

Agilent LC日常基本維護

安捷倫客戶服務工程師

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Keys to a Highly Performing HPLC



- Low Delay Volume
- Low Extra-Column Volume
- Accurate and Consistent Composition
- Accurate and Consistent Flow
- Low Pressure Pulsation
- No Leaks
- No Unnecessary Backpressure
- Cleanliness
- Lamp Light Output

Tubing and
Connections

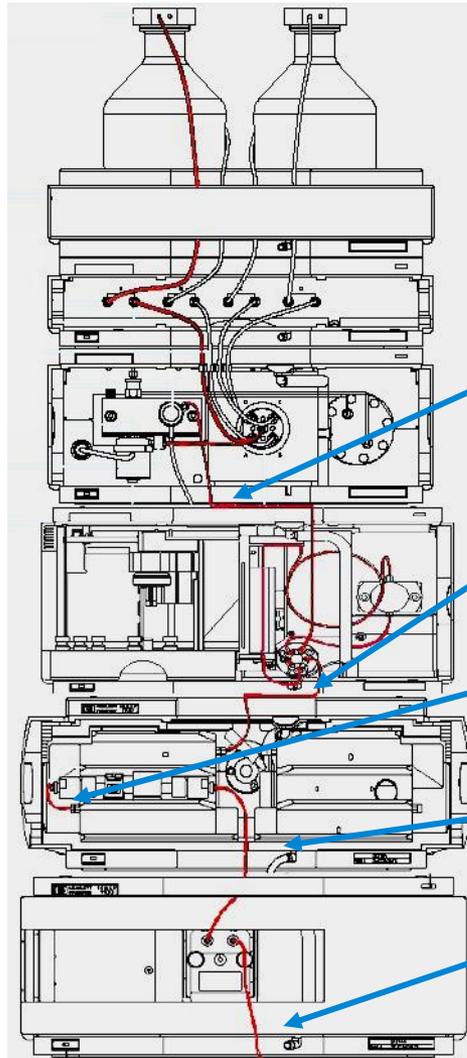
Pump

System
Maintenance

Detector



Tubing



0.17mm ID capillaries = standard LC

0.12mm ID capillaries = RRLC

400mm/0.17mm (G1312-87303) Pump to ALS

700mm/0.17mm (G1312-87304) Pump to Therm. ALS

180mm/0.17mm (G1313-87305) ALS to TCC inlet

380mm/0.17mm (01090-87306) Therm.ALS to TCC inlet

180mm/0.12mm (G1313-87304) ALS to heat exchanger

280mm/0.12mm (01090-87610) Therm.ALS to heat exchanger

90mm/0.17mm (G1316-87300) heat exch. outlet to column inlet

105mm/0.12mm (01090-87611) heat exch. outlet to column inlet

380mm/0.17mm (G1315-87311) Column to DAD

150mm/0.12mm (G1315-87312) Column to DAD

600mm/0.17mm PEEK (5062-8522) Column to VWD

DAD PTFE, wide pore (5062-2462) Detector to Waste

VWD Peek, 48cm/0.25mm (5062-8535) Detector to Waste

Connection to LC/MS

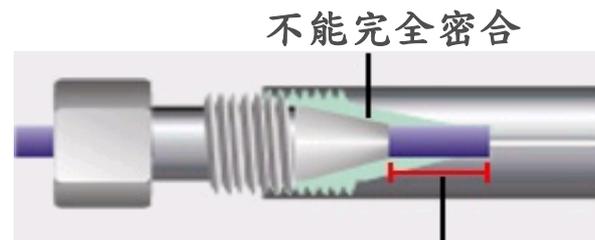
500mm/0.12mm (G1315-87305) DAD/MWD to MS

500mm/0.12mm (G1316-87309) TCC/VWD to MS

* Blue capillaries 0,25mm = Prep LC

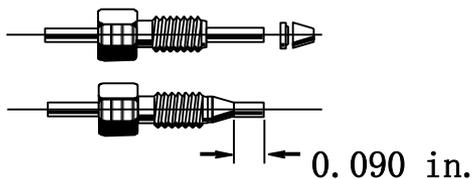


管線接頭

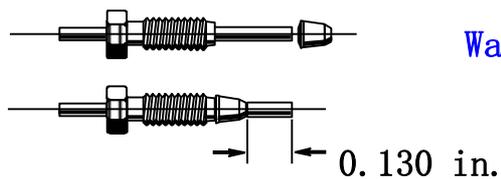


如果接頭過長，則可能漏液

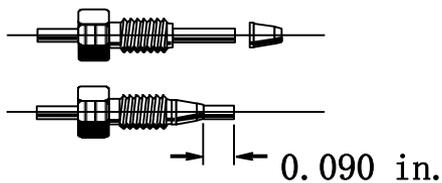
Swagelok



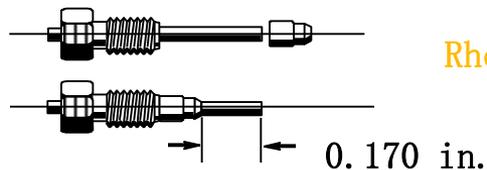
Waters



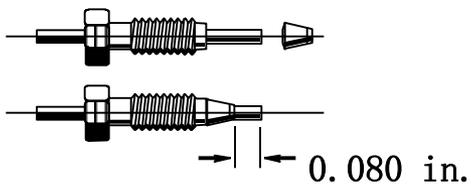
Parker



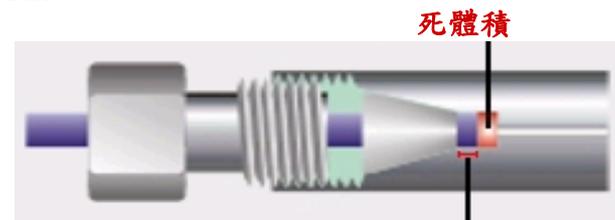
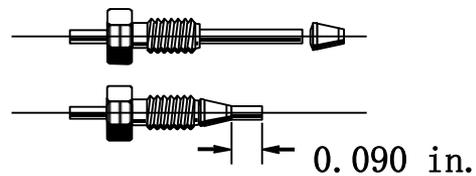
Rheodyne



Valco



Uptight



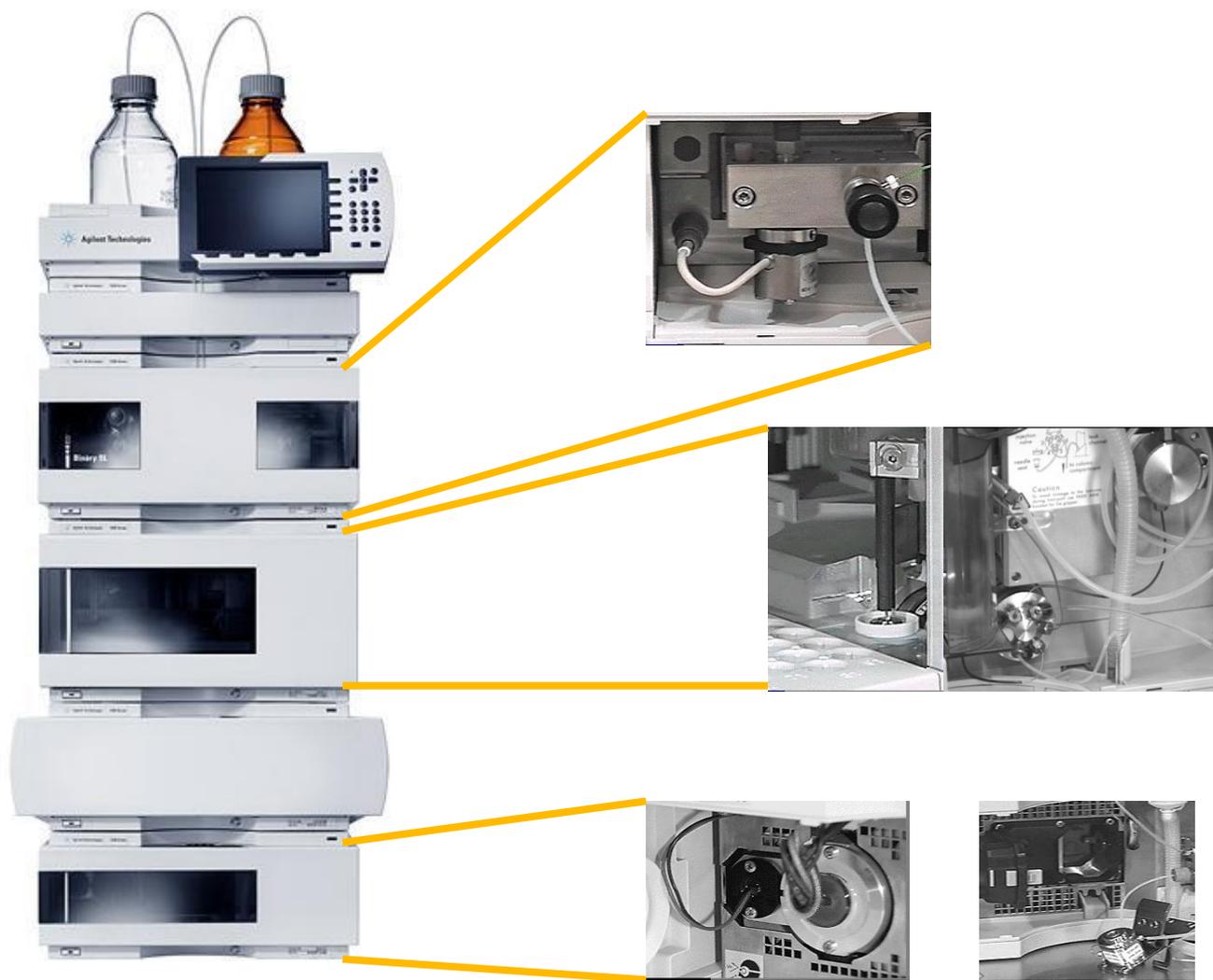
如果接頭過短，則有死體積存在，導致峰形變差

一般注意事項

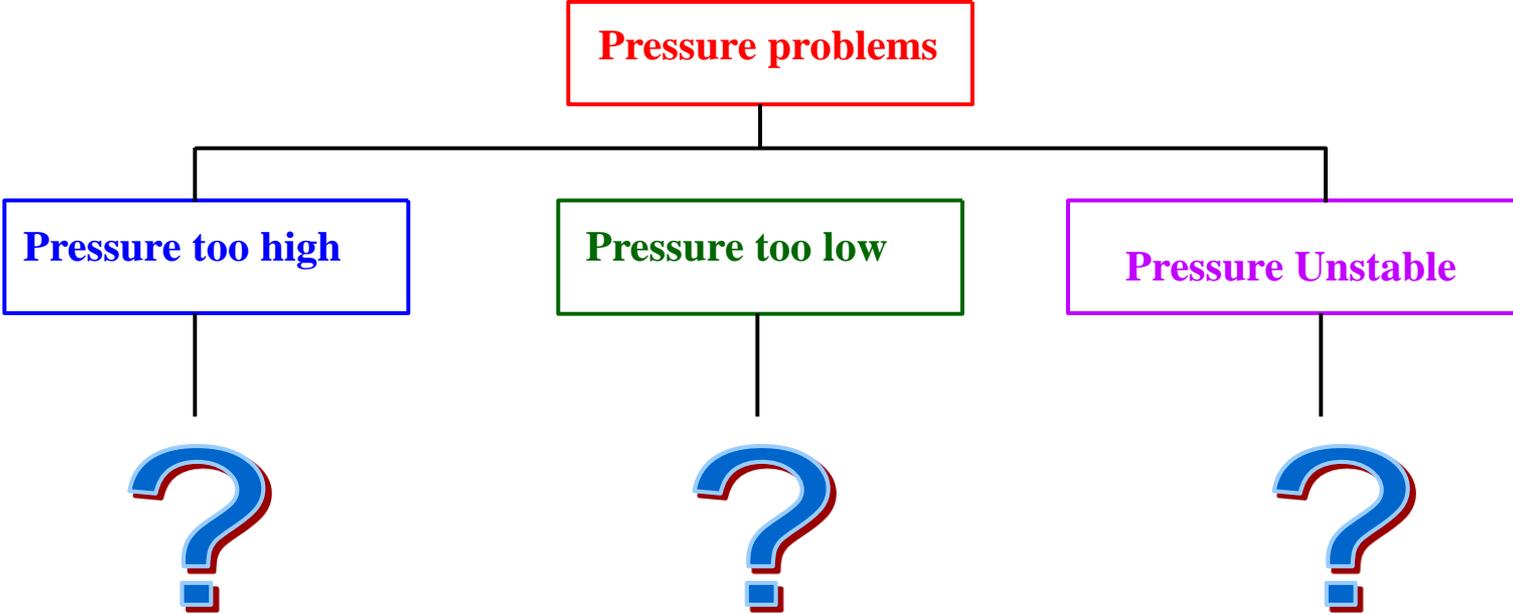
1. 定期清洗溶劑過濾頭（35%濃硝酸浸泡1小時，不使用超音波震盪）
2. 移動相應經過0.45um或更小孔徑的濾器過濾，，注意濾膜種類
3. 根據情況更換Purge閥內的PTFE濾芯（Purge閥打開，純水5mL/min，壓力超過10bar時需更換）；
4. 定期清洗線上除氣機及比例閥，尤其是長期使用水相或緩衝鹽的流路
5. 定期更換水相,可以的話至於棕色瓶中



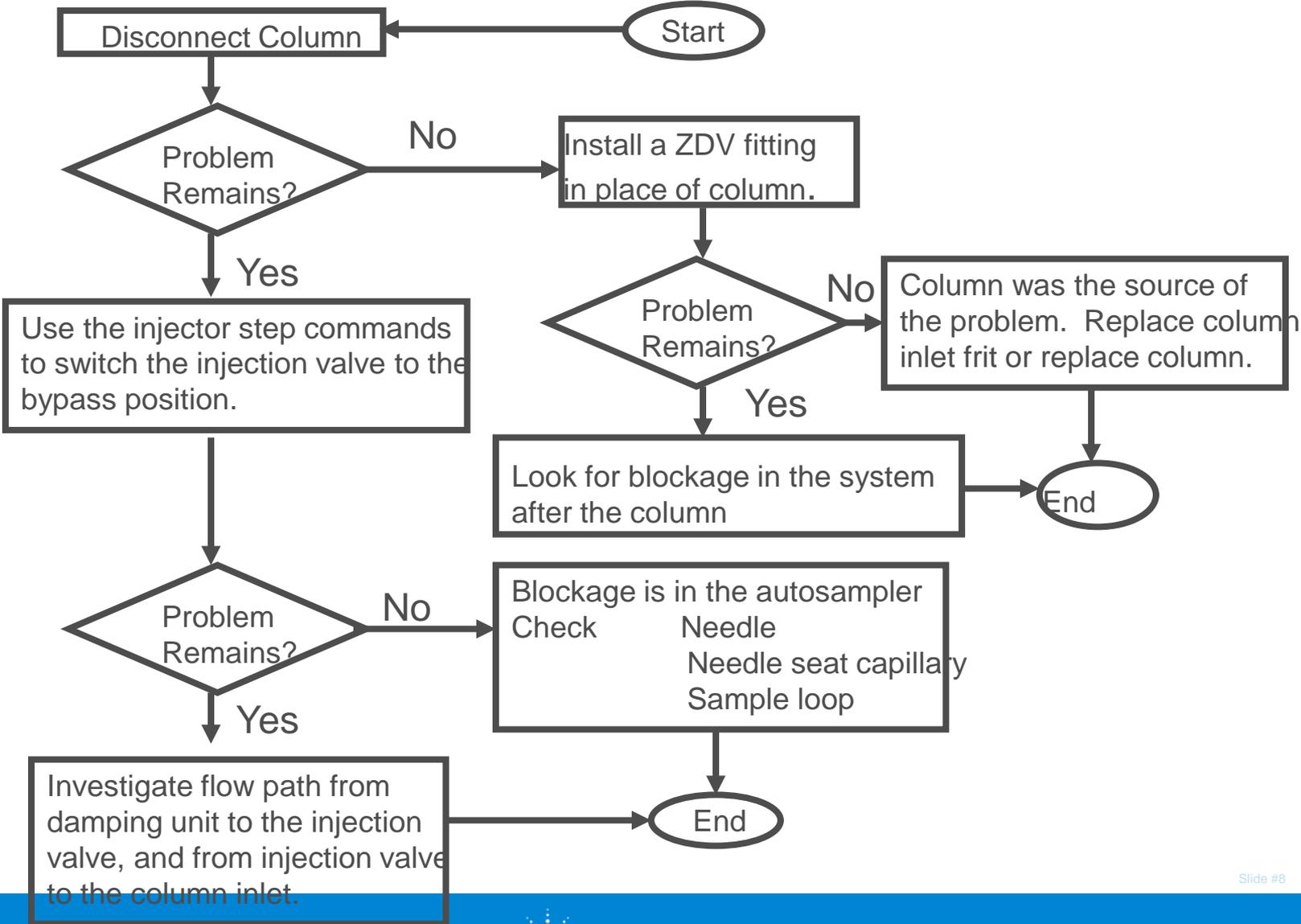
Maintenance Areas of the HPLC System



Problems with the System Pressure



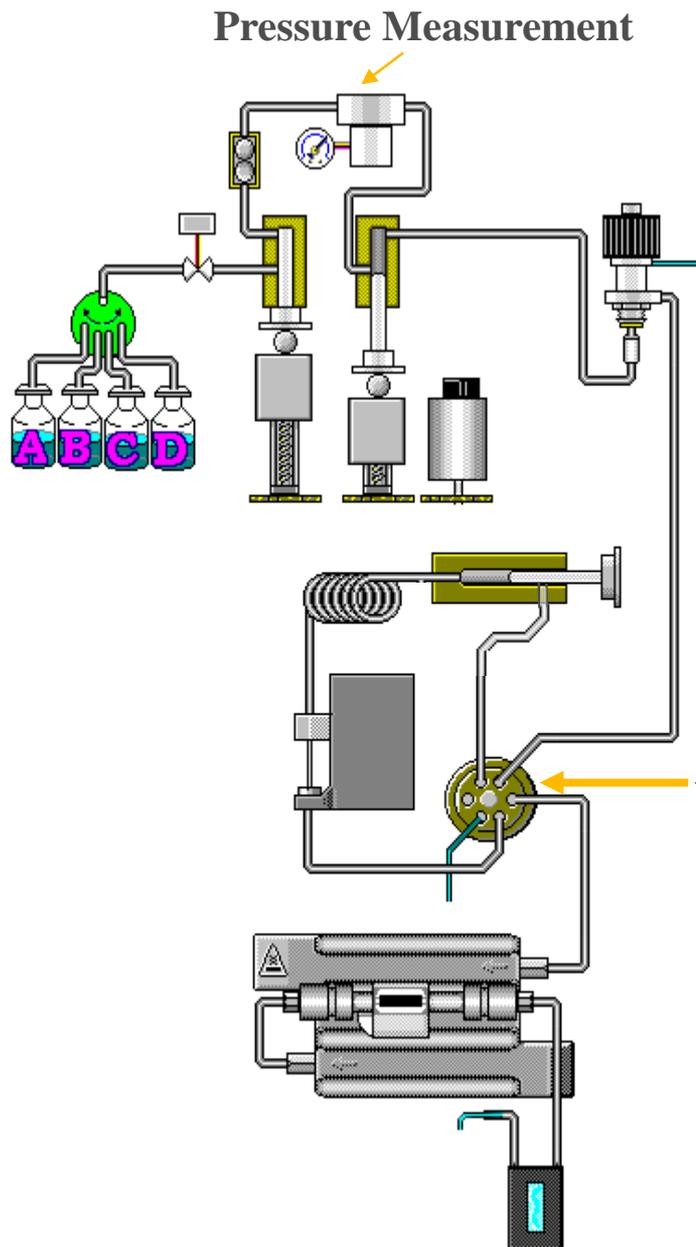
Pressure too high



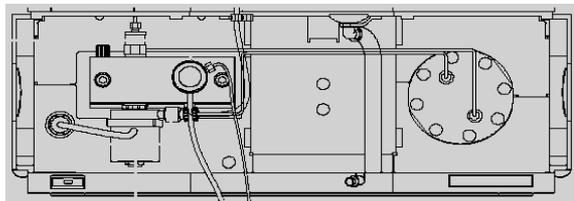
Pressure Problem I

Pressure Too High

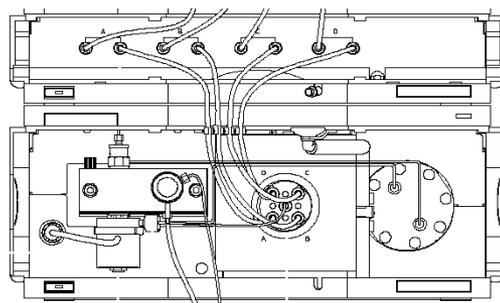
- Column inlet frit contaminated
- Frit in purge valve contaminated
- Column contaminated
- Blockage in a capillary, particularly needle seat capillary
- Rotor in injection valve plugged
- Injection needle or needle seat plugged



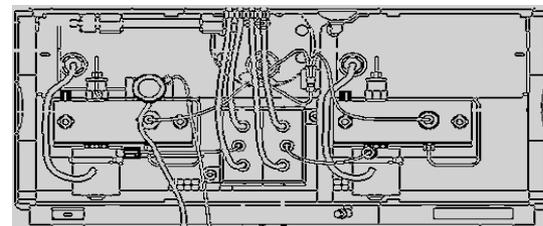
Agilent 1200 Series Pump Models



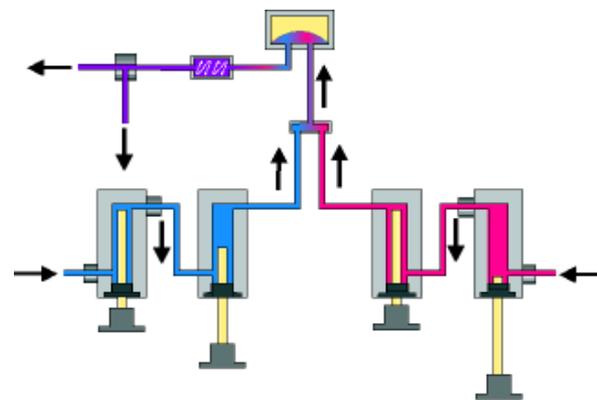
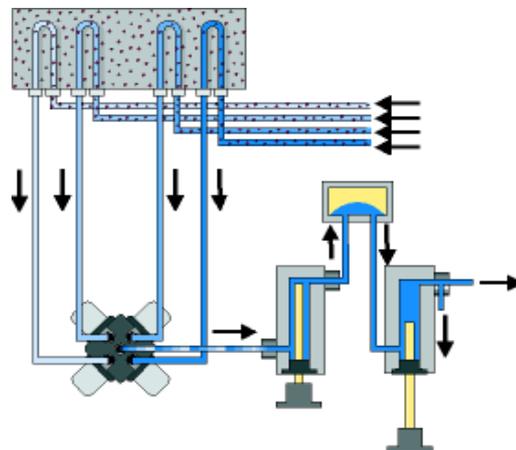
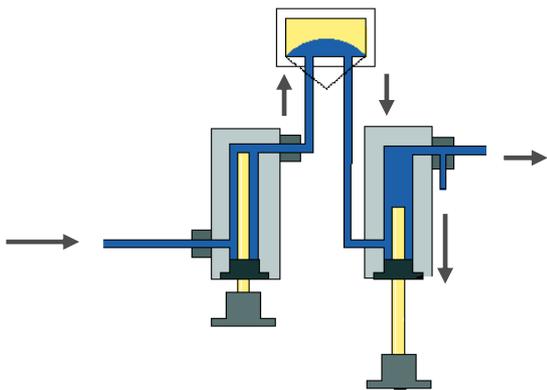
Isocratic Pump



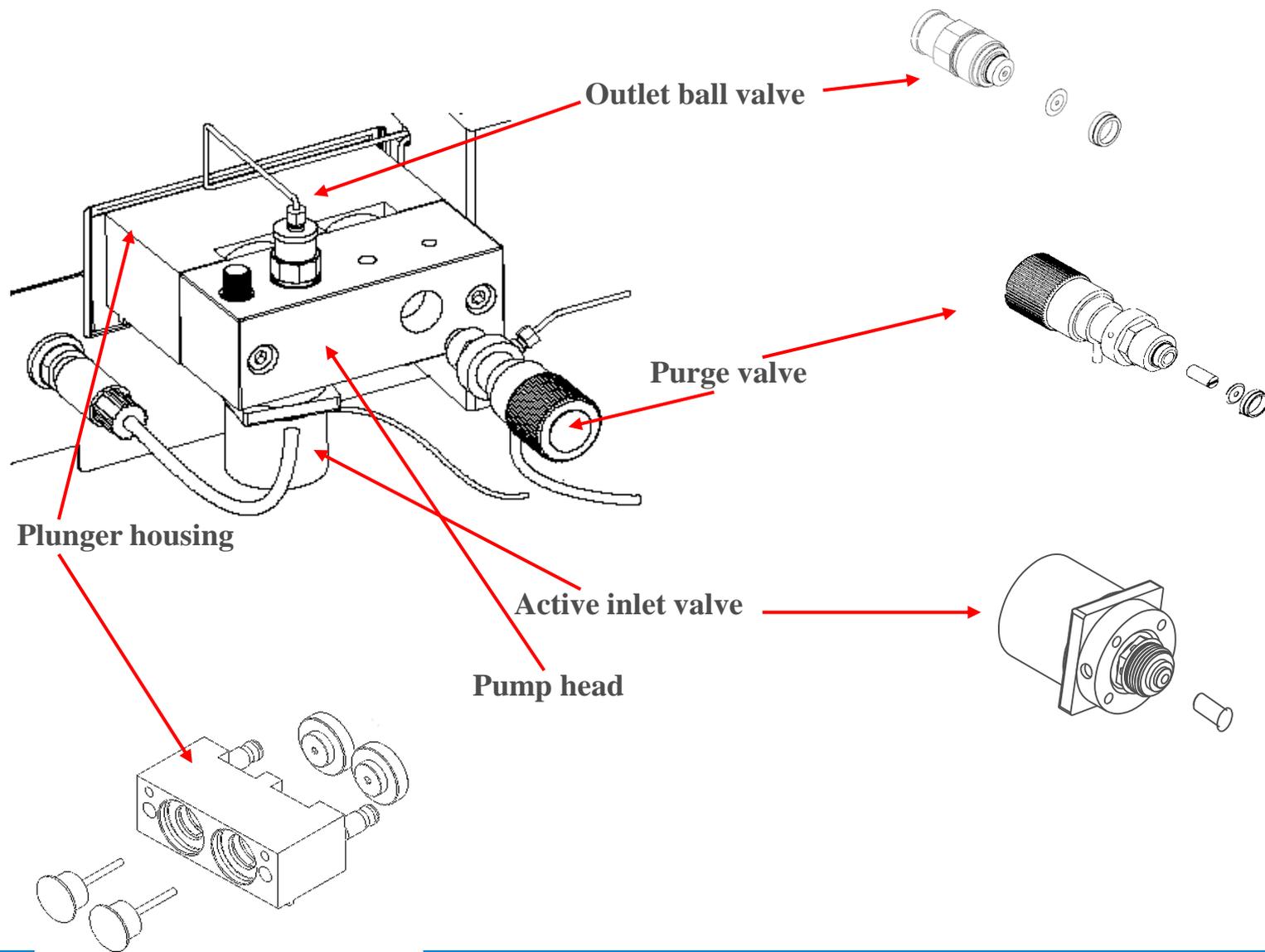
Quaternary Pump



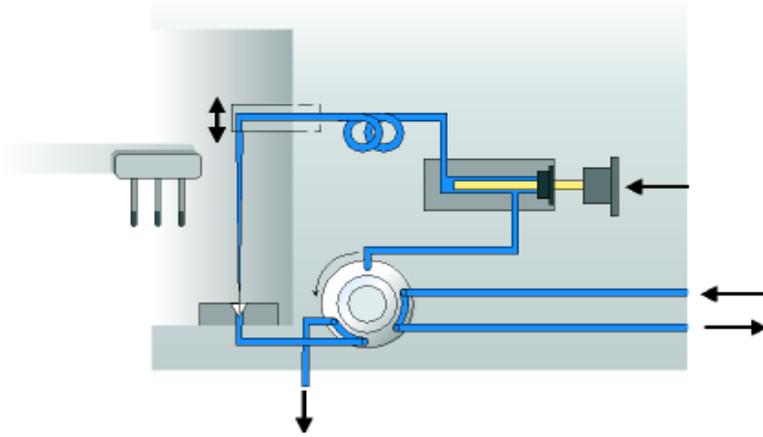
Binary Pump



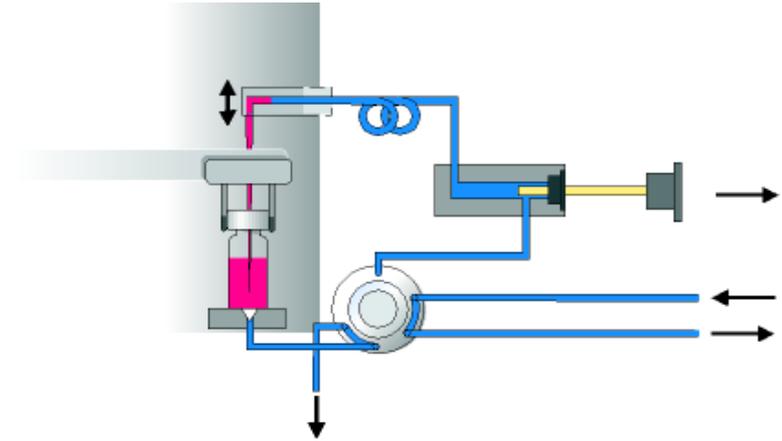
Pump Head – Main Components



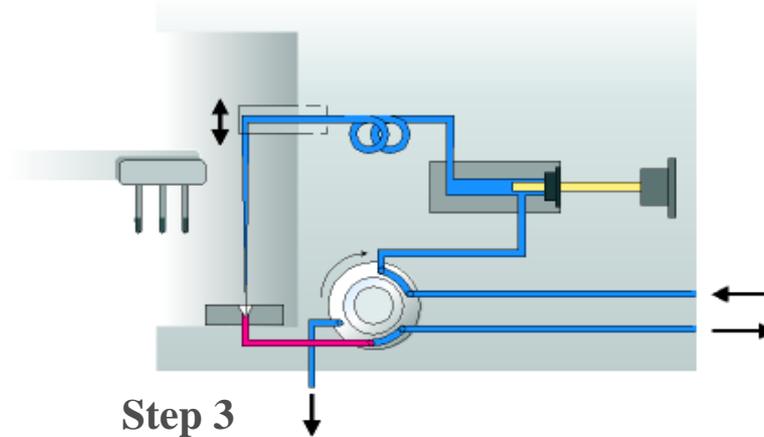
Injection Step



Step 1 準備位置(bypass)

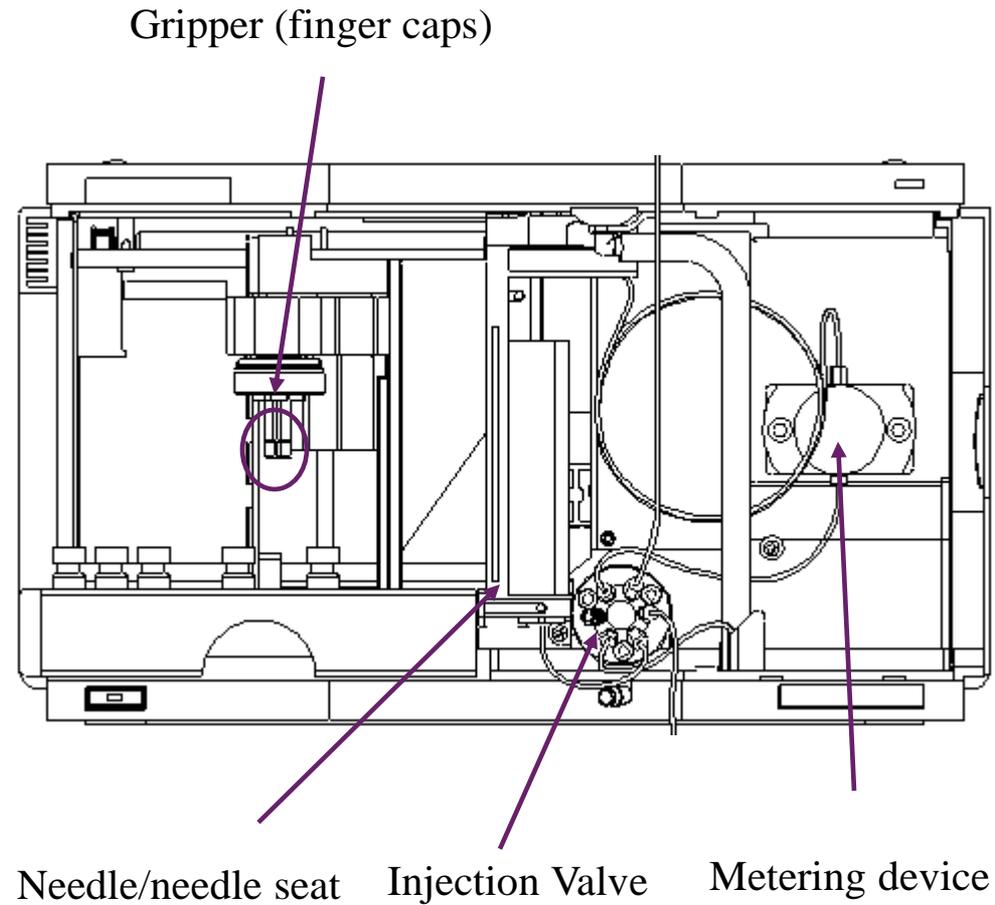


Step 2 抓取樣品瓶
吸取樣品(bypass)



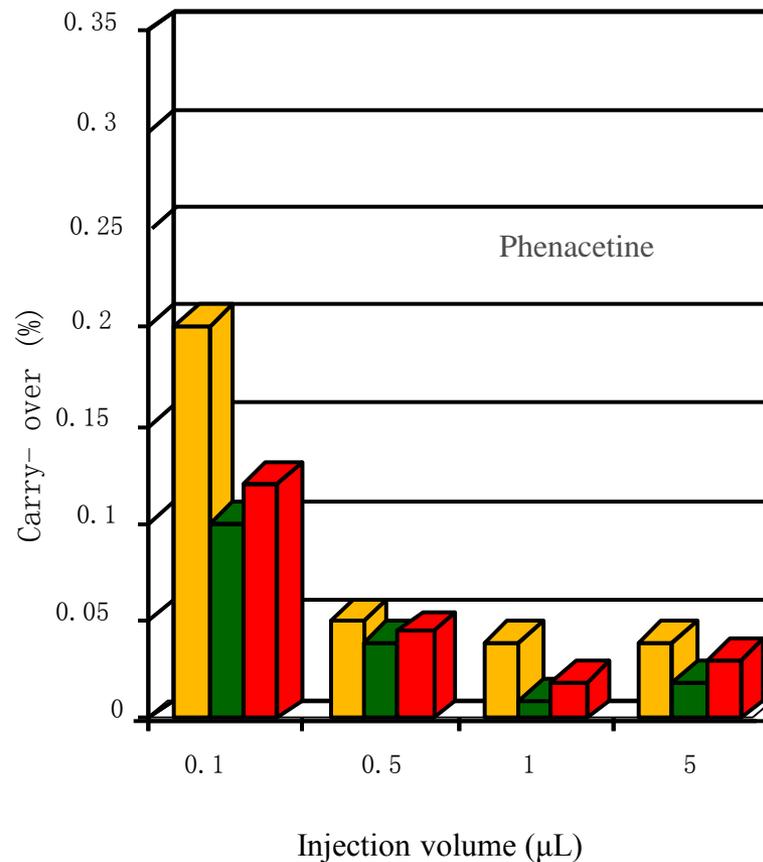
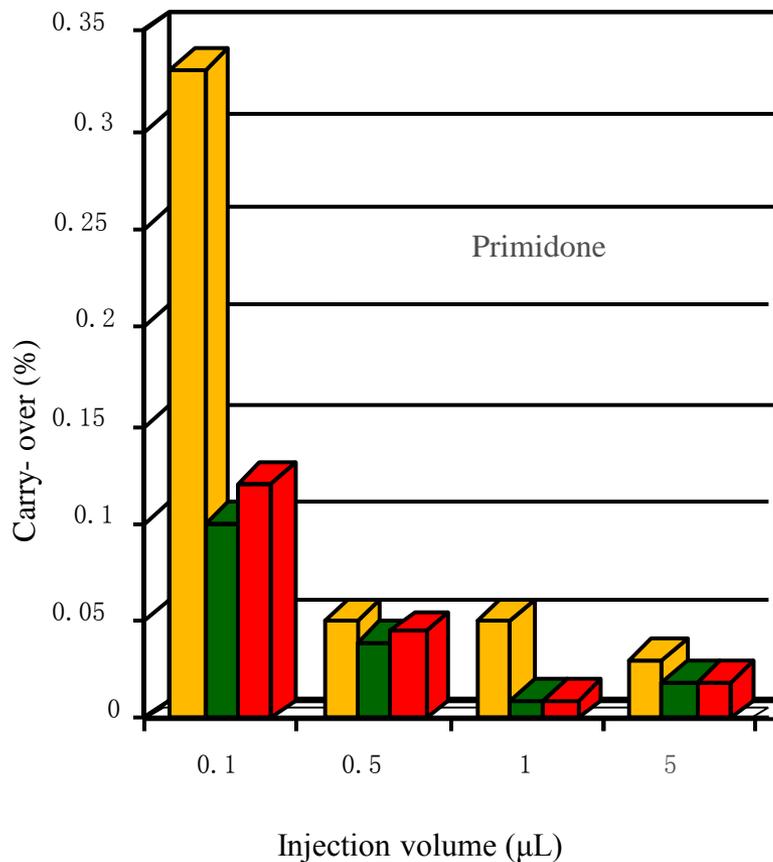
Step 3 進樣和運行位置(Mainpass)

Autosampler Maintenance



洗針對樣品殘留的影響（清洗瓶蓋帽/不蓋帽）

■ 不洗針 ■ 洗針(不蓋瓶蓋) ■ 洗針(蓋瓶蓋)



Pressure Problem II

Pressure Too Low

Solvent inlet frit plugged

Leak in a capillary connection or other part (pump seals)

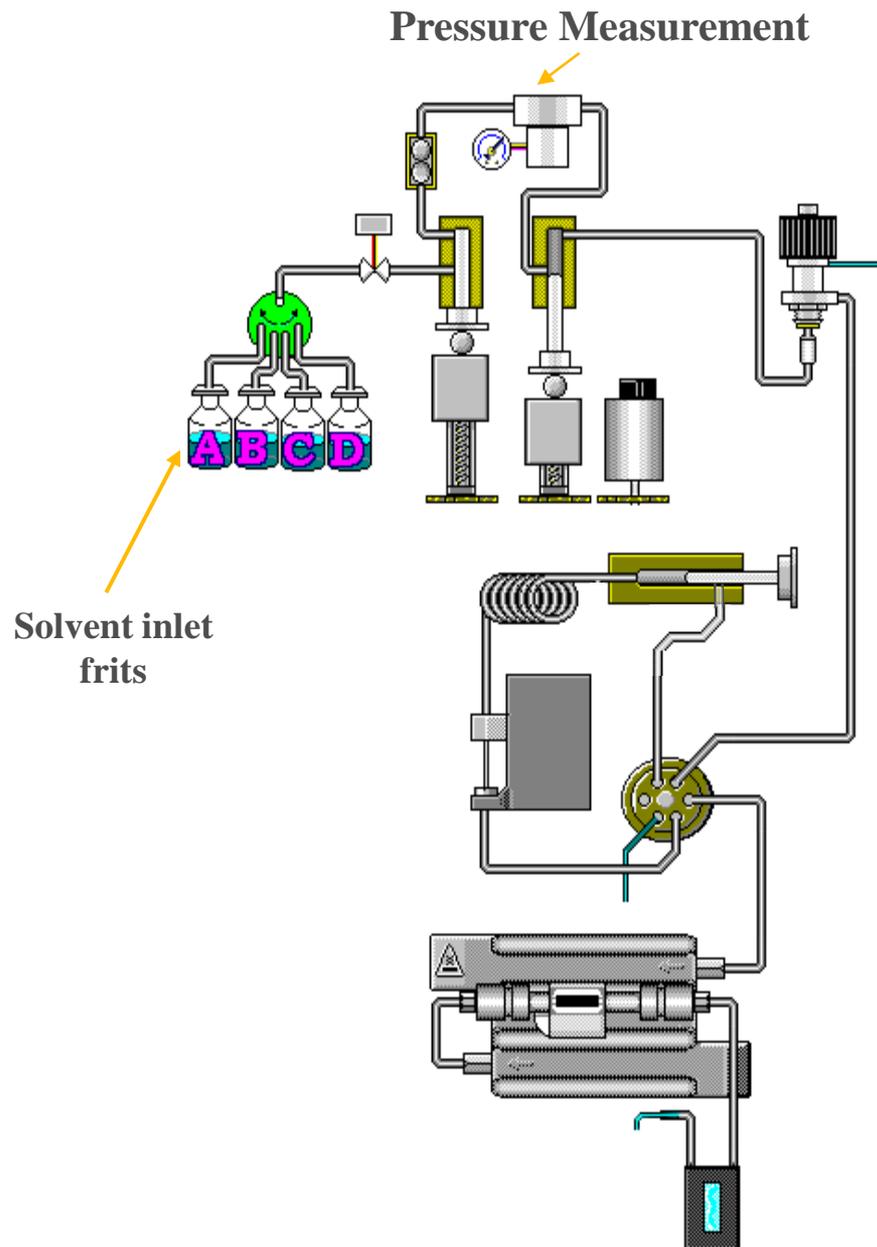
Wrong solvent or flow rate

Active inlet valve defective

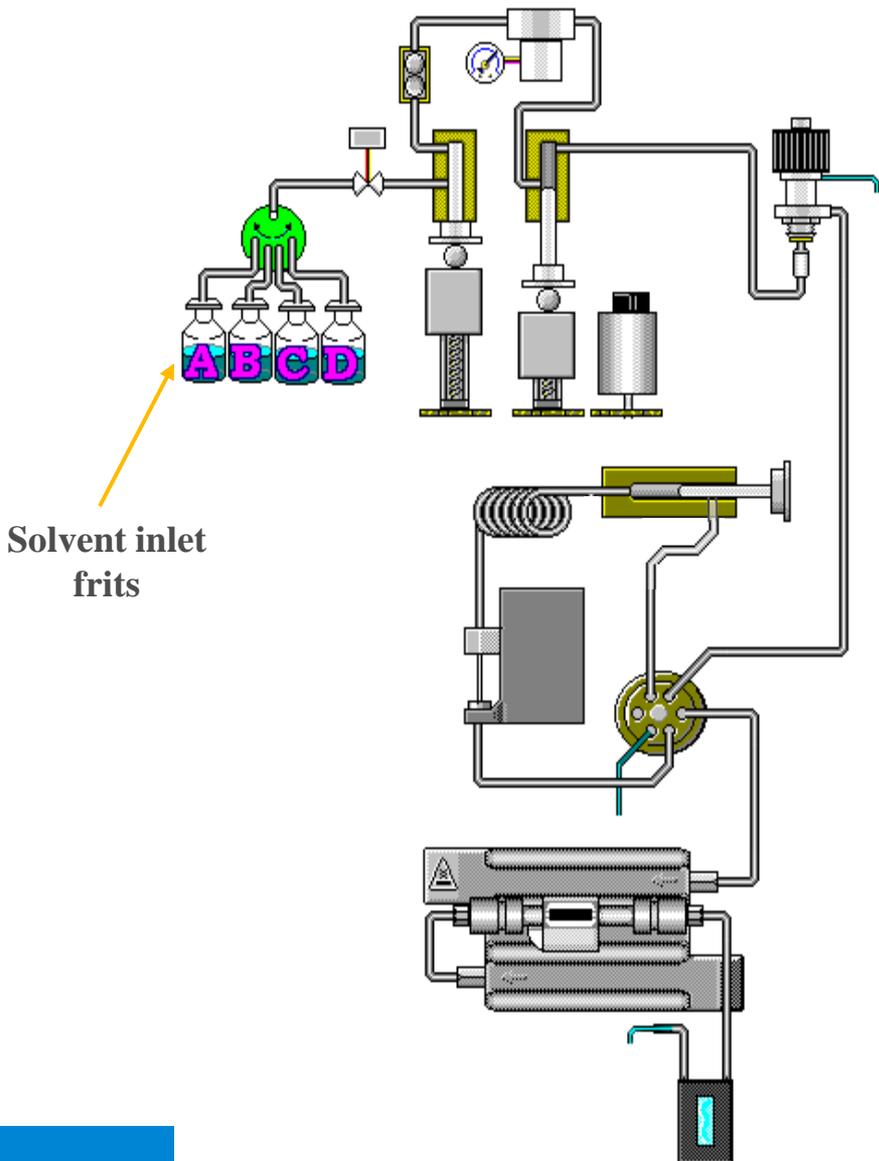
Multichannel Gradient valve incorrectly proportioning

Outlet ball valve defective

Column defective (stationary phase)



Pressure Problem III



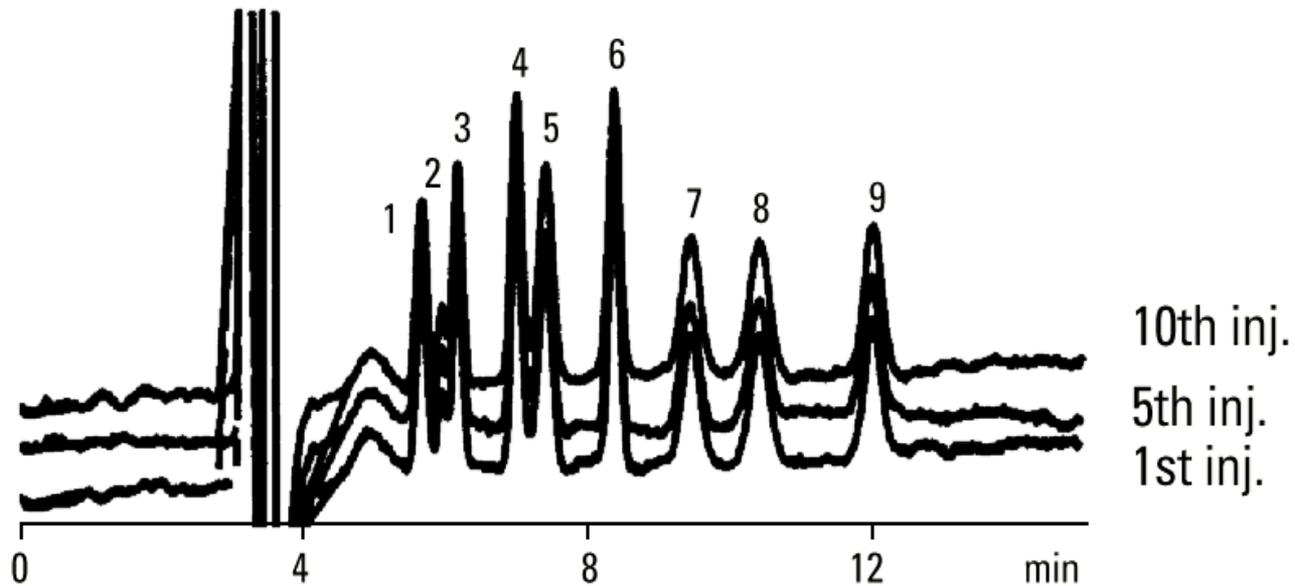
Pressure Fluctuation

- Solvent inlet frits plugged
- Solvent not degassed
- Pump seals leaking
- Outlet ball valve defective
- Active inlet valve defective

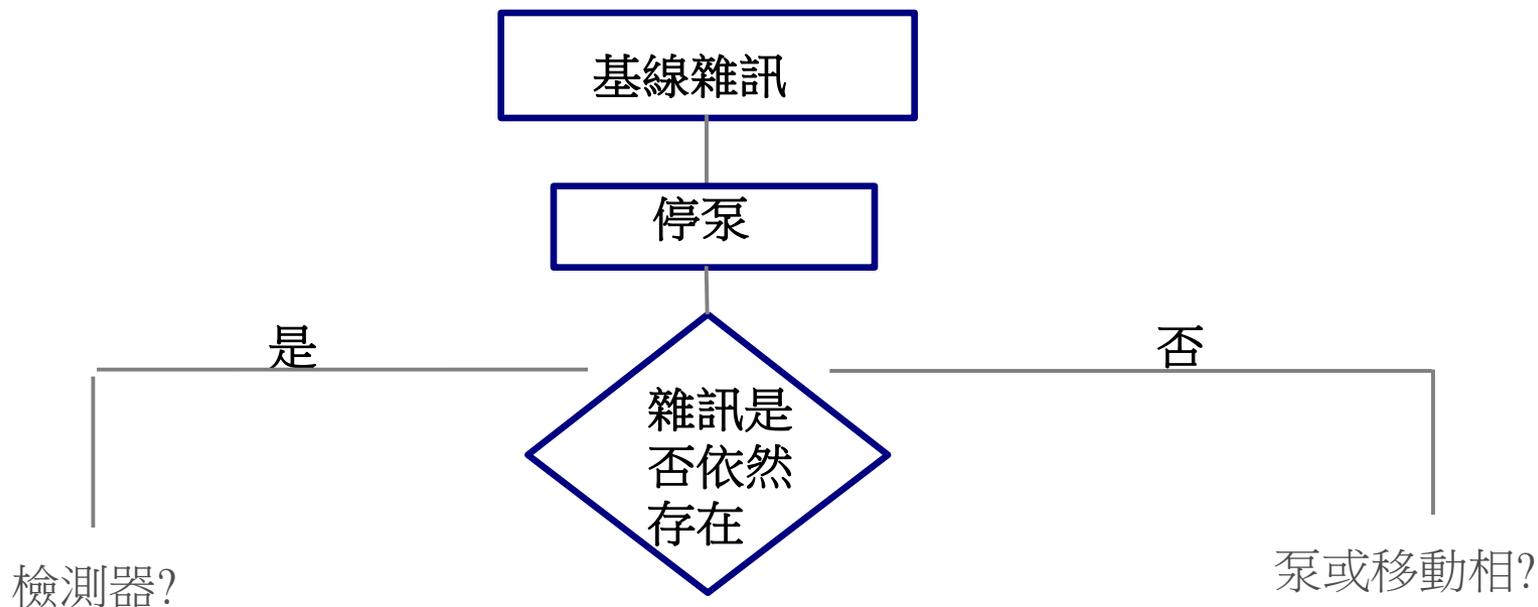
Usually an indication there is air in the pump

Reproducibility

- Area and Peak Height problems together point to the autosampler system
- Area and Retention Time problems together point to the pump



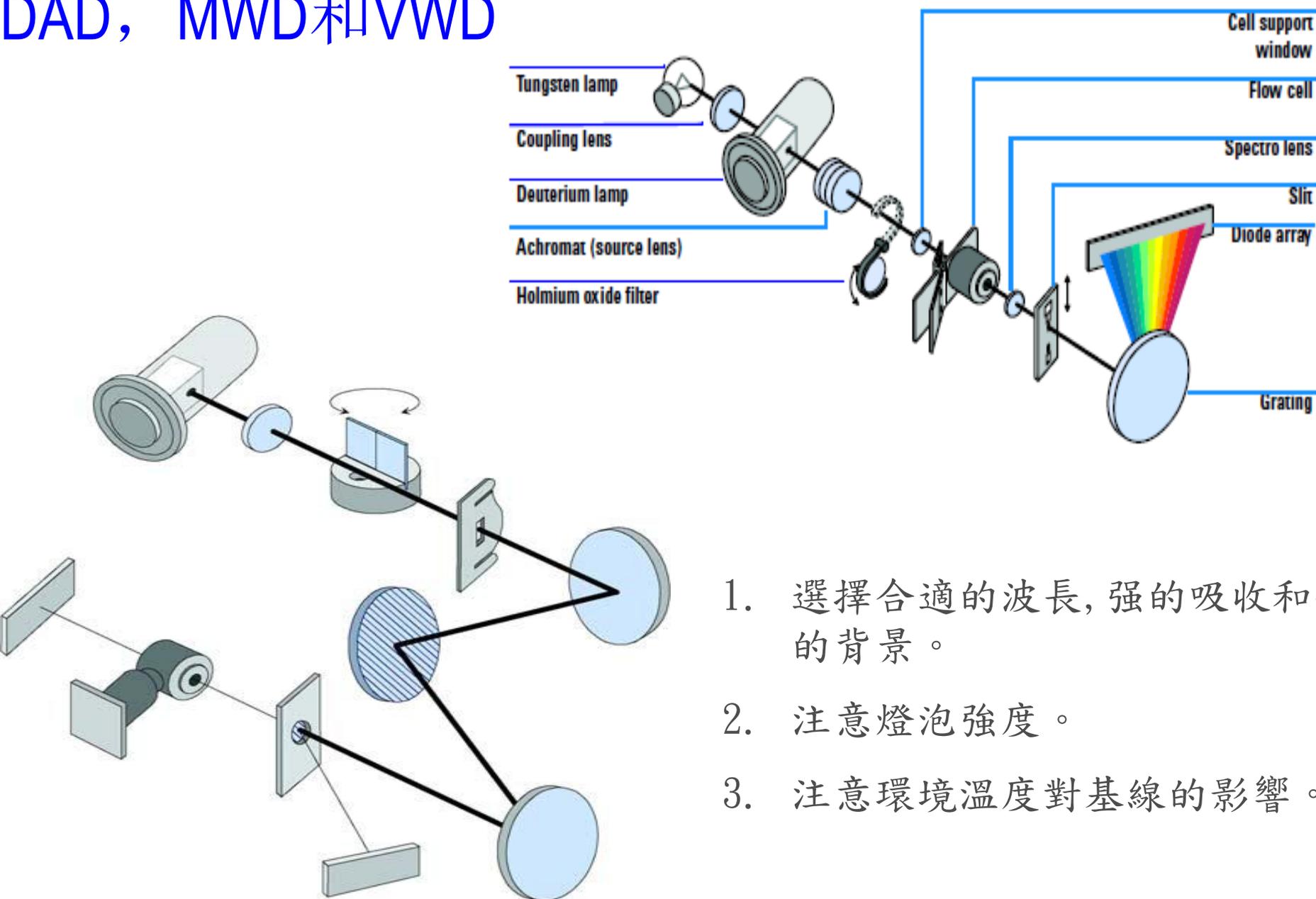
基線雜訊



其他要考慮的問題：

- ▲ 是否改變了移動相的組成？
- ▲ 是否改變了檢測波長？
- ▲ 在你的儀器上使用的最後的移動相是什麼？
- ▲ 移動相是否相溶？
- ▲ 移動相是否乾淨？

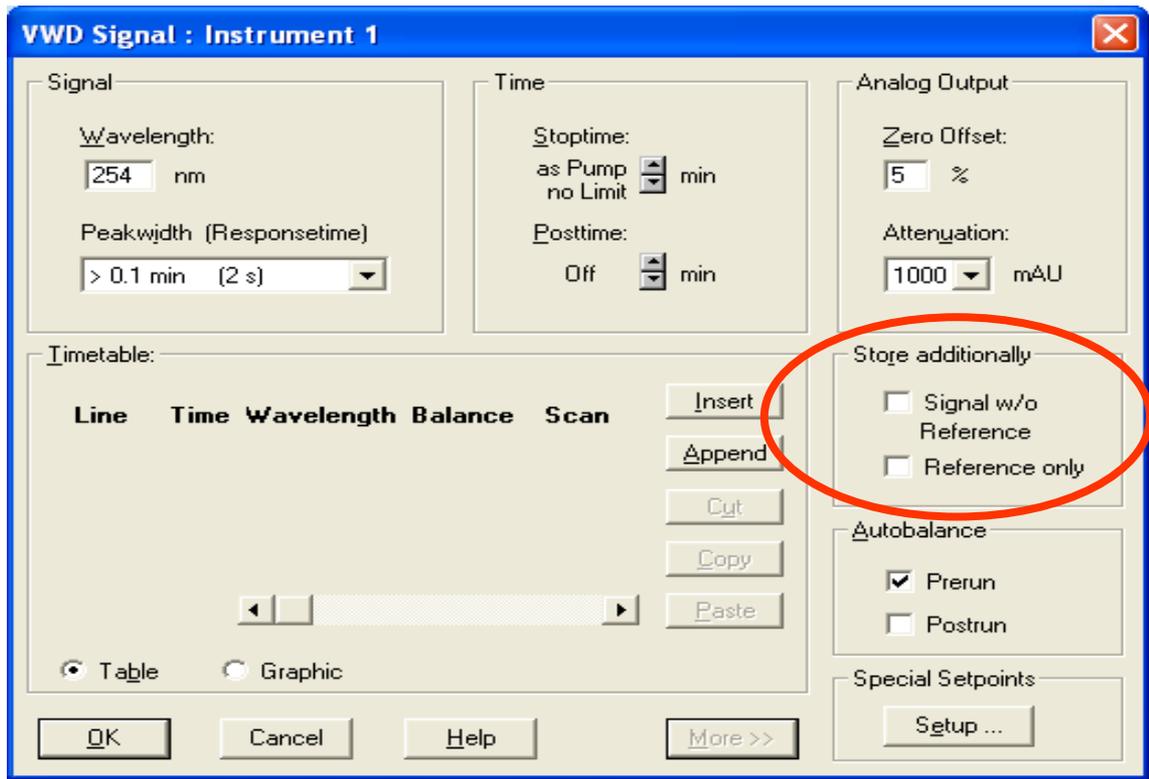
DAD, MWD和VWD



1. 選擇合適的波長, 强的吸收和低的背景。
2. 注意燈泡強度。
3. 注意環境溫度對基線的影嚮。

Measuring Signal and Reference Diodes

For troubleshooting, measure Signal without Reference and Reference Only

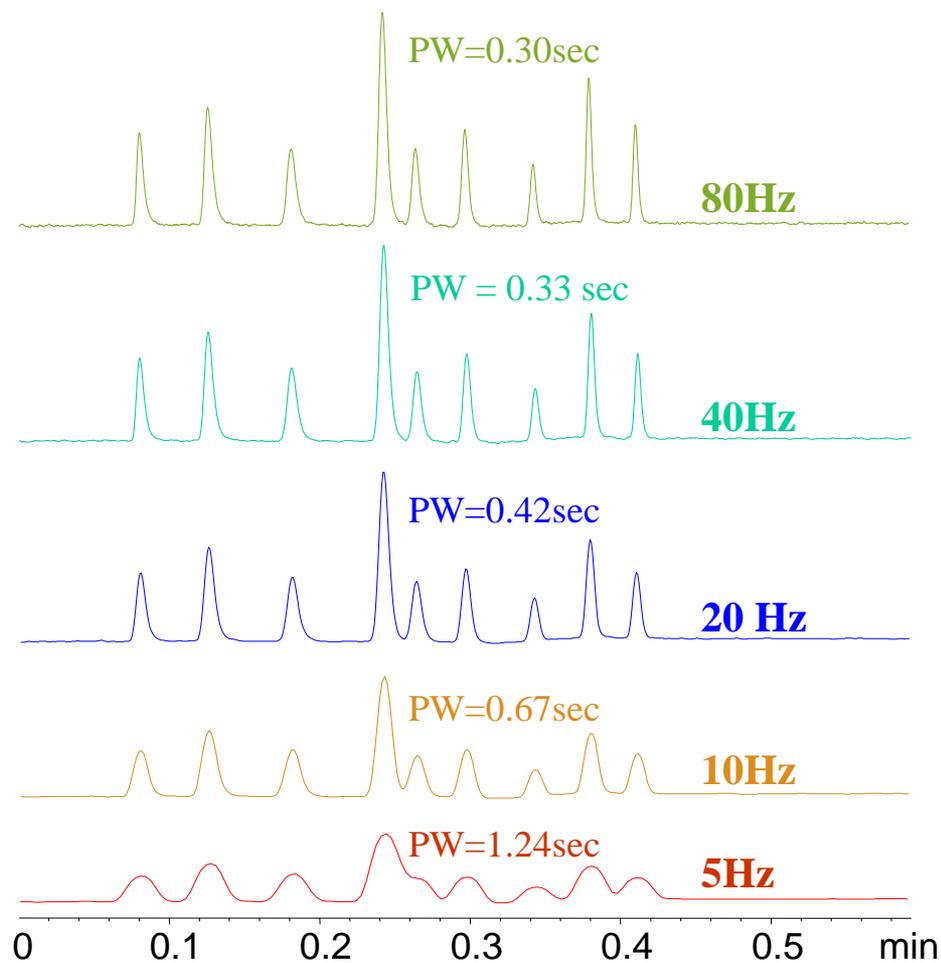


UV Absorption of Solvents

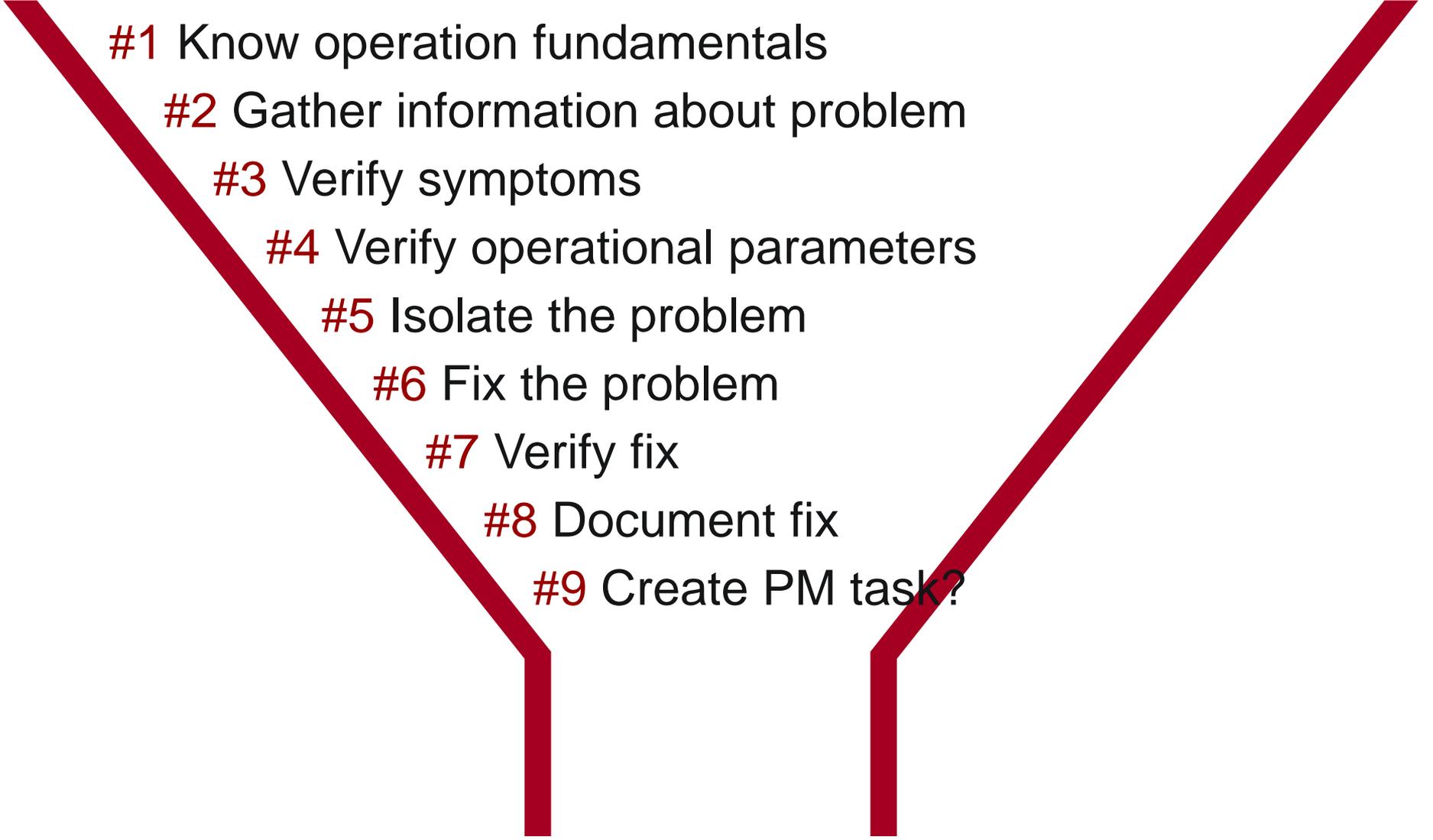
<u>Solvent</u>	<u>UV Cut-off (nm)</u>
Acetonitrile	190
Water	190
Cyclohexane	195
Hexane	200
Methanol	210
Ethanol	210
Diethyl Ether	220
Dichloromethane	220
Chloroform	240
Carbon Tetrachloride	265
Tetrahydrofuran	280 (210)
Toluene	280

UV Cut-off is the wavelength at which absorbance equals 1 AU

峰寬的影響 (Peakwidth)



Logical Troubleshooting Process Funnel

- 
- #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?

Agilent Lab Advisor



Agilent Lab Advisor for LC & CE

Logs & Results

Advanced Version

Agilent Lab Advisor for LC & CE

Service & Diagnostics

Advanced Version

Tasks

- Lab Advisor
 - System Overview
 - Configuration
 - Apps
 - Firmware Update
 - Logs & Results
- LC2
 - Service & Diagnostics
 - Instrument Control
 - EMFs
 - Status Report
- Help
 - Context Help (F1)
 - Help Topics
 - What's New?

Filter

Tests Calibrations Tools

Model	Serial #
G1312B BinPump SL	DE60555177
G1367C HiP ALS SL	DE60555138
G1316B TCC SL	DE60555164
G1315C DAD SL	DE55055142
G4208A 1200 Instant Pilot	DE64458986

Thermostat Test

- Temperature Calibration
- Temperature Two Point Calibration
- Diagnostic Buffers
- Module Infos

Name: **Thermostat Test**

Approx. Time: 6 min

Description: Evaluates the cooling and heating performance of the two peltier elements.

more... Run

Connection Address: 10.3.0.15

Version B.02.01 [028] - Advanced | Licenses 8/10

Agilent 耗材目錄訊息

HPLC Troubleshooting			
Symptom Type	Possible Cause	Solution	
Negative peaks	RI detector – solute refractive index less than solvent	No problem; reverse polarity to make positive	
	UV detector – solute absorbance less than mobile phase	Use mobile phase with lower UV absorbance; do not recycle solvent too long	
Noisy baseline	Random – Contaminant buildup	Flush column; cleanup sample; use HPLC-grade solvent	
	Continuous – Detector lamp problem	Replace UV lamp (lasts 1000 hrs)	
	Occasional – External electrical interference	Use voltage stabilizer for LC system	
	Sample volume too large	Injection volume should be 1/6 when mobile phase used for injection	
Peak doubling	Injection solvent too strong	Use weaker injection solvent or mobile phase	
	Blocked frit	Replace and use 0.5 µm porosity in-line filter	
	Column void or channeling	Replace column; for some columns, fill in void with packing	
	Unswept injector flowpath	Replace injector rotor	
	Void at head of column	Replace column, top off column with packing	
	Column overloaded with sample		Use higher capacity stationary phase
			Increase column diameter Decrease sample size
	Single peak – interfering components	Sample cleanup; prefractionation	

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