ML/MIN
1
“S” LINE
P
P
S
2- SPLIT VENT
50 ML/MIN
2
Split Vent
Trap
PURGE
VALVE
“ON”
SPLIT
COLUMN
HEAD PRESSURE
BACK-PRESSURE
REGULATOR/GAUGE
COLUMN FLOW = .5 ML/MIN
The split vent flow will now be
set to 50 ml/min
PRIMARY FLOW PATH
REDUCED FLOW PATH
16 May 2011
6820 update training


2-2填充式注射口Purged Packed inlet
proportional
valve
flow
frit
flow
sensor
pressure
sensor
septum purge
regulator
septum purge
vent
FS
PS
SPR
flow control loop
Flow-controlled mode recommended for packed columns

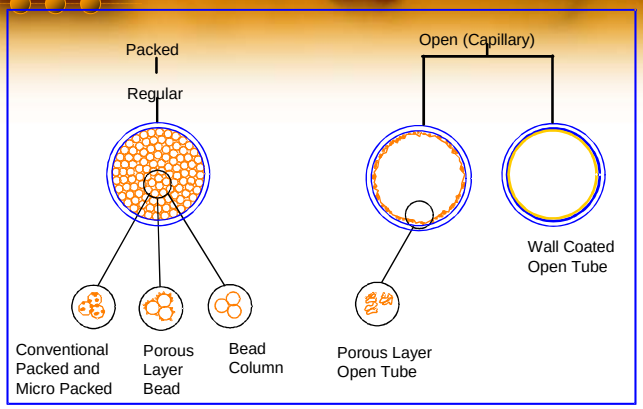
3分析管柱分離原理



Separation is a Partitioning Process



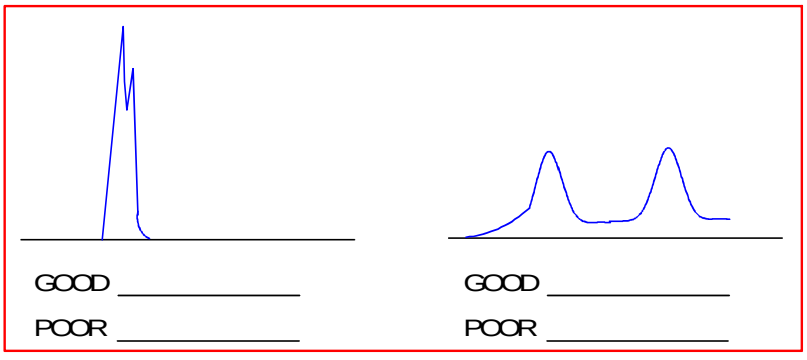
Column Types



	PACKED	SERIES 530	WSCOT (wide)	WCOT (narrow)
LENGTH (meters)	.5-10	5-100	5-100	5-100
I.D. (mm)	2-4	.530	.3-.75	.1-.25
FLOW RATE (ml/min)	10-60	4-30	1-30	0.3-1.0
PRESSURE DROP (psi)	10-90	1-20	1-40	5-90
SAMPLE CAPACITY	100ng/peak		100 ng/peak	50 ng/peak

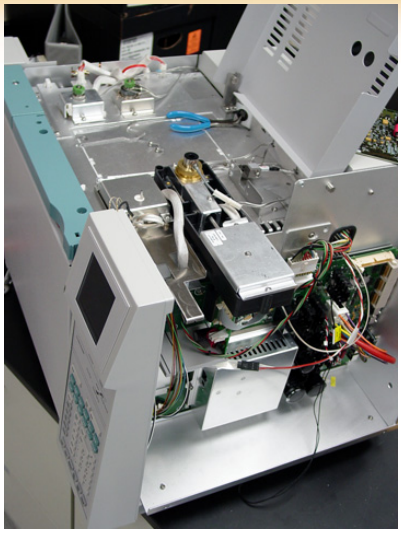
Column Separation Characteristics

- ***Efficiency:***
  Ability of the column to produce sharp peaks
- ***Resolution:***
  Ability of the column to separate two peaks from each other
- ***Selectivity:***
  Ability of the column to determine chemical and/or physical difference in two peaks

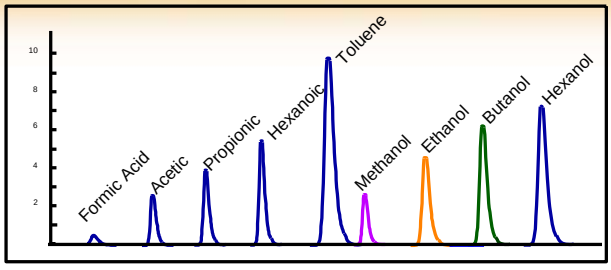


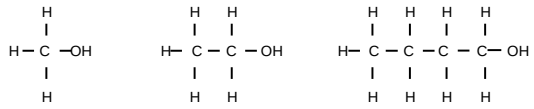
4偵測器Detectors

- ✓ ***偵測器detectors***
  - FID
  - Micro ECD
- ✓ ***Manual pneumatics***



4-1火焰離子偵測器 FID

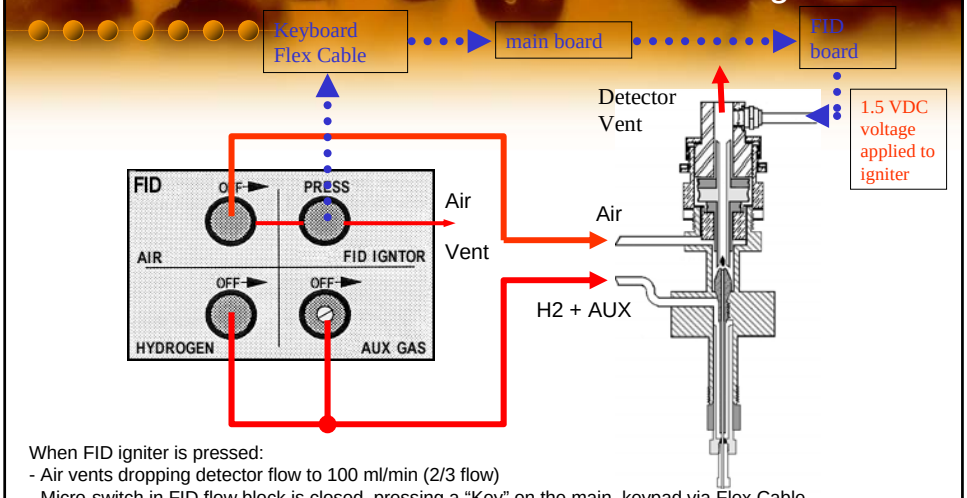




Methanol Ethanol Butanol

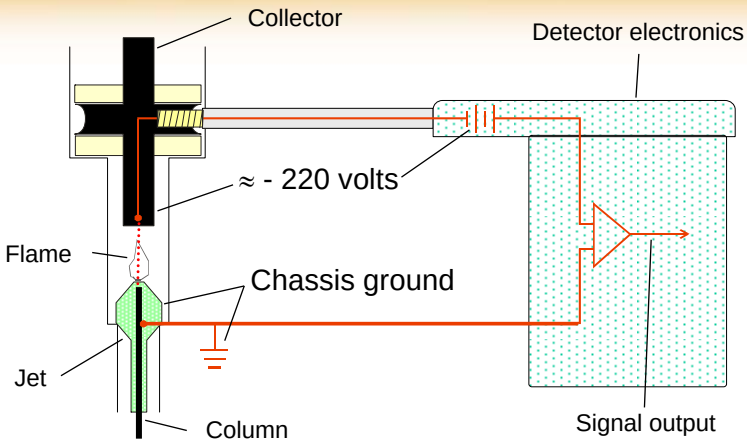
Response is proportional to the number of carbon-hydrogen bonds.

6820 FID Flow Diagram



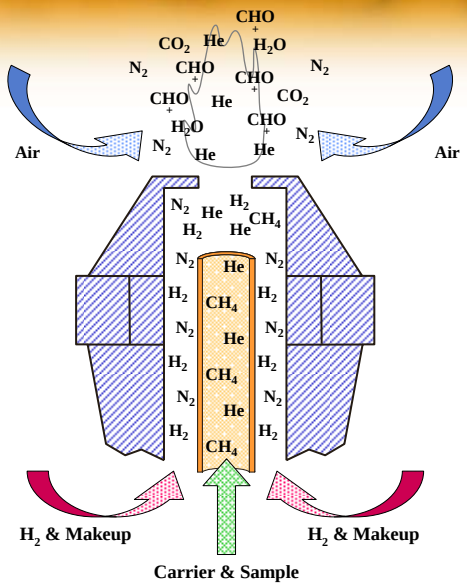
When FID igniter is pressed:
- Air vents dropping detector flow to 100 ml/min (2/3 flow)
- Micro-switch in FID flow block is closed, pressing a “Key” on the main keypad via Flex Cable
- The “Key-press” is detected by firmware, which activates the ignitor circuit on the FID interface board
- FID ignitor circuit applies 1.5 VDC to the glow-plug
- Flame lights with a faint “pop”
- Button is released, restoring air flow and un-pressing the keypad button which is sensed by firmware, turning off the ignitor voltage

FID Functional Schematic



FID Response/Selectivity

Air	FID air supply
CH_4	Methane in sample
CHO^+	Methyl cations
H_2	FID hydrogen
He	Carrier gas
H_2O	Water
N_2	Makeup gas

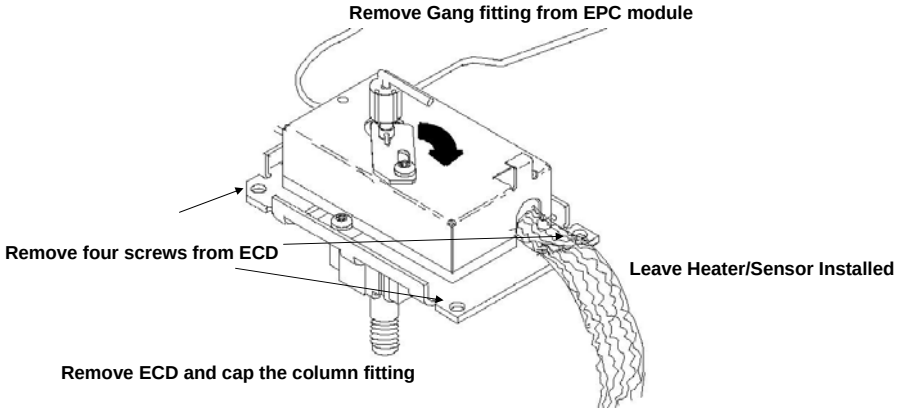


FID Undetectable Compounds

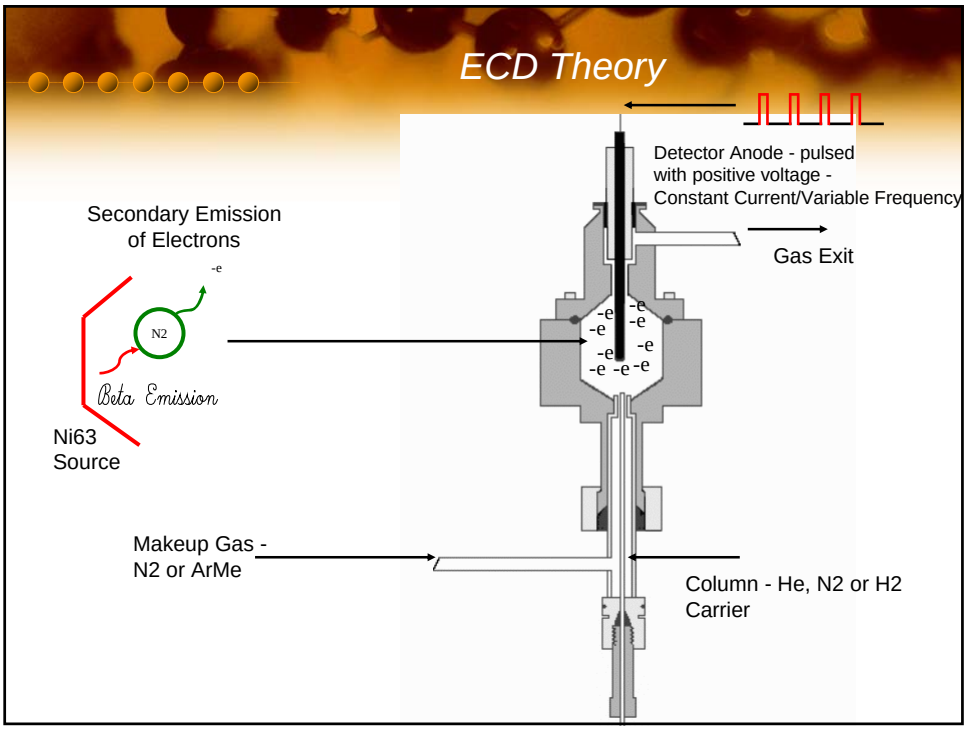
Compounds that yield little or no response from an FID		
Rare Gases	NH_3	CS_2
Nitrogen Oxides	H_2	COS
Silicon Halides	CO	O_2
H_2O	CO_2	N_2
Perhalogenated Compounds	HCOH	HCOOH

4-2電子捕捉偵測器 Electron Capture Detectors

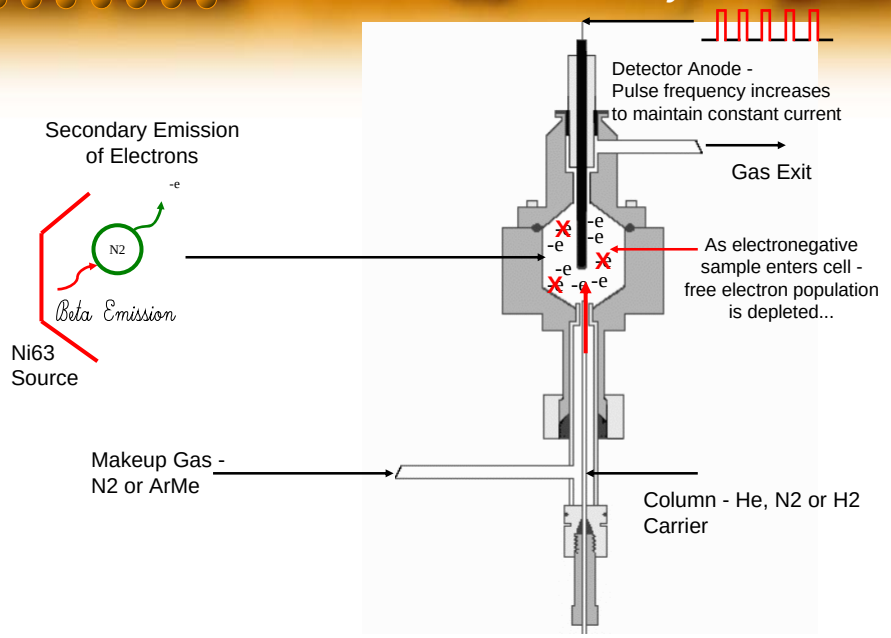
To ship the ECD:



ECD Theory

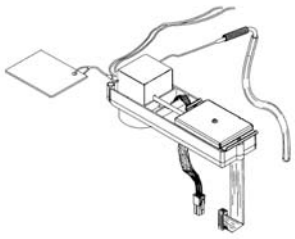


ECD Theory



6820 Electron Capture Detector - Display Menus

Front/Back Detector



FRONT/BACK DET (ECD)
Temp
255 300<
Output
157

Output
in hertz

Setting the electrometer to “OFF”
Turns off the pulser to the anode

CONFIG FRONT/BACK DET
Electrometer
Off/On<

5儀器操作

- *開機步驟:*

  打開氣體鋼瓶, 層析儀電源開關,電腦(確認儀表壓力是否到達)
  儀器連線(Cerity QA/QC, Instrument 1 on-line)
  (FID)等偵測器溫度到達設定值!點火
  U-ECD 打開偵測器氣體
  等訊號穩定,即可分析樣品

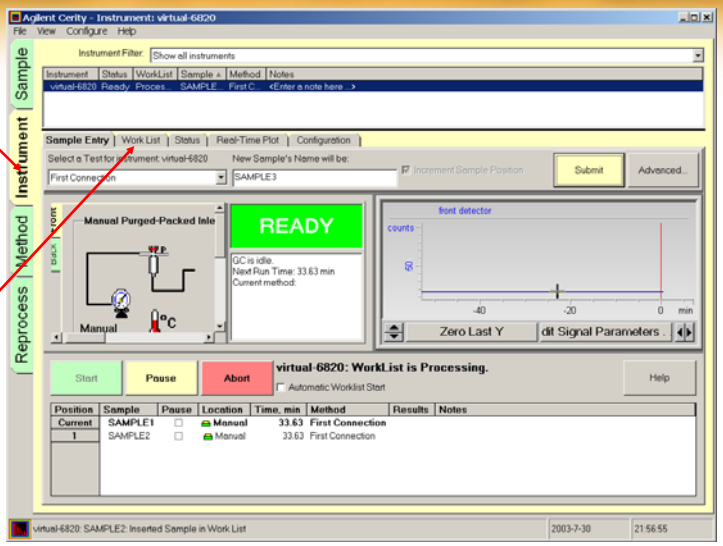
- *關機步驟:*

  OVEN降溫至室溫50℃, 偵測器(FID<100℃)
  關閉偵測器氣體,
  關閉層析儀電源
  關閉氣體鋼瓶

5儀器操作-軟體介面

Main functions easily accessed by selecting 1 of 4 Views

Sub-functions accessible by selecting a Page View

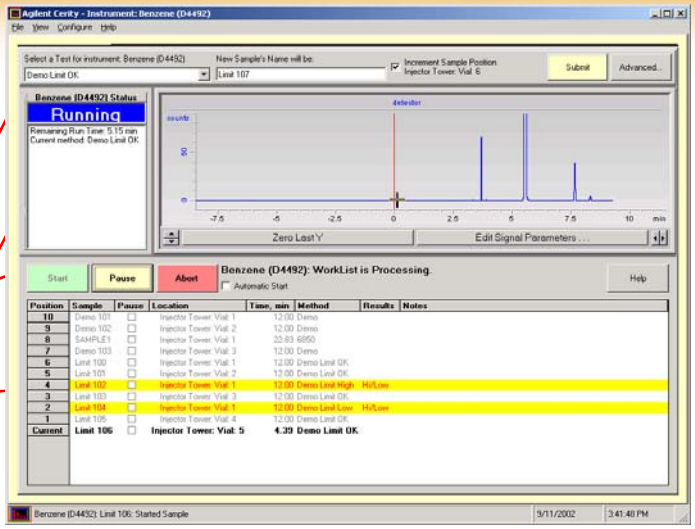


分析樣品

Just enter Test to be performed

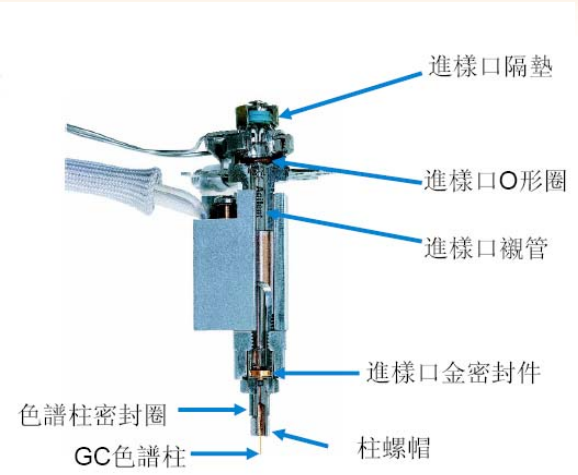
Instrument Status & Real Time Plot

Worklist With Results

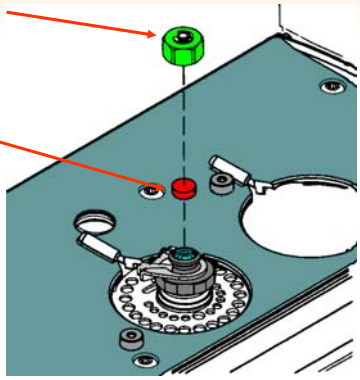


6.日常維護與保養
- 6-1注射口保養
- 6-2分析管柱保樣與維護
- 6-3偵測器保養與維護

6-1注射口Split/splitless inlet



Replace Septum



定期更換

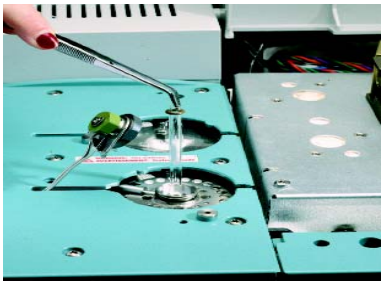
避免鎖太緊(hand tight)

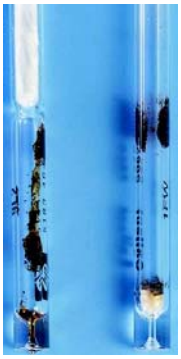
更換氣化管 Replace Liner

Purpose:sample evaporated complete in gas phase

Replace :

- peak shape degradation
- poor reproducibility
- sample decomposition
- Ghost peak





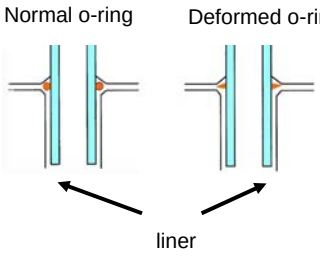
Liner 氣化管

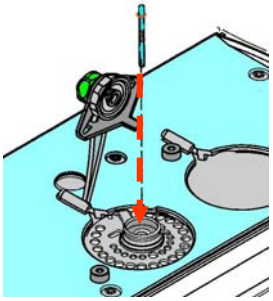
	去活性低壓降, 870uL	分流	5183-4647
	去活性, 900uL	不分流	5181-3316
	去活性, 2mm ID, 250uL	直管型	5181-8818
	廣泛適用, 870uL	分流/不分流	5183-4711
	去活性, 900uL	不分流	5062-3587
	去活性, 800uL	不分流	5181-3315
	1.5mm ID, 140uL	直管型	18740-80200
	2mm ID, 250uL	直管型	18740-80220
	4mm ID, 玻璃棉, 990uL	分流	19251-60540

B-4 O-ring

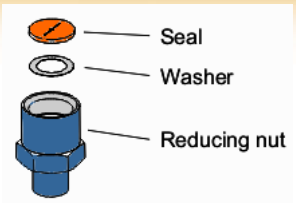
O-ring can become deformed or “flattened” over time when compressed between the insert retainer weldment and the inlet body.

Slide new o-ring over liner about ¼” and drop liner into inlet. Liner should contact the Gold Seal. Gently tighten insert retainer on the inlet weldment.





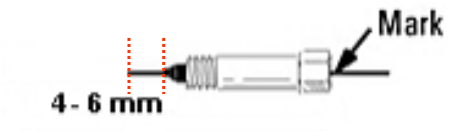
B-3 Seal & Washer



Remove the column and reducing nut.

Clean or replace seal and washer.

Trim and reinstall column.



新的分流平板(密封金墊)

- *改進的生產技術，保證更加光滑的表面，從面進一步提升密封性*
- *專有的電鍍技術保證更惰性的金屬表面*
- *新的包裝裡含有墊圈　價格不變*
- *更好的包裝確保分流平板的清潔度*

- 新的分流平板部件號 (5188-5367)
- (推薦用於分流\不分流進樣口)

B-5 Ferrule

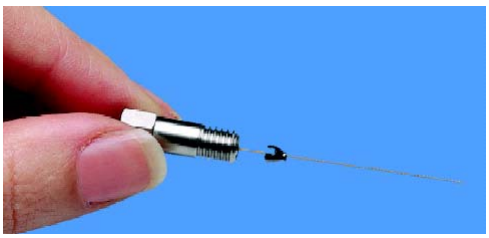
Why replace ?

oxygen

sample loss

poor retention time reproducibility

increase of noise



Ferrule Installation

	GC安裝位置	6890 GC管柱安裝位置	5890GC管柱安裝位置
Inlet	Split/splitless Inlet	管柱凸出ferrul 4~6 mm	管柱凸出ferrul 4~6 mm
	Purged packed Inlet	管柱凸出ferrul 1~2 mm	管柱凸出ferrul 2 mm
Det.	FID/NPD	伸到底後往下拉1 mm	伸到底後往下拉1 mm
	TCD	伸到底後往下拉1 mm	伸到底後往下拉1 mm
	ECD/uECD	管柱頂端至Nut約70±1 mm	管柱頂端至Nut約75 mm
	FPD	管柱頂端至Ferrul約153 mm (NEW FPD 145 mm)	管柱頂端至Nut約162 mm

6-2分析管柱維護-Condition column

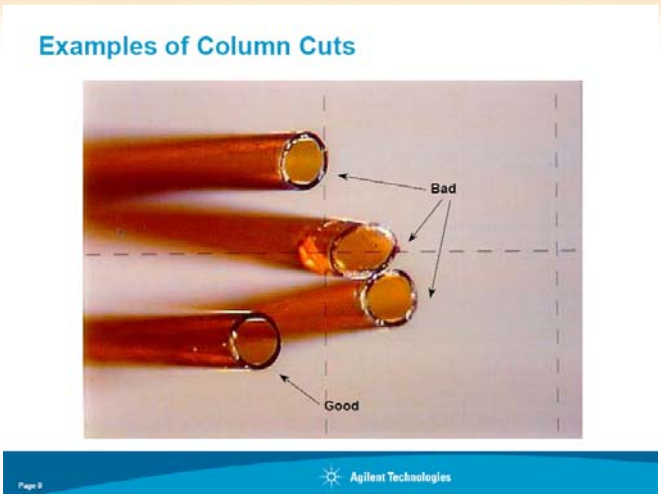
1. 首先使所有加熱部位（如進樣口和偵測器）降温
2. 關閉載氣，將原色譜柱拆下，用no-hold ferrule(5181-3308)封住偵測器端
3. 將管柱接在進樣口上，開通載氣流量(依色譜柱管徑選擇適當流量)
4. 切割毛细管柱出口，將出口端插入盛有甲醇的燒杯，正常情况下 應能够觀察有氣泡產生，否則應即時檢查漏氣、色譜柱斷裂、或 進樣口參數設置
5. 保持烘箱開放，用載氣流量跑 15-30min，以排除O2 (色譜柱壓力選擇如後)
6. 關閉烘箱，提升進樣口温度，缓慢升温至分析方法的最高温度
7. 温度升至方法最高温度+20度 (請勿超過色譜柱恆常耐受最高溫度)
   後繼續烘烤2小时（或参考色譜柱出廠建議的烘烤時间），降低爐温，將色譜柱出口端接到偵測器

- ***觀察blank baseline 是否穩定（不進樣的空针，2~3次）***
- ***不要使用H2做載氣烘烤色譜柱，以免引起爆炸。***

A-2 Common Causes of Column Performance Degradation

1. Physical damage to the polyimide coating
2. Thermal damage
3. Oxidation (O_2 damage)
4. Chemical damage by samples
5. Contamination

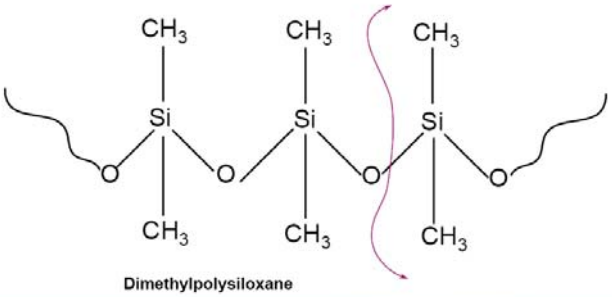
A-2-1 Physical damage to the polyimide coating



A-2-2/3 Thermal /oxidation damage

Thermal Damage

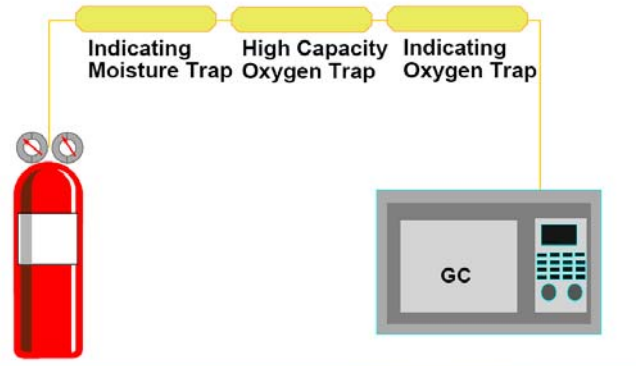
Degradation of the stationary phase is increased at higher temperatures. Breakage along the polymer backbone.



Dimethylpolysiloxane

Gas purifiers

Configurations for Carrier Gas Purifiers



A-2-4 Chemical damage by samples

Chemical Damage

Bonded and cross-linked columns have excellent chemical resistance except for inorganic acids and bases

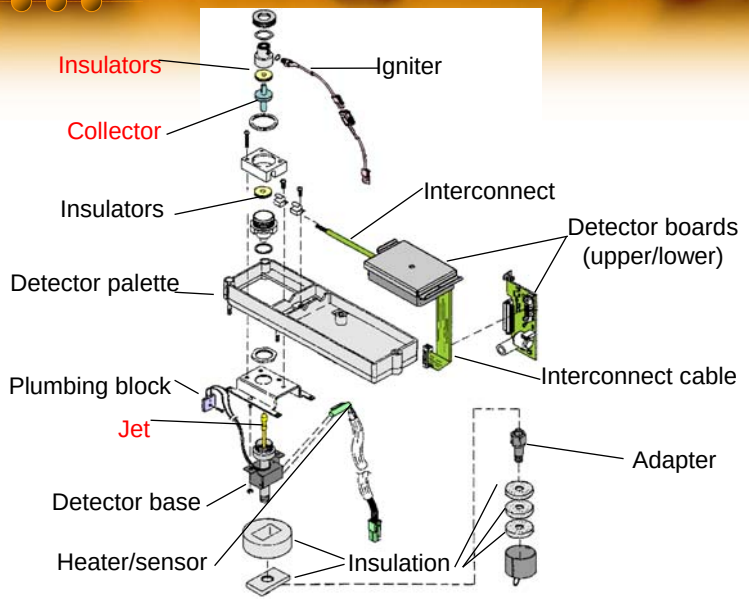
HCl NH_3 KOH $NaOH$

H_2SO_4 H_3PO_4 HF etc.

Chemical damage will be evident by excessive bleed, lack of inertness or loss of resolution/retention.

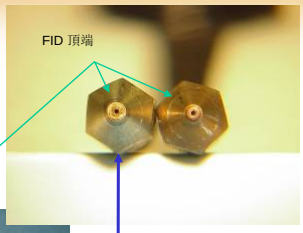
Agilent Technologies

6-3偵測器維護與保養 -FID Parts Explosion

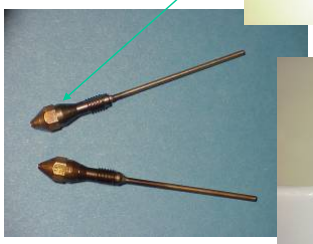


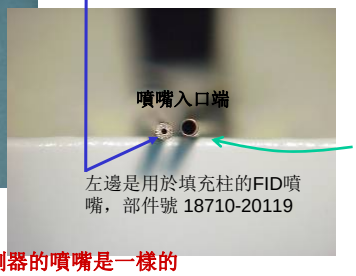
噴嘴的識別區分

用於填充柱和毛細管柱的 FID 噴嘴很難從噴嘴頂端的外觀和長度上加以區分，



把FID 噴嘴安裝到檢測器上





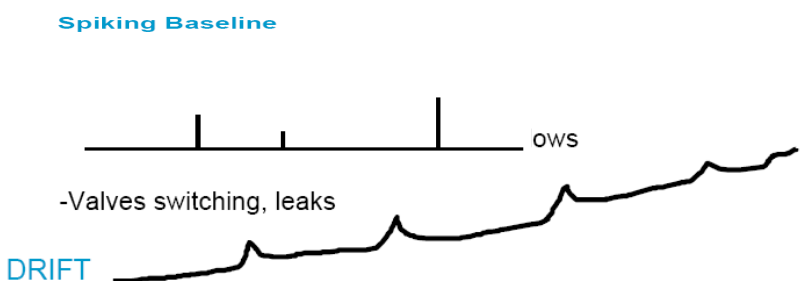
左邊是用於填充柱的FID噴嘴，部件號 18710-20119

用於填充柱的 FID 噴嘴,其管內徑小於毛細管柱噴嘴的管內徑. 可從噴嘴的入口端觀察到

*** 注: 用於NPD 和 FPD 檢測器的噴嘴是一樣的**

FID Typical Problems

- ***Flame blowing out or not lighting***
- ***Spiking***
- ***Low sensitivity***
- ***Noise***
- ***Drift***



Electron Capture Detectors (ECD)

Electron Capture Precautions

CARRIER GAS ..... **dry and pure - 99.9995 %**

SEPTUM ..... **low bleed (precondition)**

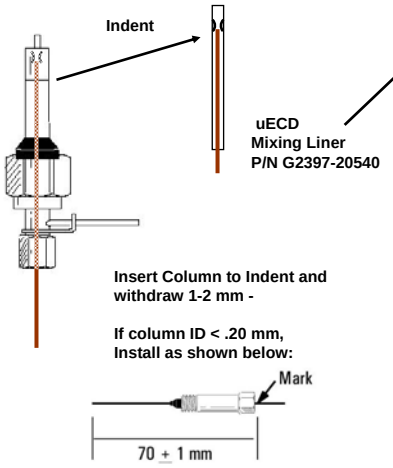
SOLVENTS ..... **clean, preferably hydrocarbon NEVER halogenated**

QUANTITATION ..... **check linearity**

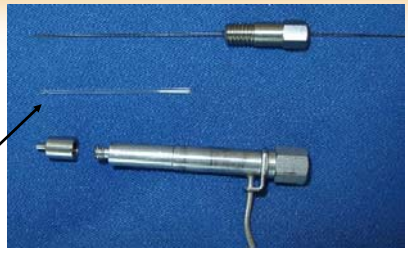
汙染 –熱烤u-ECD
將u-ECD端管柱拆下用盲封封住偵測器端(確認沒有漏氣)!設定OVEN 250℃,u-ECD 350 ℃ ,烘烤overnight,觀察基線

uElectron Capture Detectors

Micro Electron Capture Column Installation - using Mixing Liner
(Compatible with 5890/6890 ECD)

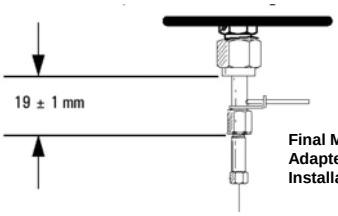


uECD
Mixing Liner
P/N G2397-20540



Insert Column to Indent and withdraw 1-2 mm -

If column ID < .20 mm, Install as shown below:



Final MUG Adapter Installation