

Acquity
Ultra Performance LC™

培训教程 (UPLC仪器原理)



ACQUITY UPLC™系统

通过设计实现超能



Acquity™
Ultra Performance LC

检测器:

光学或ELSD及质谱
可调UV 或二极管矩阵

FLR

为UPLC™优化的检测池
高速检测

样品管理:

低扩散 XYZZ' 形式
快循环时间
低交叉污染
可使用板或样品瓶
可选样品组织器



色谱柱管理器:

加热/冷却柱温控制
革新枢轴转动设计
多个色谱柱出口至检测器位置

二元溶剂管理器:

高压混合
二元梯度
四种溶剂选择
在线脱气
低扩散设计
UPLC 压力能力

ACQUITY UPLC™系统

技术创新诸要素



- ! 小颗粒杂化填料 (**1.7 μ m**)
- ! 更高耐压(达**15000Psi**)、低系统体积的输液单元
- ! 降低自动进样器的进样周期时间、减少交叉污染
- ! 高速检测器，光学及质谱
- ! 新的色谱单元之间的通讯协议
- ! 功能齐全的诊断能力
- ! 为系统完整性而设计的软件



创新

整体技术设计的系统

Waters
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ACQUITY UPLC



- 检测器
- 样品管理器
- 色谱柱管理器
- 二元溶剂管理器
- 样品组织器

Acquity
Ultra Performance LC™

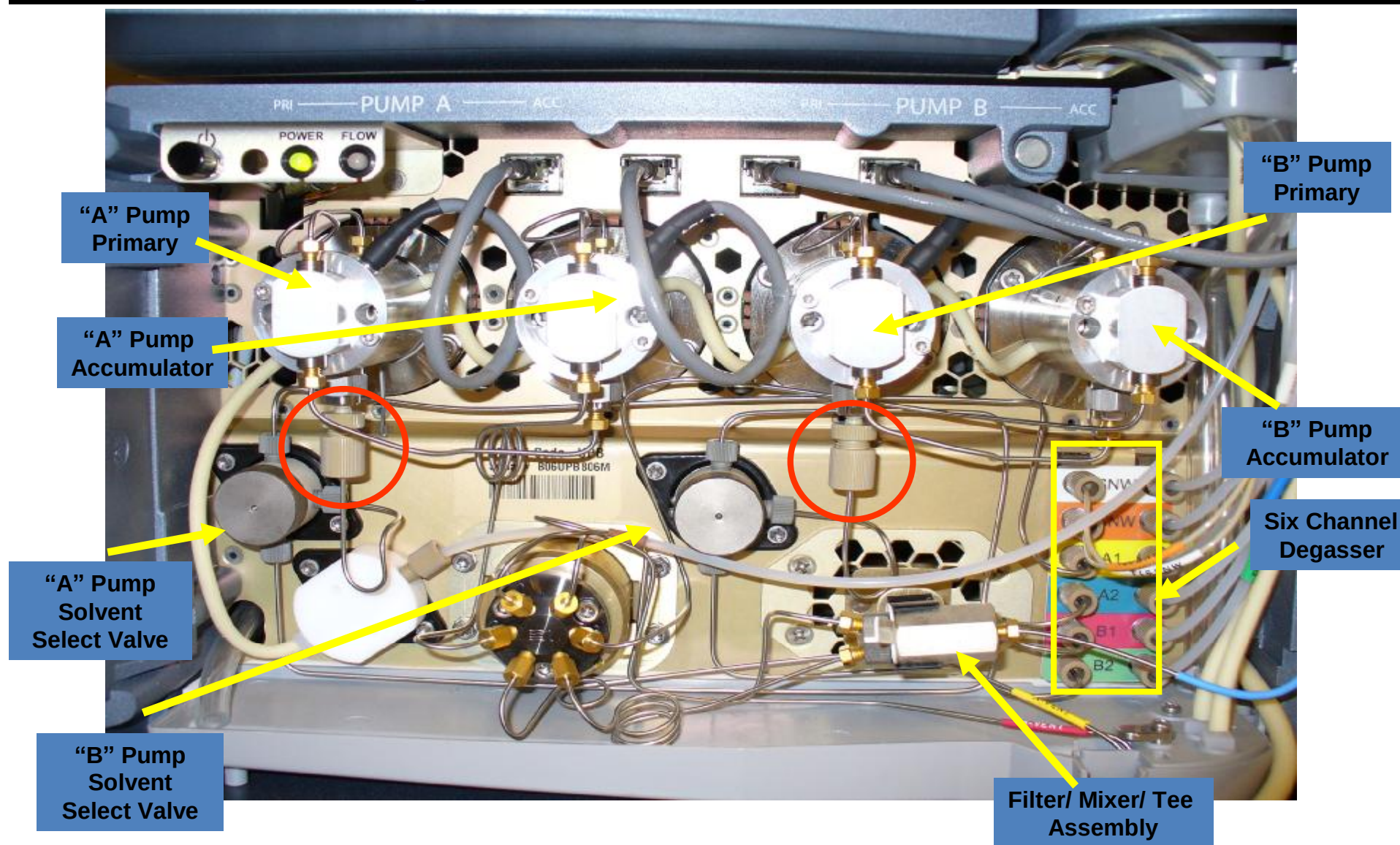
二元溶剂管理器



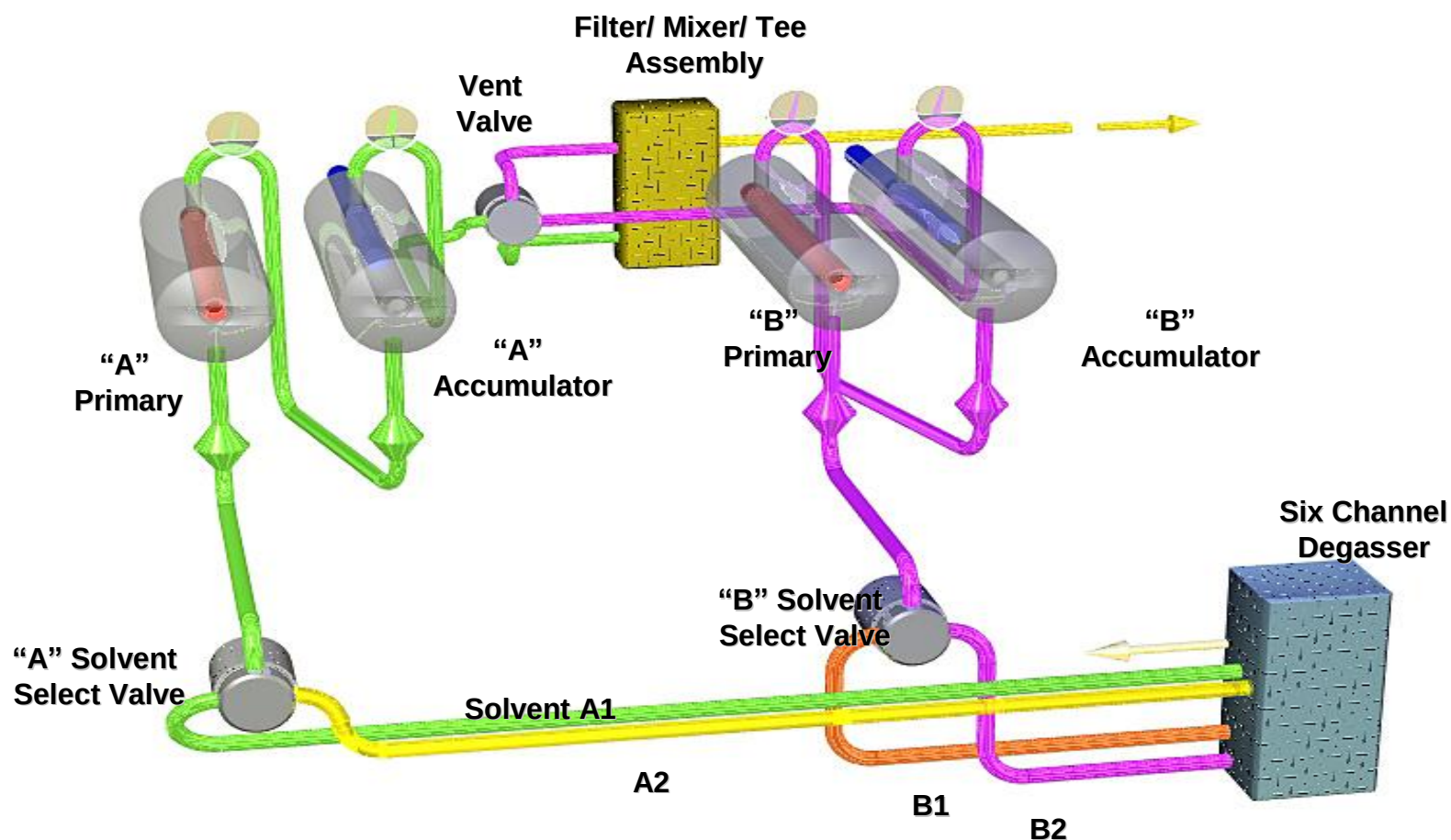
- i 低体积流路
- i 连续/平行流路
- i 两台连续流路泵并联输出二元梯度
- i 四种溶剂可选 **A1, A2, B1, B2**
 - 二元并非四元
- i **UPLC** 耐压能力
 - 高达 **15,000 psi**
- i 用户诊断



二元溶剂管理系统的流路

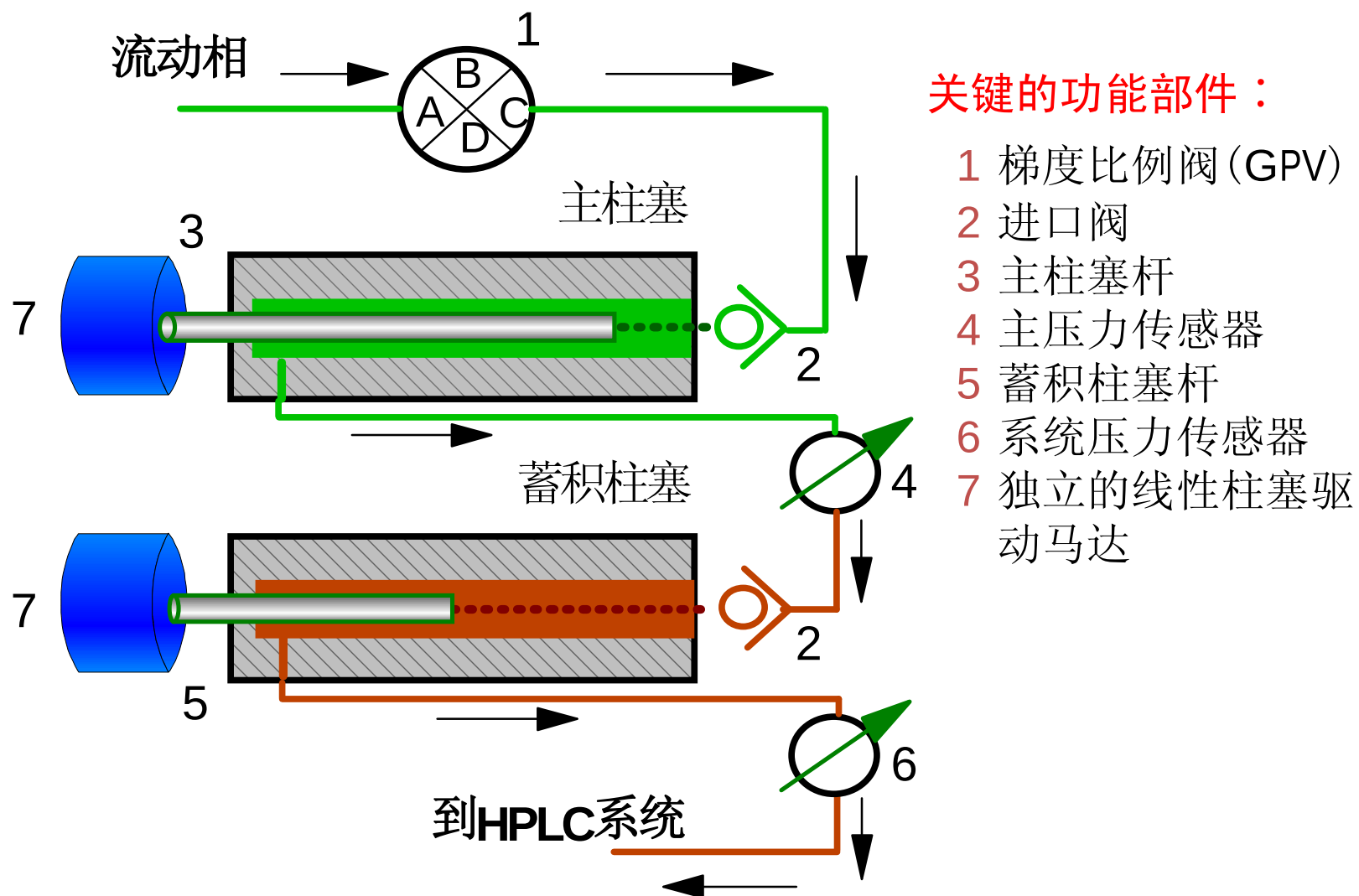


低系统体积流路设计

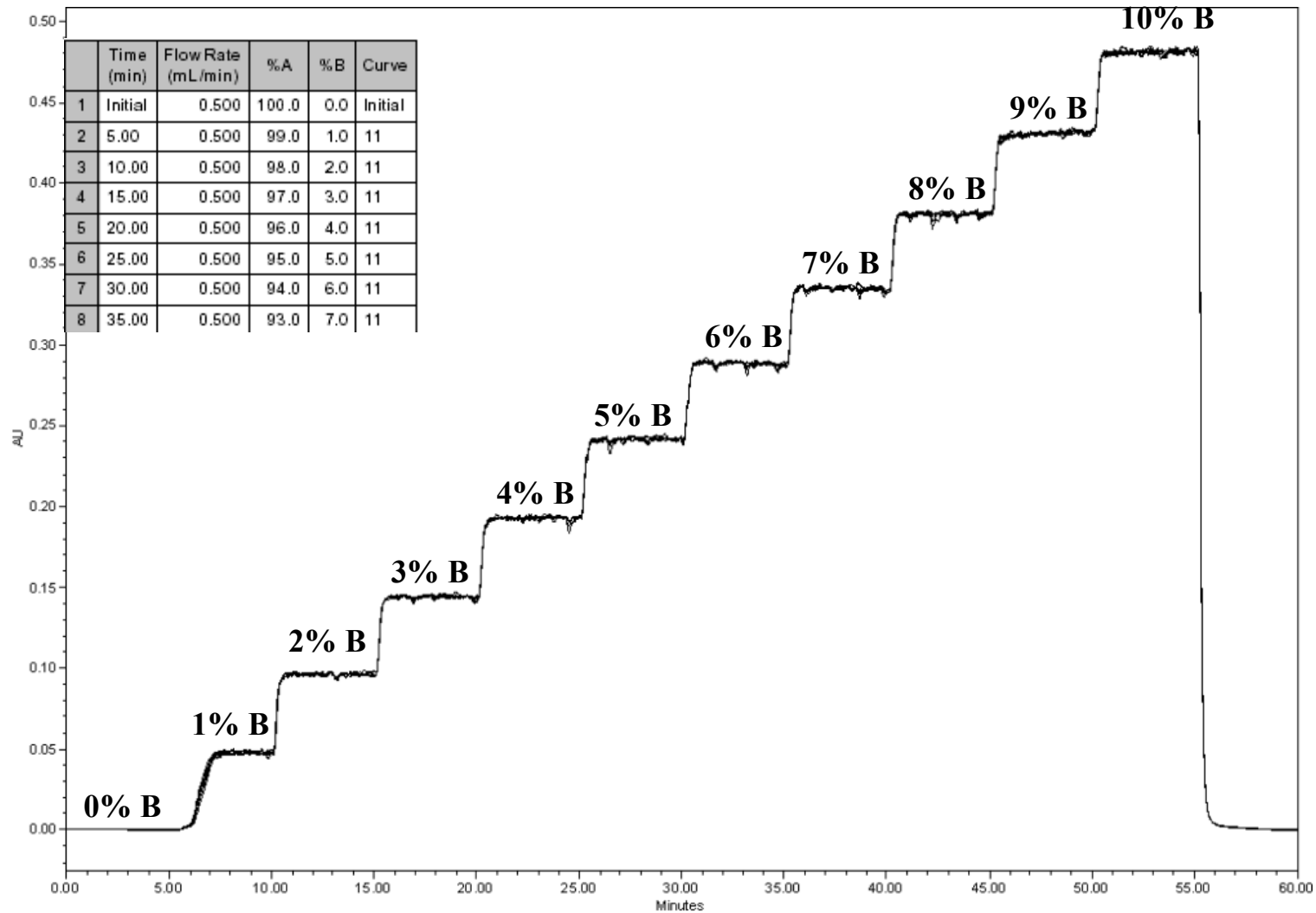


系统体积低于 $120\ \mu\text{l}$

回顾2695溶剂管理系统的创新设计



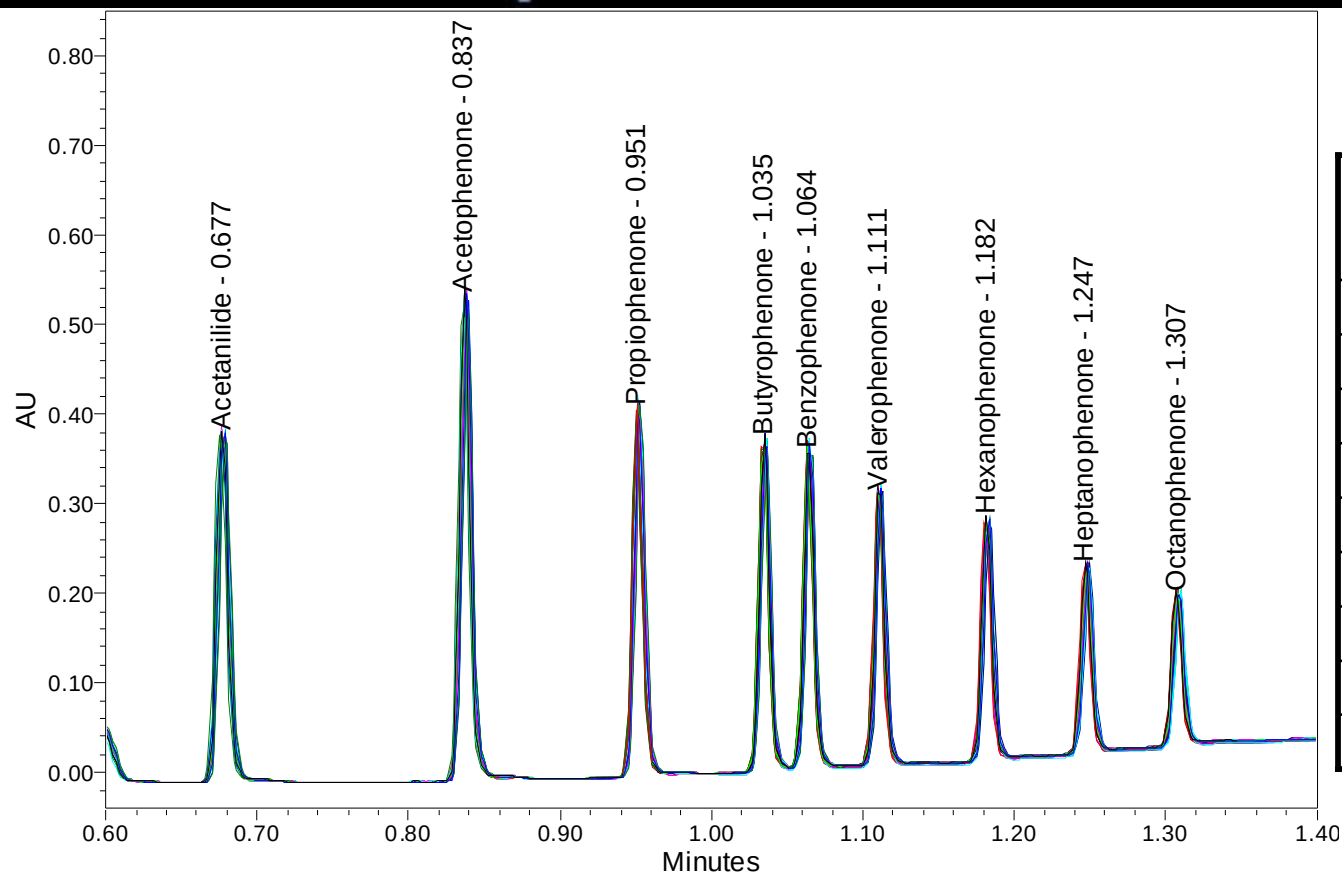
梯度组成的准度 – 1%梯度步长



梯度流速的重现性



N = 34



Peak	Standard Deviation (minutes)	Rt % RSD
乙酰苯胺	0.00083	0.122
苯乙酮	0.00085	0.101
苯丙酮	0.00082	0.086
苯丁酮	0.00080	0.077
二苯甲酮	0.00080	0.075
苯戊酮	0.00081	0.073
苯己酮	0.00079	0.067
苯庚酮	0.00082	0.066
苯辛酮	0.00090	0.069

色谱柱: **2.1×30mm, 1.7μm Bridged Hybrid**

流动相A: **0.1% FA 水溶液**

流动相B: **0.1% FA乙腈溶液**

流速: **0.6 mL/min**

梯度: **在1分钟内从5~95%的 B**

温度: **30°C**

样品: **Phenone测试混合物**

进样体积: **5μL**

检测波长: **214nm**

压力: **5,900psi**

流路



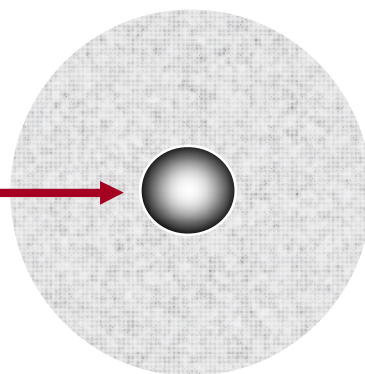
- i 流路入口线
 - 溶剂 (4 条线)
 - Strong Wash Solvent** (强洗针溶剂)
 - Weak Wash Solvent** (弱洗针溶剂)
 - Seal Wash** (密封垫清洗)

- i 流路出口线
 - Purge/waste** (冲洗/废液)
 - Needle Wash/Syringe**(洗针/注射器)
 - Plunger (seal) wash** (柱塞密封垫清洗)
 - Degasser vent** (脱气机放空)

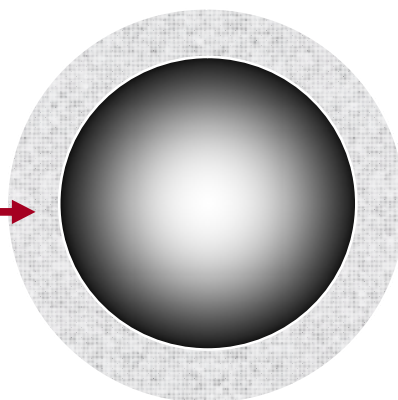
管路



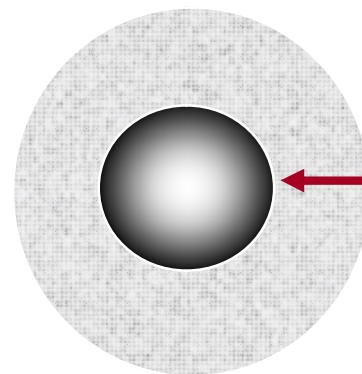
0.005"



0.020"



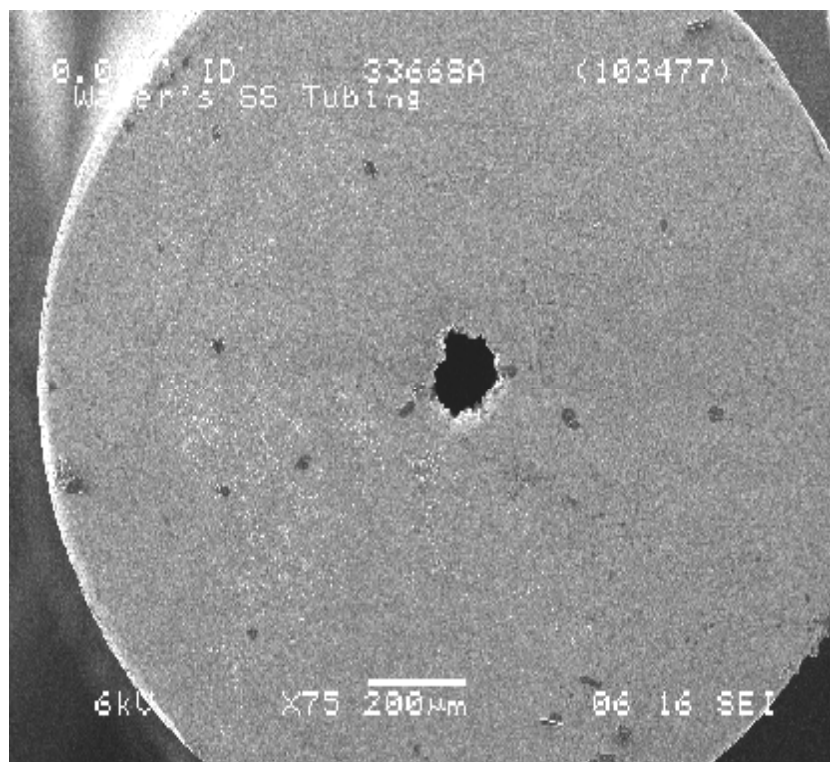
0.009"



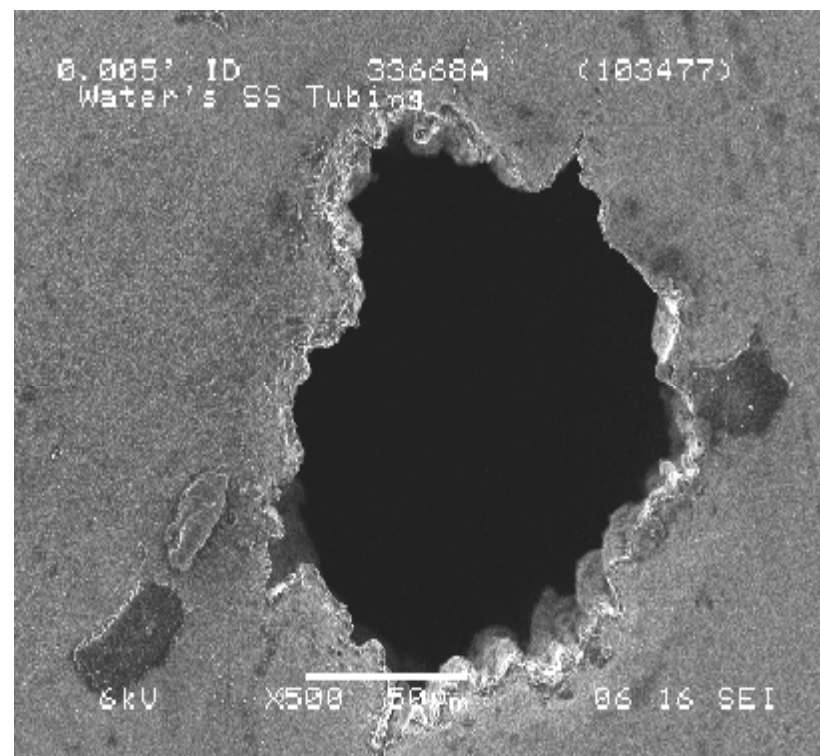
标准 0.005" 316 SS 管路



放大75 倍



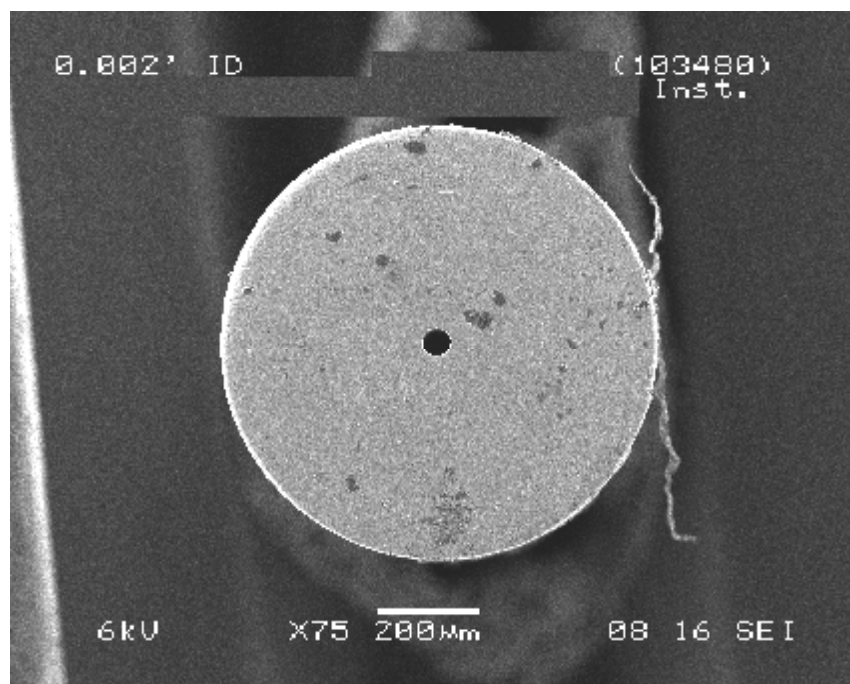
放大500倍



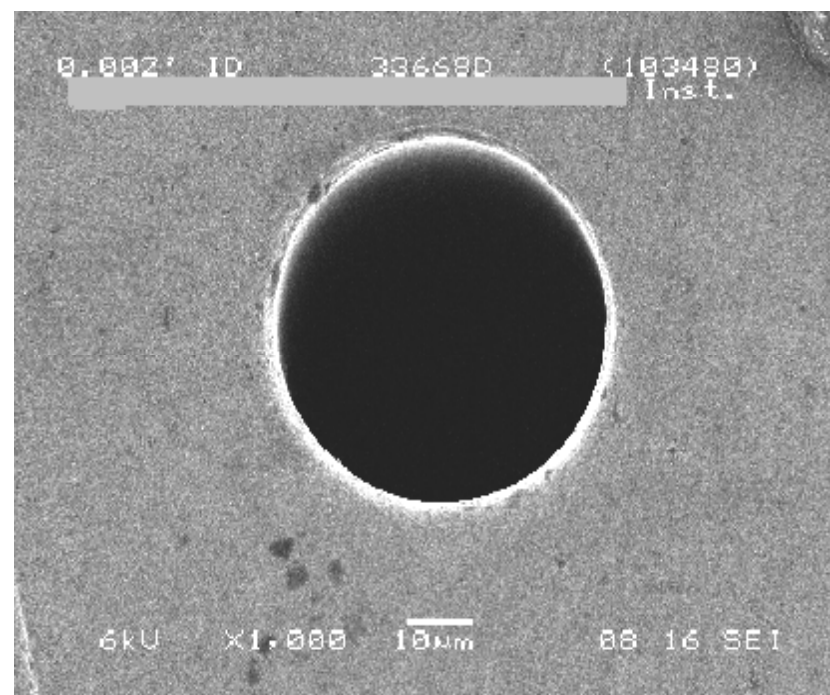
ACQUITY 0.002" 316 SS 管路



放大75 倍



放大1000倍



样品管理器



新技术 - 全新的进样程序

- i **0.1 – 50 μ L** 进样范围
- i 快速循环周期
 - 单洗针约 **30** 秒,
 - 双洗针少于 **60** 秒
- i 压力辅助样品注入
- i 针内针取样
- i 针校正传感器
- i 低样品交叉污染
- i 正反馈传感器
- i **4 到 40°C** 温控, 两块样品板
- i 选件: 样品组织器



样品瓶和样品板



Max Recovery

样品组织器 (样品管理器的选件)



- i **Sample Organizer(样品组织器)**
- i 扩展样品容量
 - **338 x 2 mL 瓶**
 - **8,448 个样品**
 - o 使用 **384 孔板**时
- i 软件控制
- i 袖珍设计

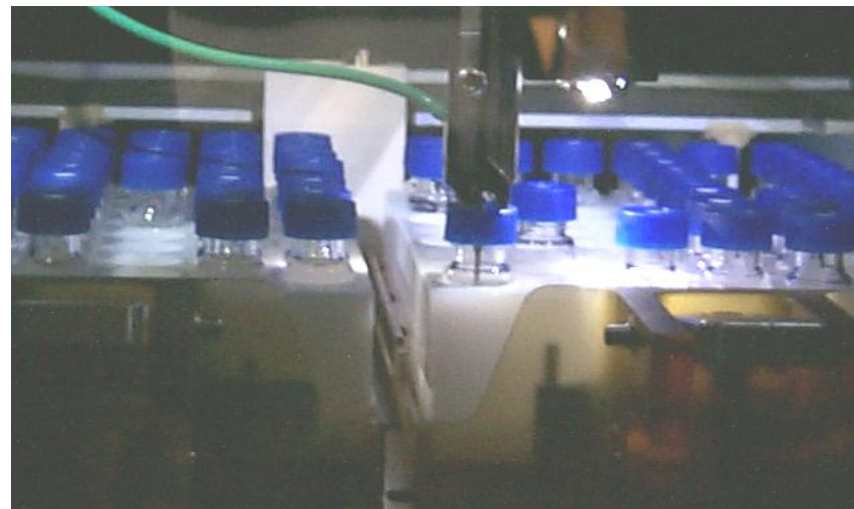


样品管理器概貌



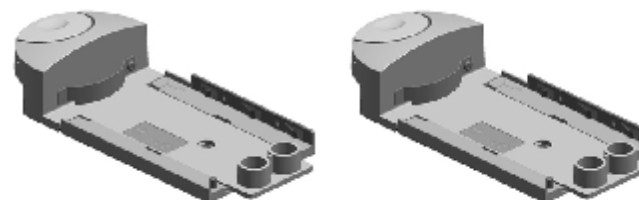
i 样品室

- 宽视场照明灯照亮样品间
 - 灯的开关可以控制
- **LED**灯在样品取样头上使你能看清它的位置



i 两个样品盘固定器，包含

- 一个符合**SBS**的托盘
- **24/48**样品瓶位
- 两个**4ml**瓶
 - 放置“**stat**”样品或标样



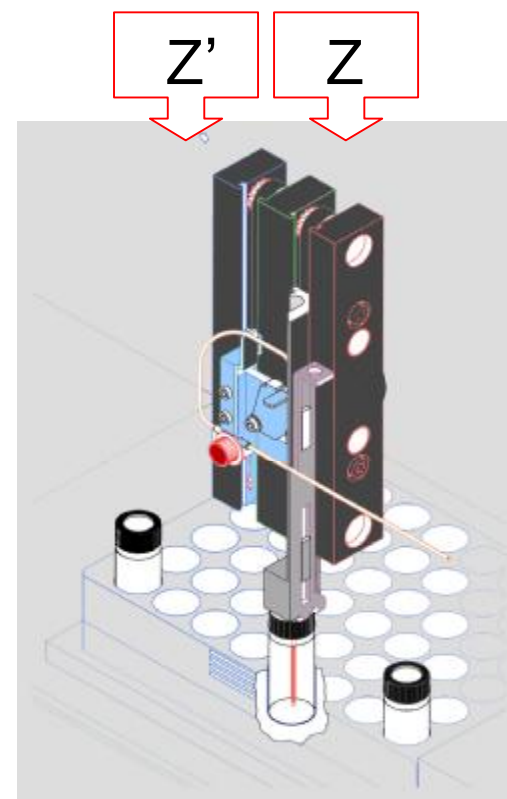
针内针设计



针内针设计的益处:

—事实上消除了取样针在穿透样品瓶盖时，瓶盖碎屑及硬物使进样针弯曲造成的麻烦

—进样针内的低扩散



3. 上样探头和吸样

极低的交叉污染



i 置换和清洗程序

- 计量注射器的置换
- 强溶剂清洗
- 用弱溶剂置换
- 弱溶剂清洗
- 清洗后的注射器再充满

— 首次空白 **<0.002%**; 后续空白 **<0.0002%**

○ 远好于: **0.005%** (指标)

ACQUITY UPLC®

可选用的loop环

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1µL

2µL

5µL

10µL

20µL

50µL

色谱柱箱 / 柱加热器



- i 与样品管理器一体化
 - **65(90)°C** 上限
- i 枢轴定位
 - 堆叠方式
 - o 光学检测器
 - 向外打开方式
 - o **MS** 检测器
 - 优化的流路距离
 - 最低扩散
 - 两侧均有色谱柱通道



转出到 **MS**入口

色谱柱箱 / eCord 技术



- 色谱柱历史的无纸追溯
- 固化的色谱柱出厂数据
 - 唯一的色谱柱**ID**
 - 分析合格证书
 - **QC** 测试数据
- 可变的色谱柱使用数据
 - 色谱柱使用数据
 - 在柱寿命期间全程收集
- 固定器, 绳索和芯片永久地系在柱子上
- 微芯片用**16mm**硬币形不锈钢罐包裹
- 永久性读/写存储器



色谱柱在线过滤器

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50 mm
Column



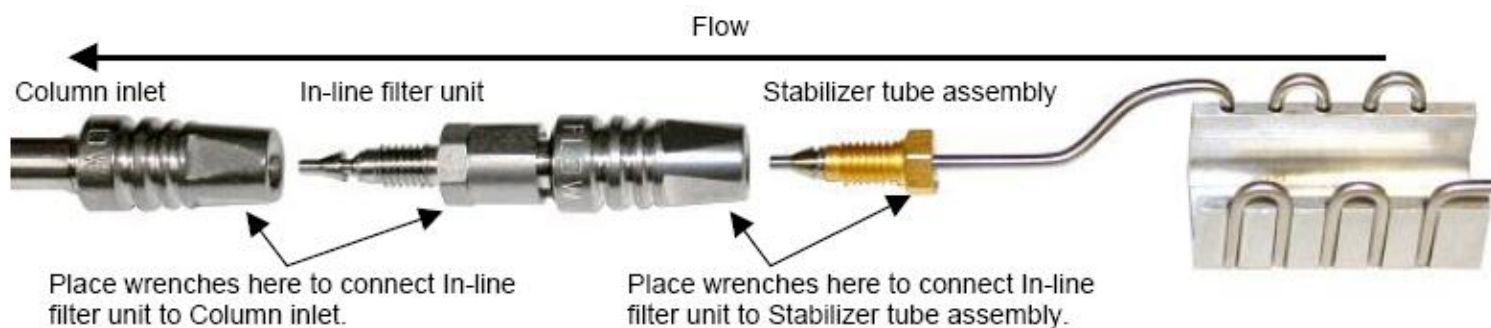
100 mm
Column

色谱柱管理器 内置柱预热器及柱温稳定器

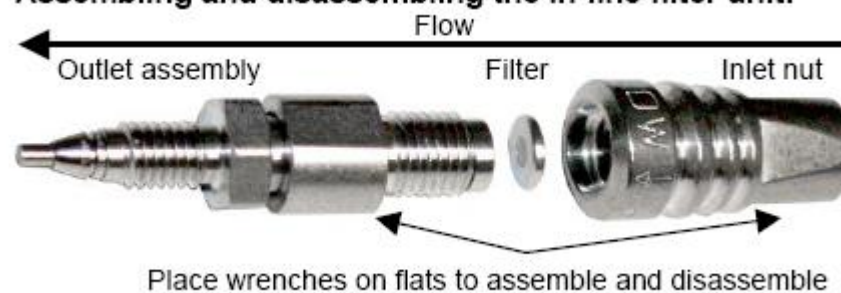


ACQUITY 在线过滤器

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Assembling and disassembling the in-line filter unit:

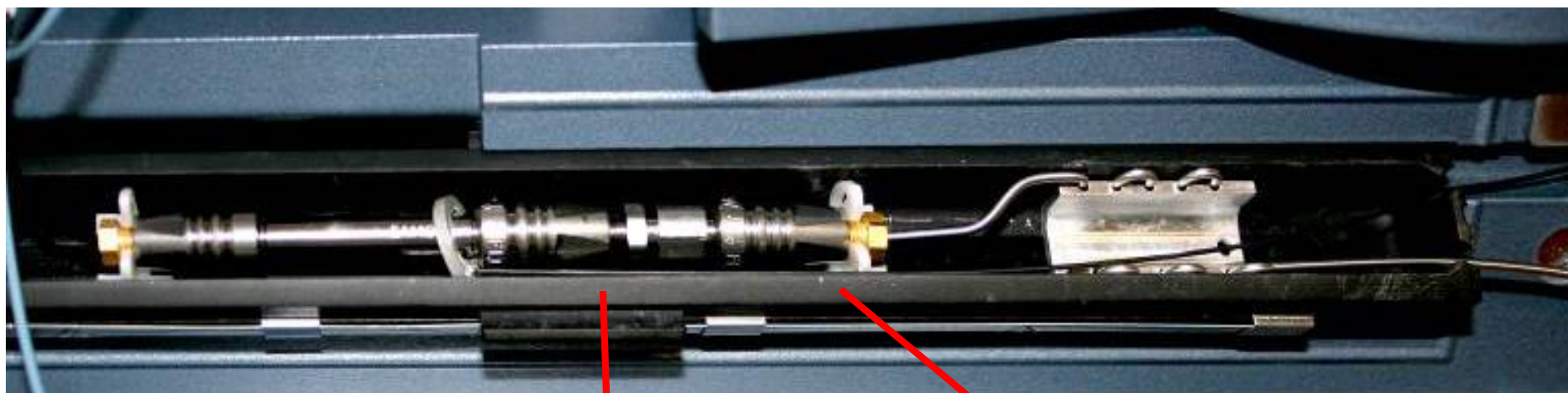


色谱柱在线过滤器

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i **PN 205000343**

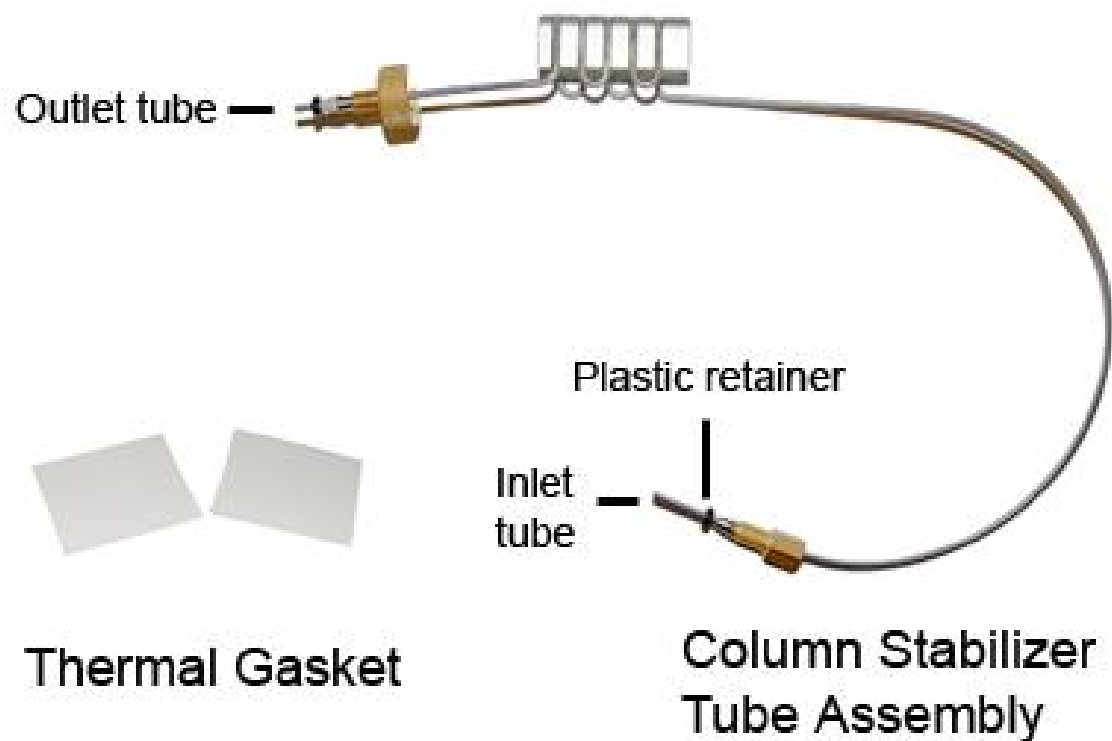
i **0.2 μm可更换滤芯, 5个/包, PN 700002775**



50/100mm色谱柱用稳定器

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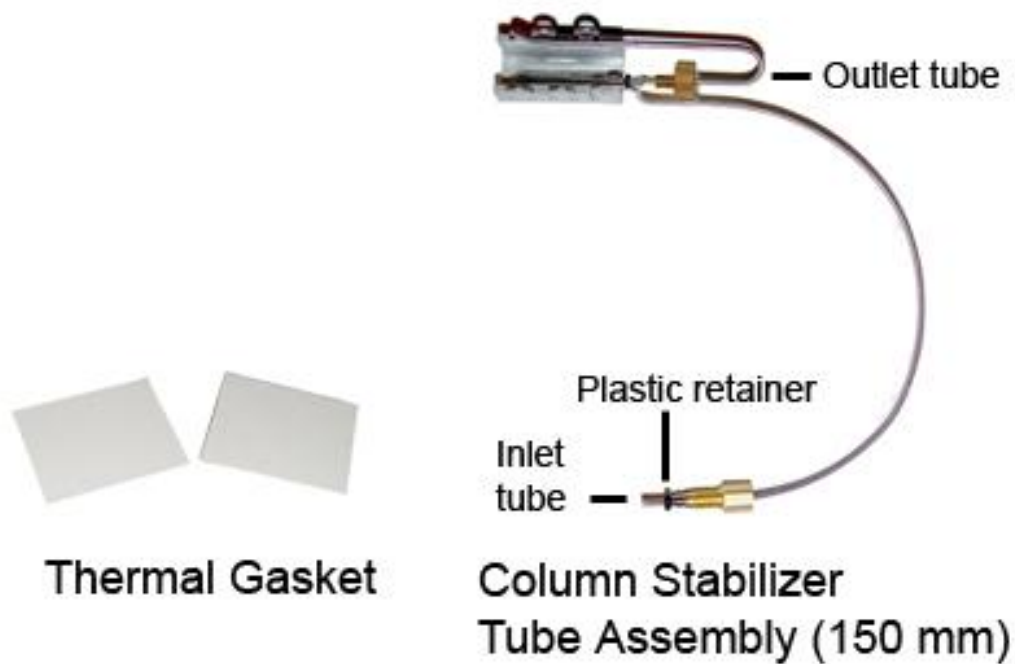
柱温稳定器组件, 50/100 mm (# 205000291)



150mm色谱柱用稳定器



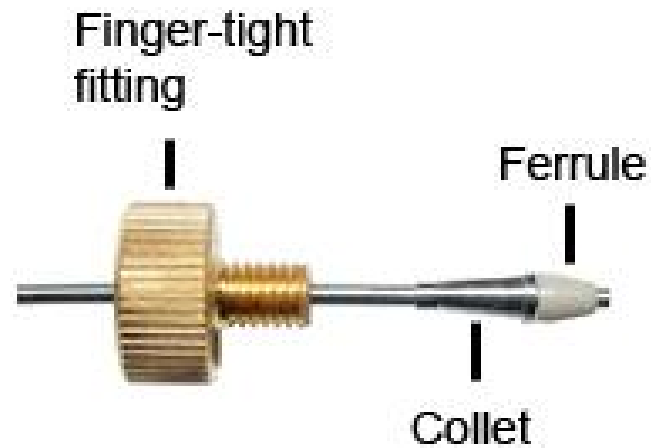
柱温稳定器组件, 150 mm (# 205000365)



高压手紧接头



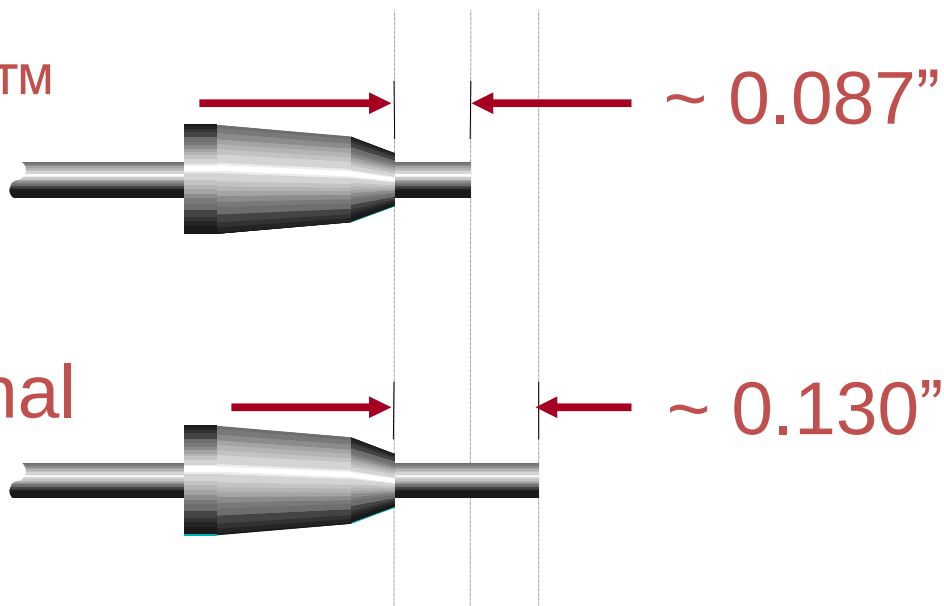
手拧紧可重复使用的 **10-32** 接头组件(# **700003139**)



ACQUITY UPLC™ 的接头



ACQUITY UPLC™



Waters® Traditional

UPLC 检测技术的挑战



- i 更快流出的峰需要：
 - 更高的数据采集速度
 - 更低的池体积
- i 较小的峰需要最大的 **S/N**
 - 高的光通量/传输
 - 更快的数字滤波时间常数
- i **UPLC** 分离需要低扩散流通池
 - 保持峰形
 - 不产生高反压



UPLC™流动池设计

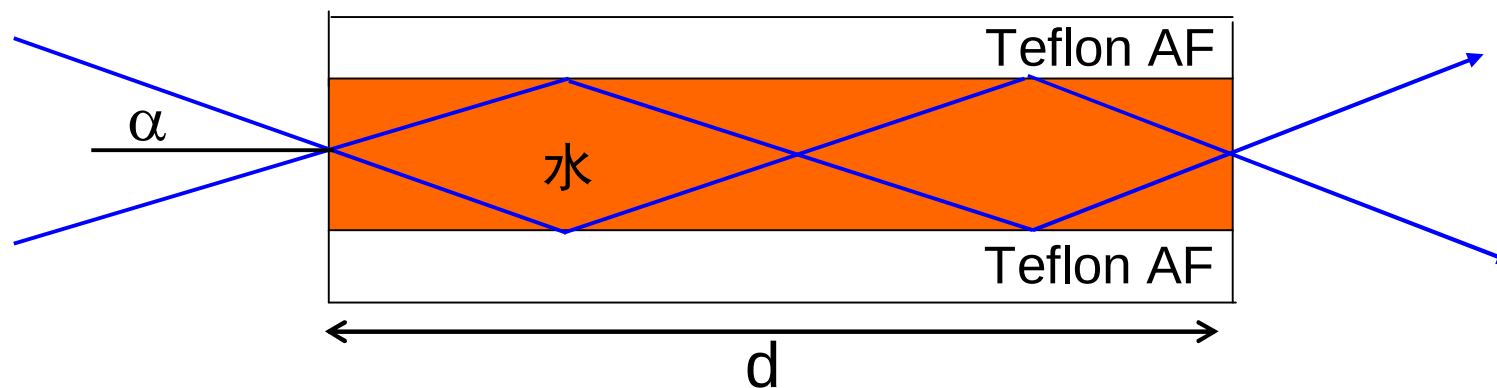


i 光引导的UPLC™流动池

—**10mm**光程长

—流动池通道即低吸收系数的 **Teflon AF** 管的内部

—壁上全内反射，犹如光纤涂层



UPLC™流动池设计

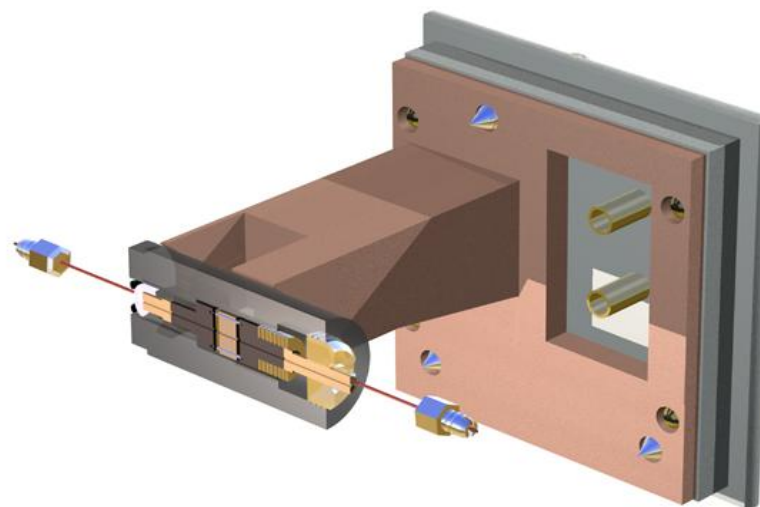


- i 分析型流通池 (**10mm, 500nL**)

- 更佳的色谱分离度
- 推荐用于与质谱串联时, 因为其体积小

- i 高灵敏度流通池 (**25mm, 2400nL**)

- 追求最高的灵敏度
- 更高的峰高(**Beer's law**)
- 通常有更好的信噪比



$$A = \epsilon \cdot c \cdot l$$

反压调整器



- i 反压调整器(**250Psi**)能确保溶解在流动相中的气体不会产生气泡,从而保证基线平稳不受气泡噪音的干扰
- i 如果**UV**检测器是系统中的最后一个检测器,一定要装配反压调整器
- i 如果在**PDA/TUV**后面还连接有质谱或其它检测器,则无需装配反压调整器

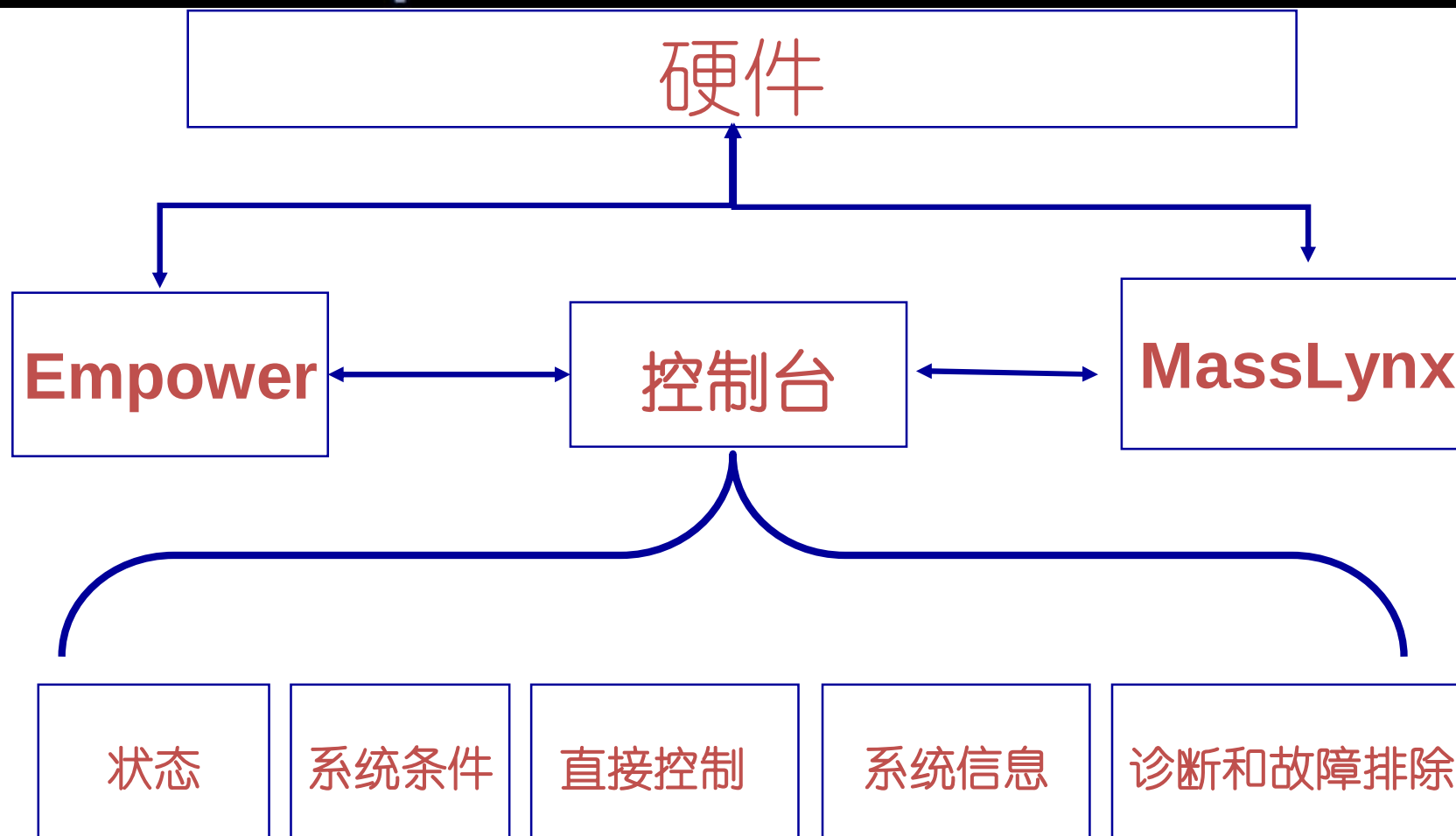
ACQUITY 对软件的要求



- i **Empower build 1154/2154**
 - **Service pack ACQUITY**
 - **Instrument Control Operating Procedure (ICOP)**
 - o **V1.1 (老版本)**
 - o **V1.30 (新版本)**
 - **Firmware**
- i **MassLynx 4.0/4.1**
 - **Service Pack 4**
 - **Instrument Control Operating Procedure (ICOP)**
 - **Firmware**

ACQUITY 的软件控制

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控制台



状态

系统条件

直接控制

系统信息

诊断和故障排除

每个模块

溶剂灌注

每个模块

每个模块

每个模块

快速浏览

HPLC

模块控制

色谱柱

快速浏览

流程图

清洗

系统设置

日志

显示图

交互式显示

冲洗

快速切换

维护计数器

性能

A/S 配置

流速/波长

模块

任务菜单

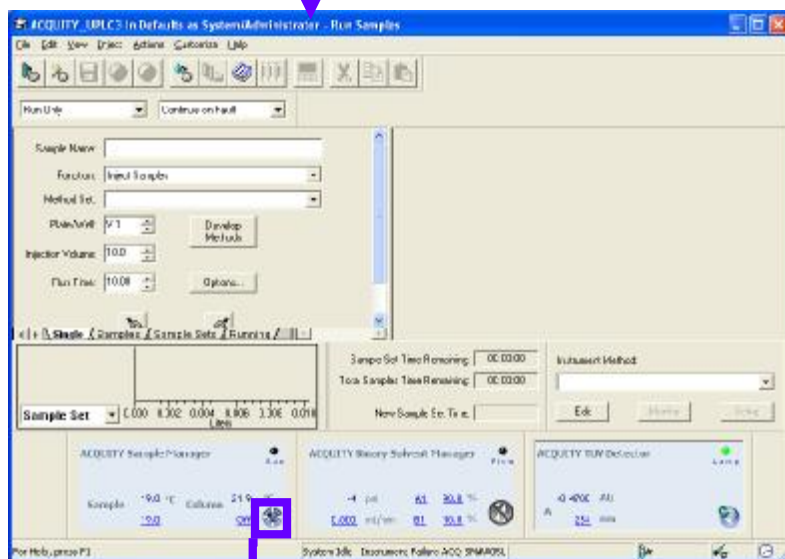


访问控制台界面

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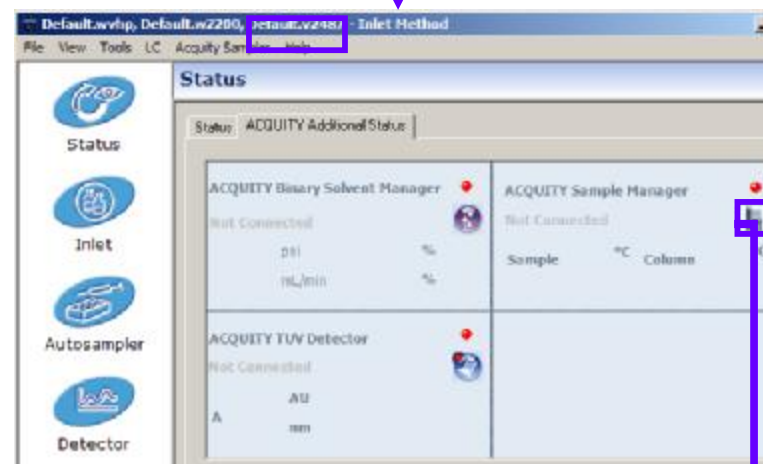
Empower

运行样品界面



MassLynx

液相方法界面



控制台图标

EMPOWER 2

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ACQUITY_UPLC3 in Beta_ELSD_092605_EMPH as System/Administrator - Run Samples

File Edit View Inject Actions Customize Help

Run Only Continue on Fault

Sample Name:

Function: Inject Samples

ACQUITY Binary Solvent Manager **Flow**

0 psi A1 100.0 %
0.000 mL/min B1 0.0 %

ACQUITY Sample Manager **Run**

Sample 23.2 °C Column 21.7 °C
Off Off

ACQUITY TUV Detector **Lamp**

0.0046 AU 0.1291 AU
A 254 nm B 210 nm

Sample Set 0.000 0.002 0.004 0.006 0.008 0.010 Liters Edit Monitor Setup New Sample Set Time:

ACQUITY Binary Solvent Manager **Flow**

0 psi A1 100.0 %
0.000 mL/min B1 0.0 %

ACQUITY Sample Manager **Run**

Sample 23.2 °C Column 21.7 °C
Off Off

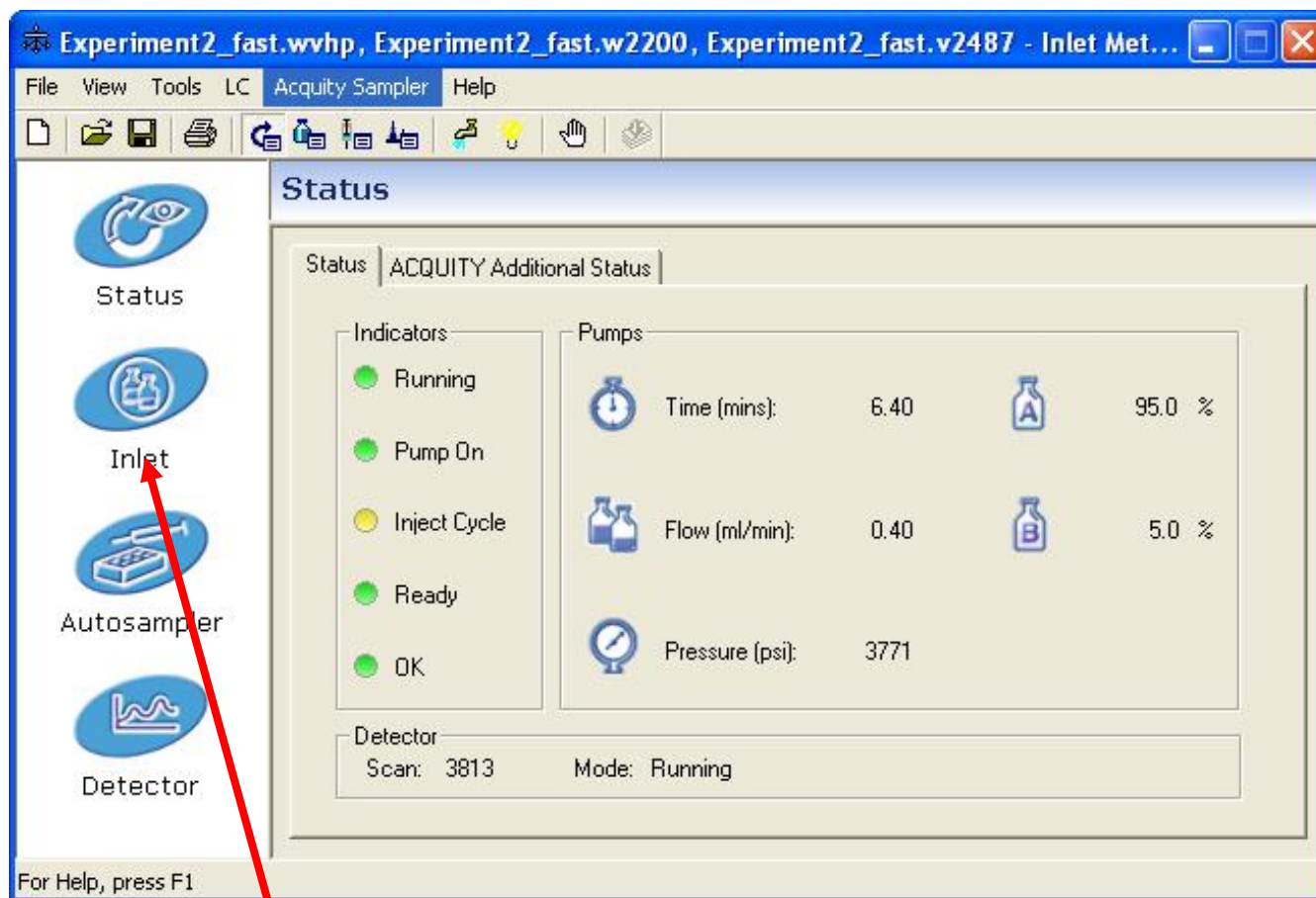
ACQUITY TUV Detector **Lamp**

0.0046 AU 0.1291 AU
A 254 nm B 210 nm

For Help, press F1 System Idle - Instrument Failure ACQ-BSM#A05

Masslynx 4.1

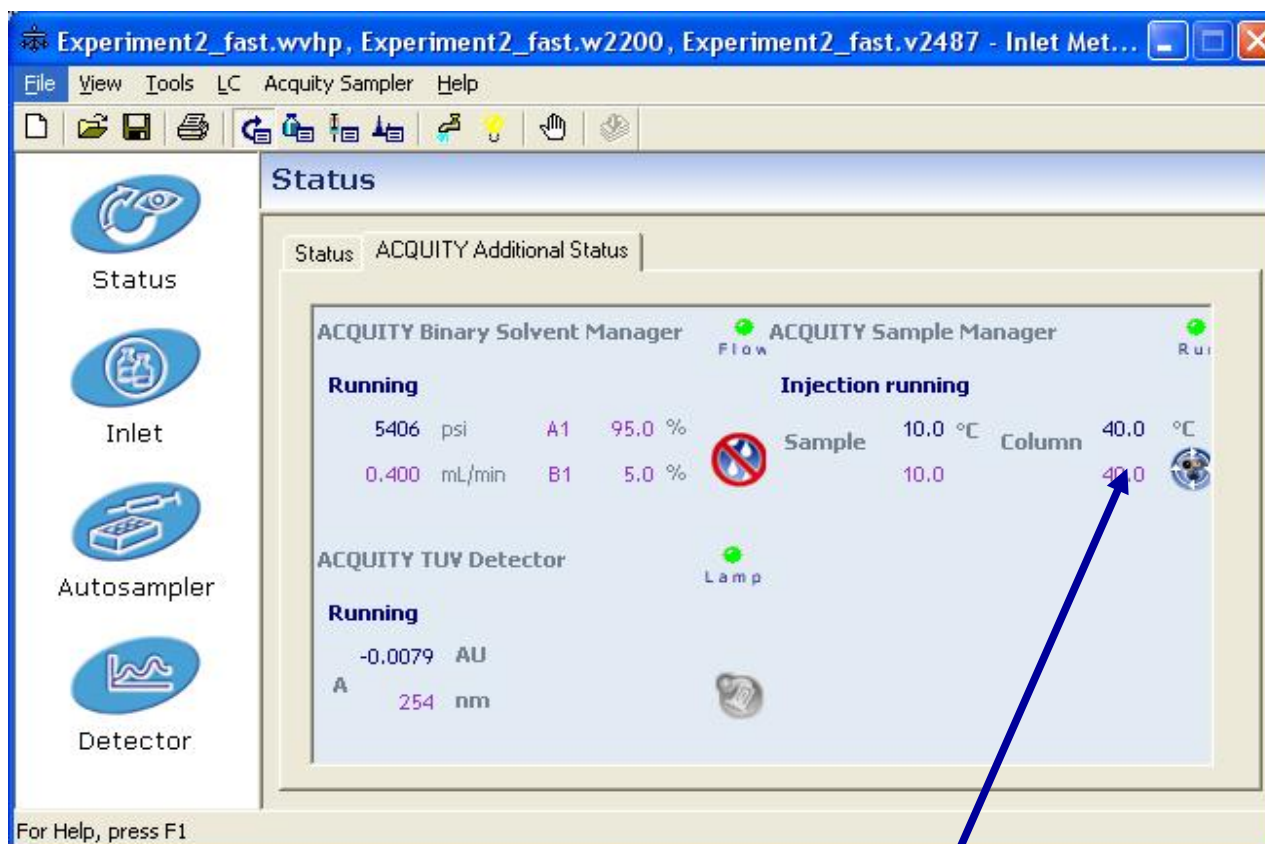
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选 Inlet

MassLynx 4.1

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ACQUITY 控制台快捷键

控制台的使用

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ACQUITY UPLC Console for System ACQUITY_UPLC3 on Node CORE-EMPOWER-27 - [System]

Control Configure Maintain Troubleshoot Help

ACQUITY UPLC System

- Binary Solvent Manager
- Sample Manager
- Sample Organizer
- TUV Detector
- Column
- Plots
- Maintenance Counters
- Logs

System Status

Acquity

A1 B1

1 psi

0.000 mL/min

A1 100.0 %

0.0 B1

Temperature

Column 21.7 °C Off

Sample 23.9 °C Off

Room 21.7 °C

TUV Detector

A 0.7550 AU

230 nm

Flow

Stop Flow

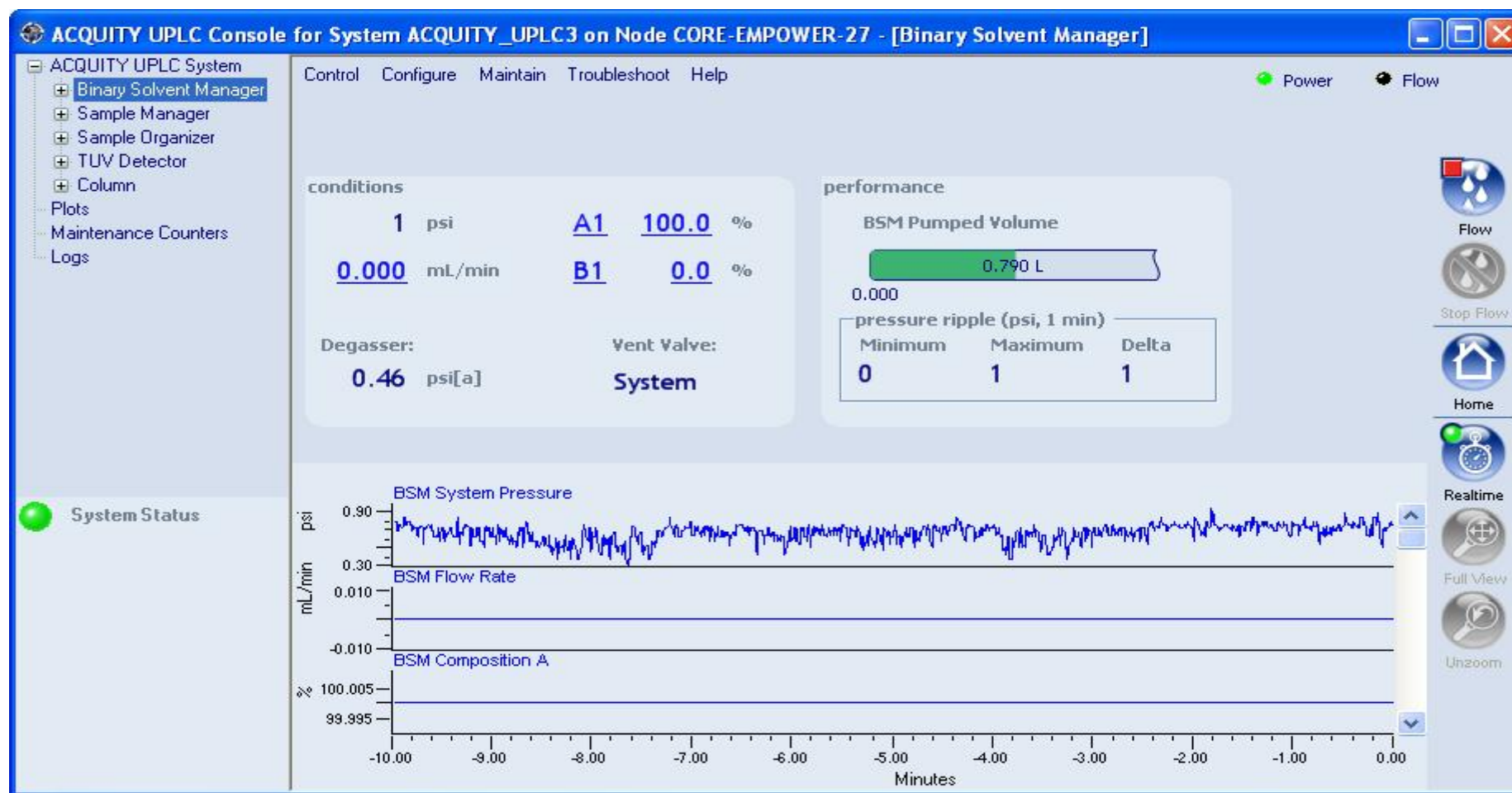
Lamp

Auto Zero

1x λ Mode

BSM状态: 泵的主控制界面

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BSM系统准备：灌注泵

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ACQUITY UPLC Console for System ACQUITY_UPLC3 on Node CORE-EMPOWER-27 - [Binary Solvent Manager]

Control Configure Maintain Troubleshoot Help

Set flow...
Stop flow
Prime A/B solvents...
Prime seal wash
Reset BSM

0.000 mL/min

A1 100.0 %
B1 0.0 %

Degasser: 0.46 psi[a]

Vent Valve: System

performance

BSM Pumped Volume

0.790 L

0.000

pressure ripple (psi, 1 min)

Minimum	Maximum	Delta
0	0	0

Prime A/B Solvents

Solvent Line(s) to Prime

☒ A A1

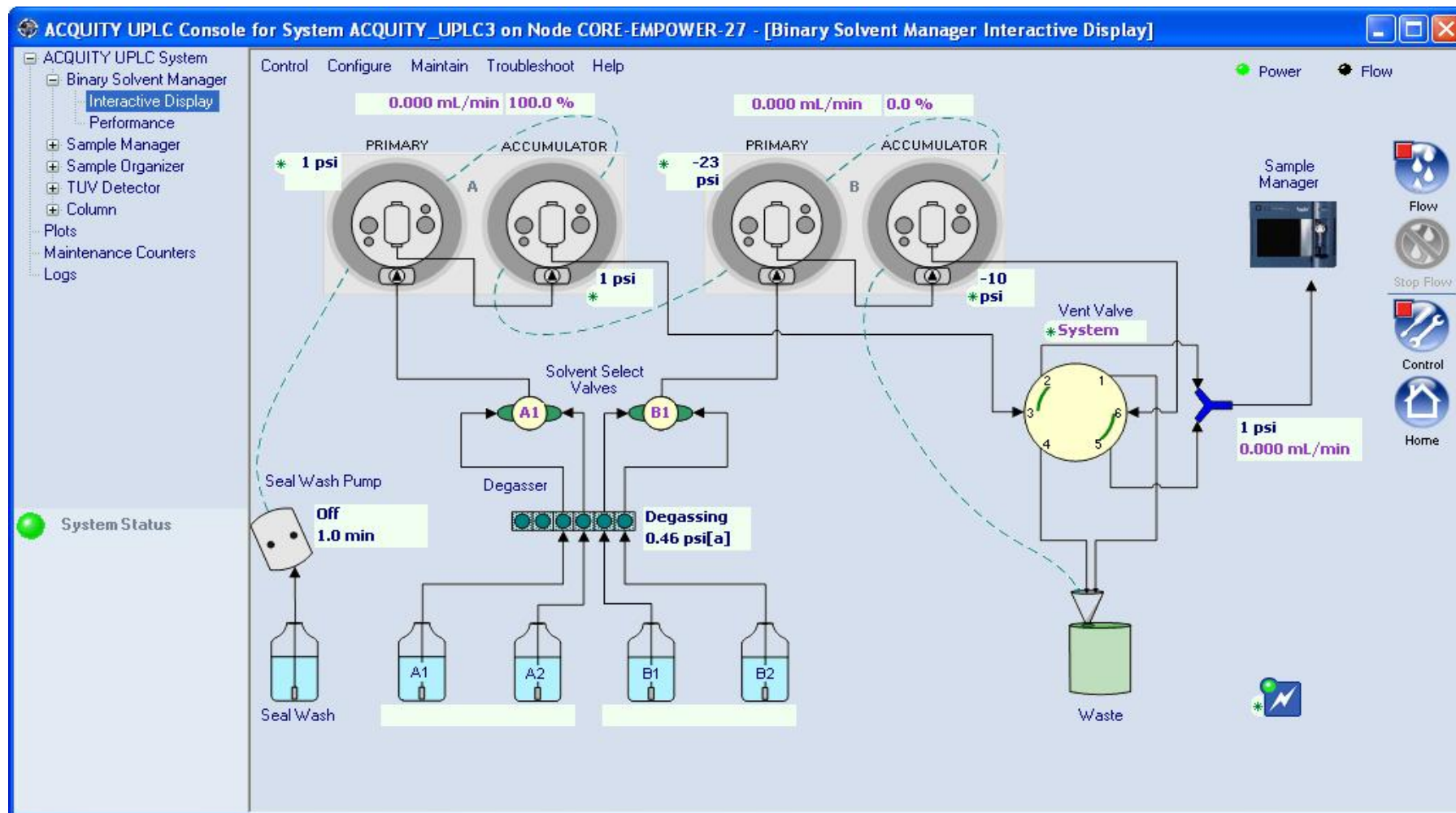
☒ B B1
B2

Time: 1.0 min

Start Close

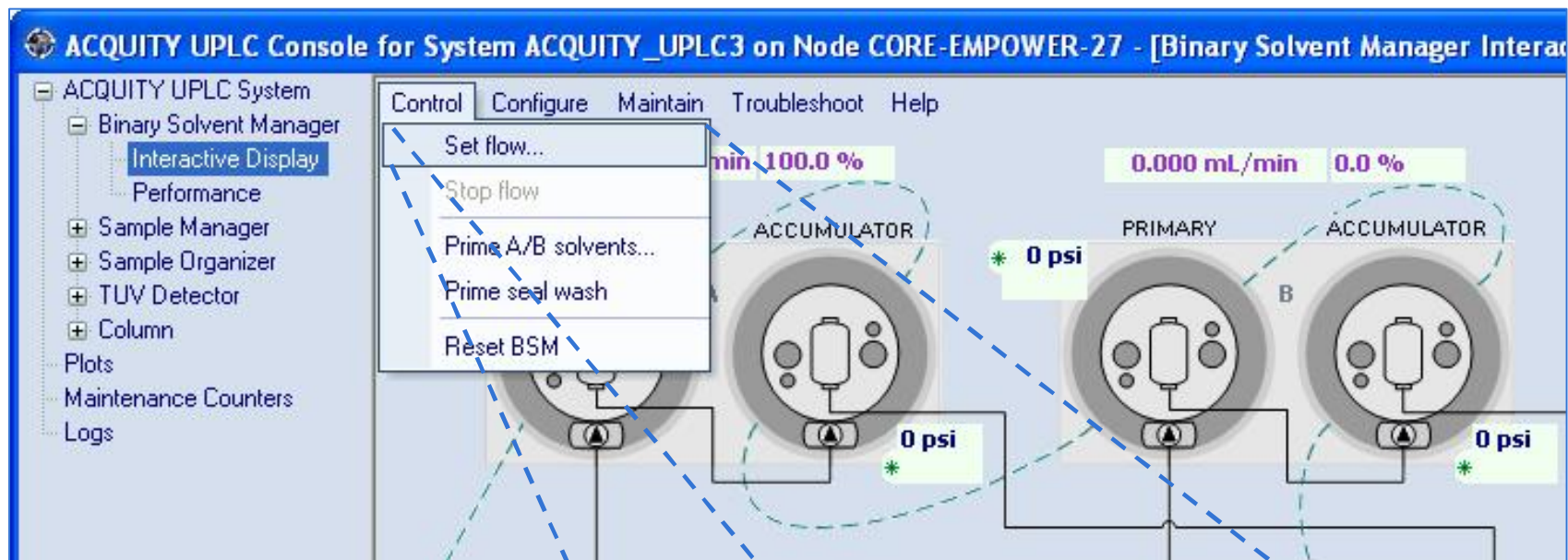
BSM状态:泵的交互式显示

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BSM: 设定流速

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Set Flow

Flow:
0.100 mL/min

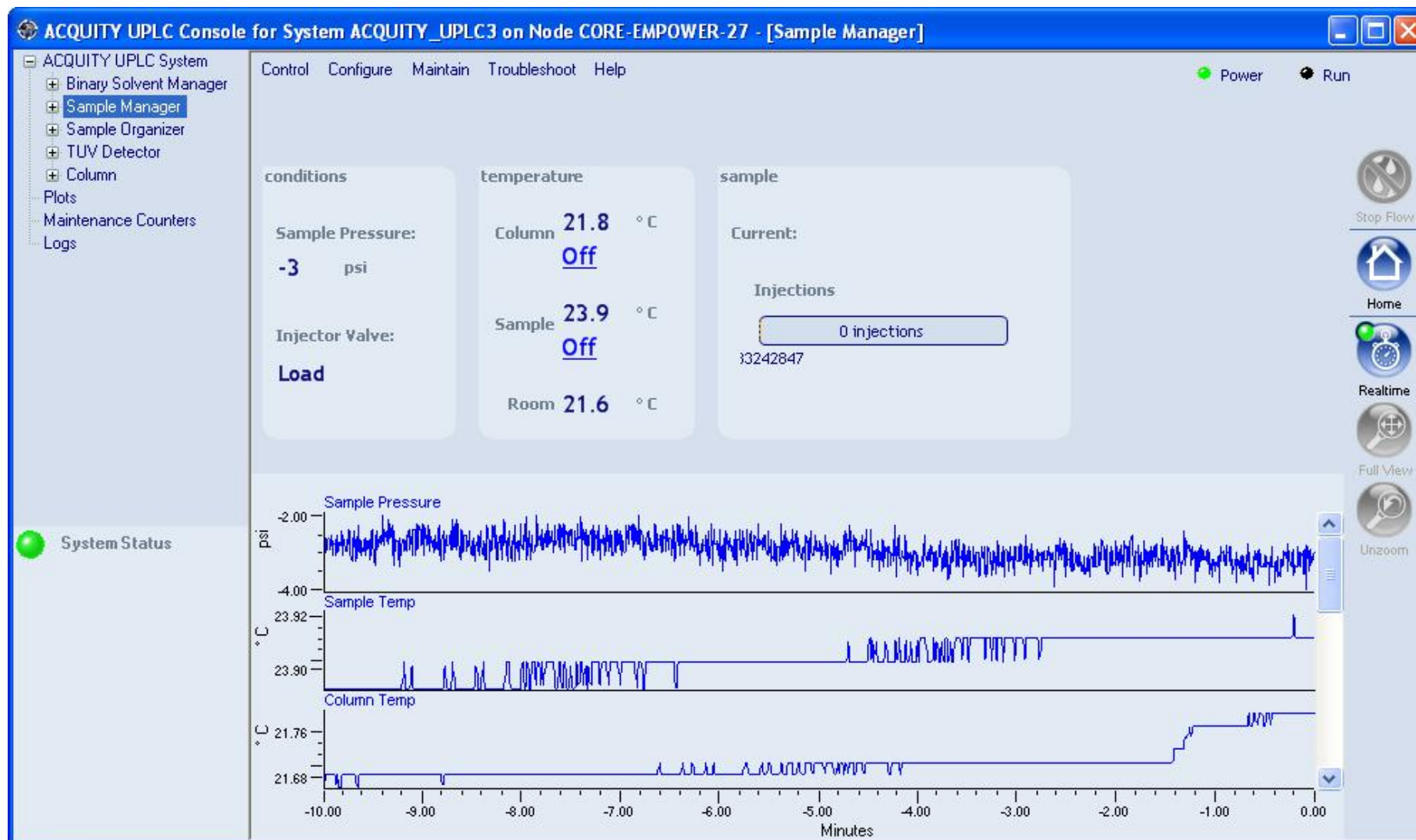
Solvent A:
0.0 %
A1

Solvent B:
100.0 %
B1

✓ ✗ ?

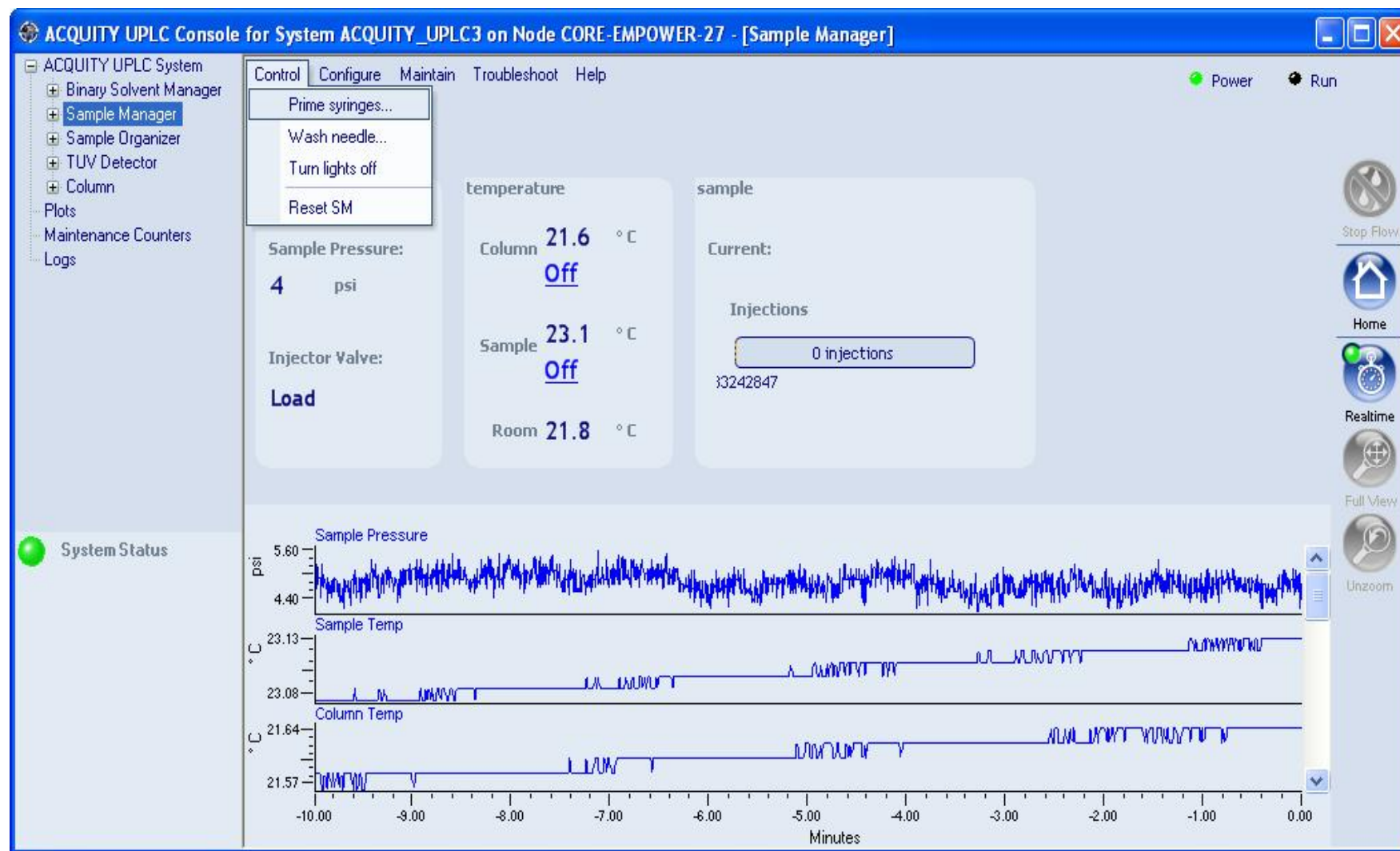
SM状态:样品管理器控制界面

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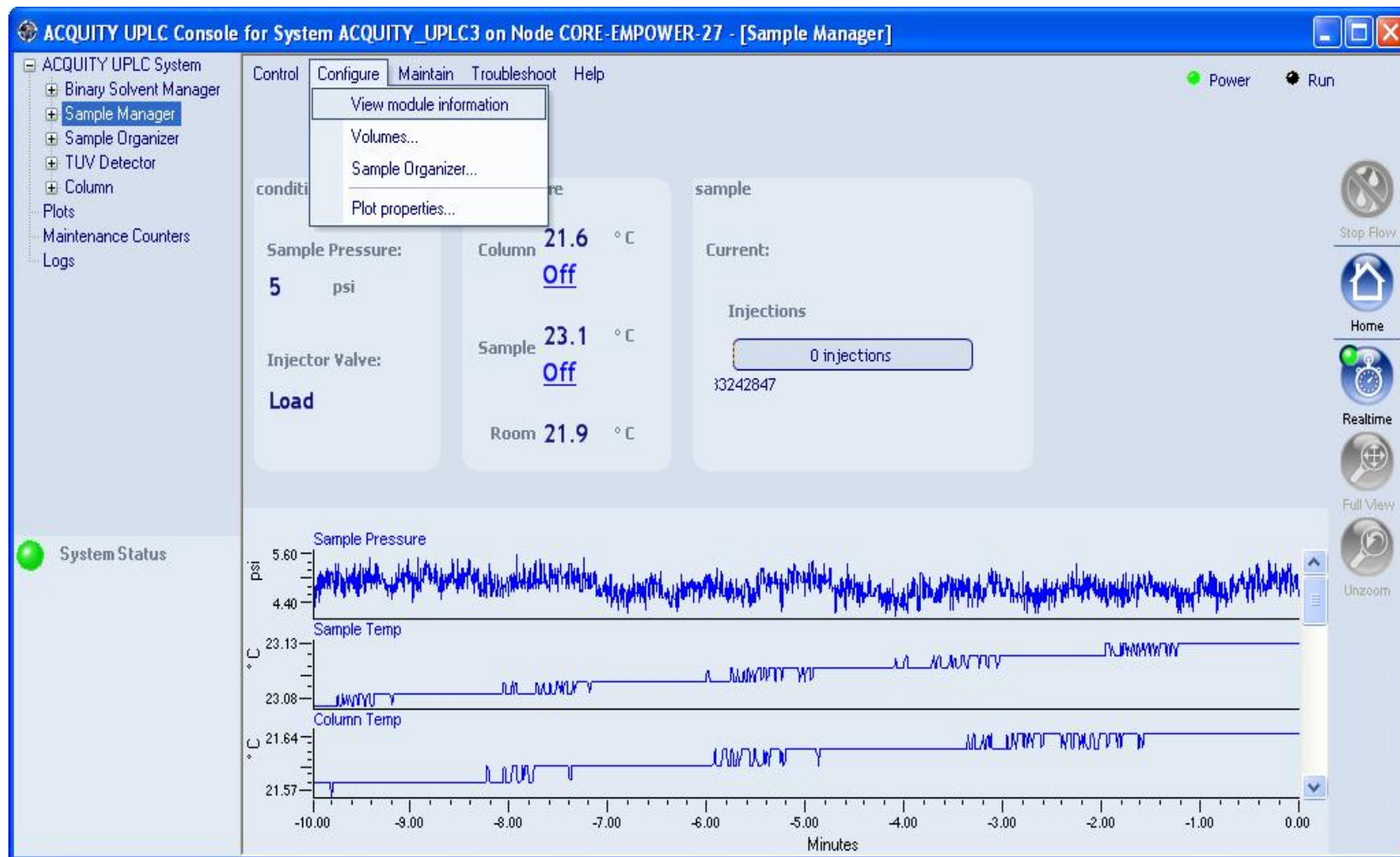
SM系统准备:清洗注射器

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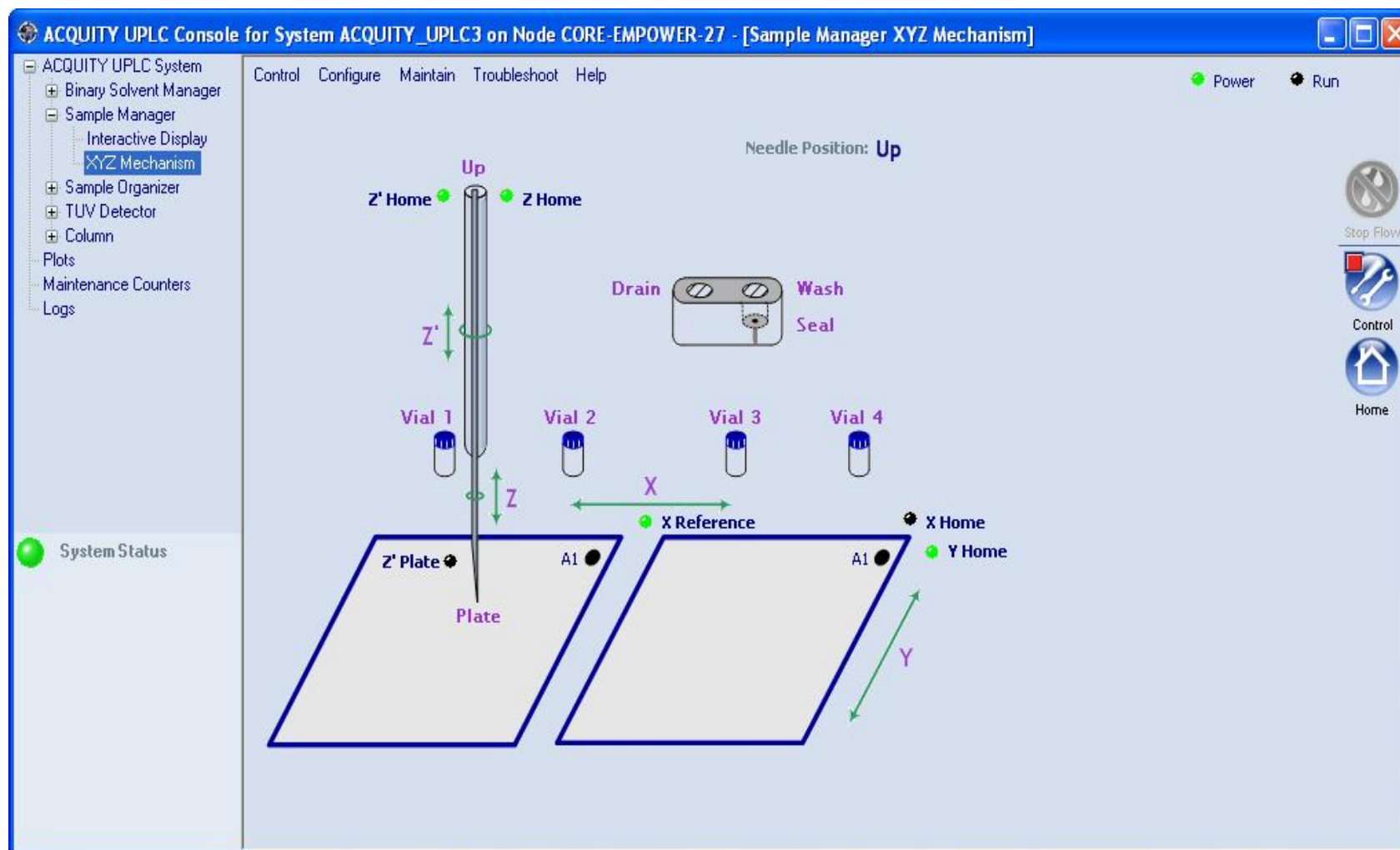
SM系统准备:配置

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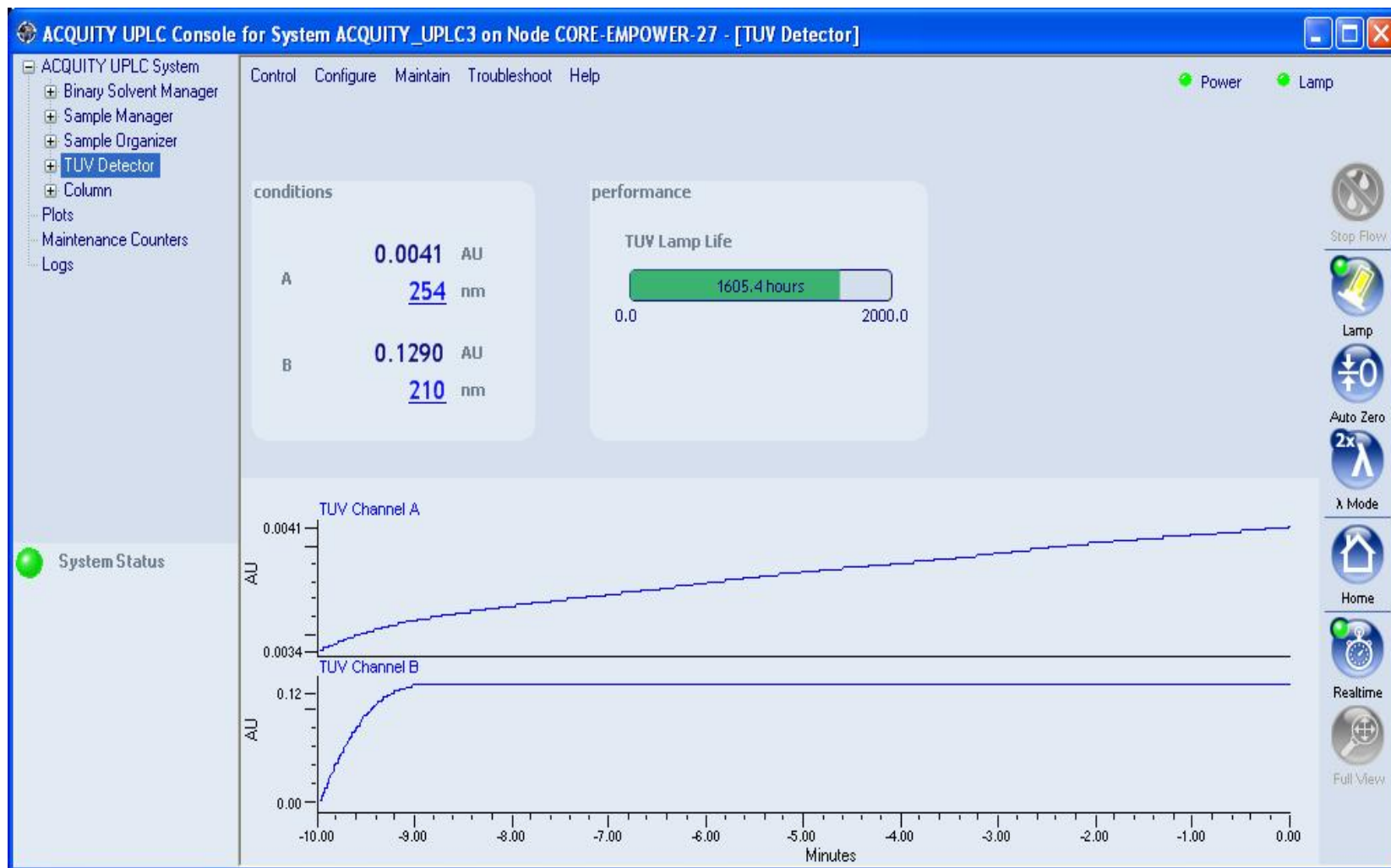
SM状态:样品管理器的控制界面

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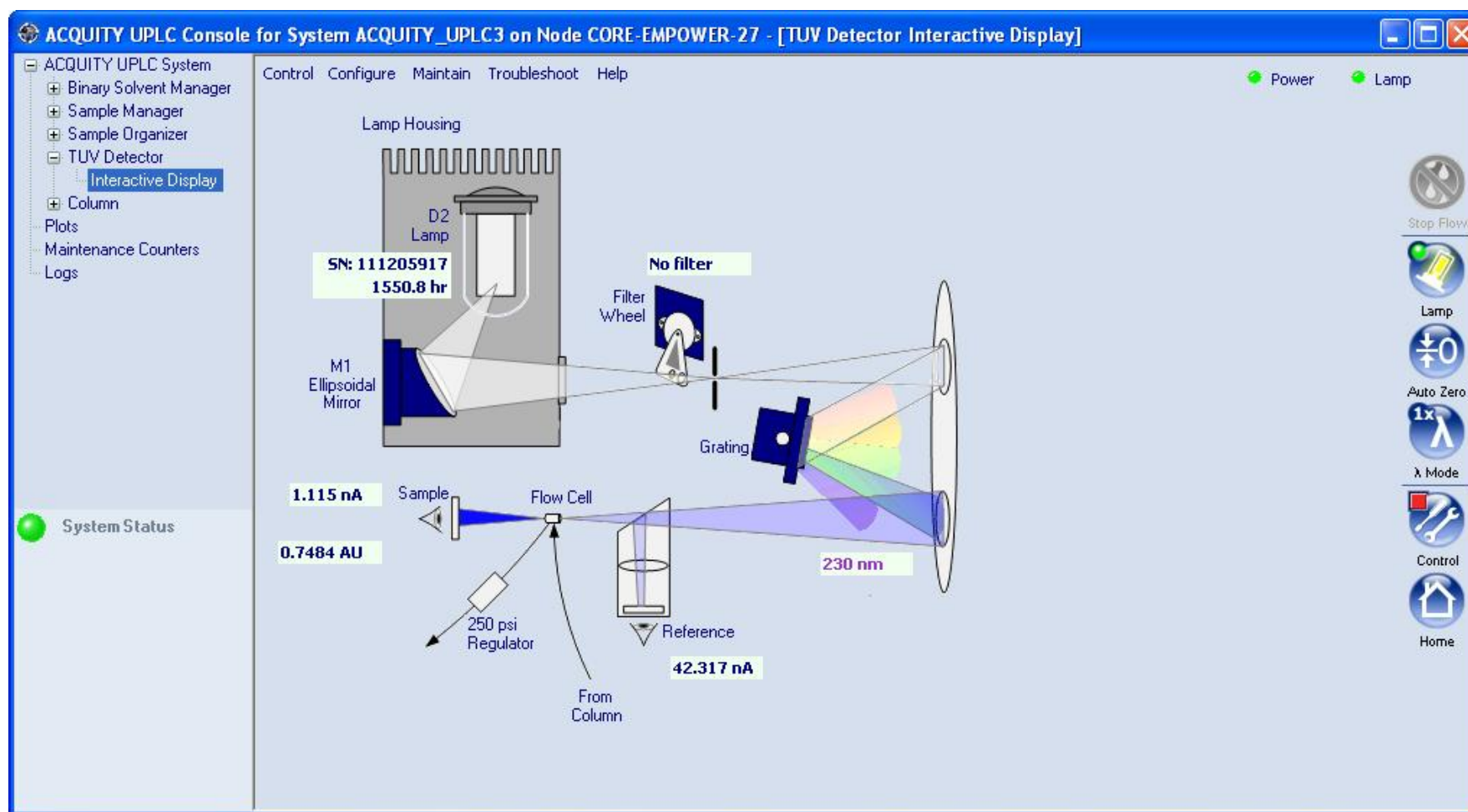
状态:TUV 控制界面

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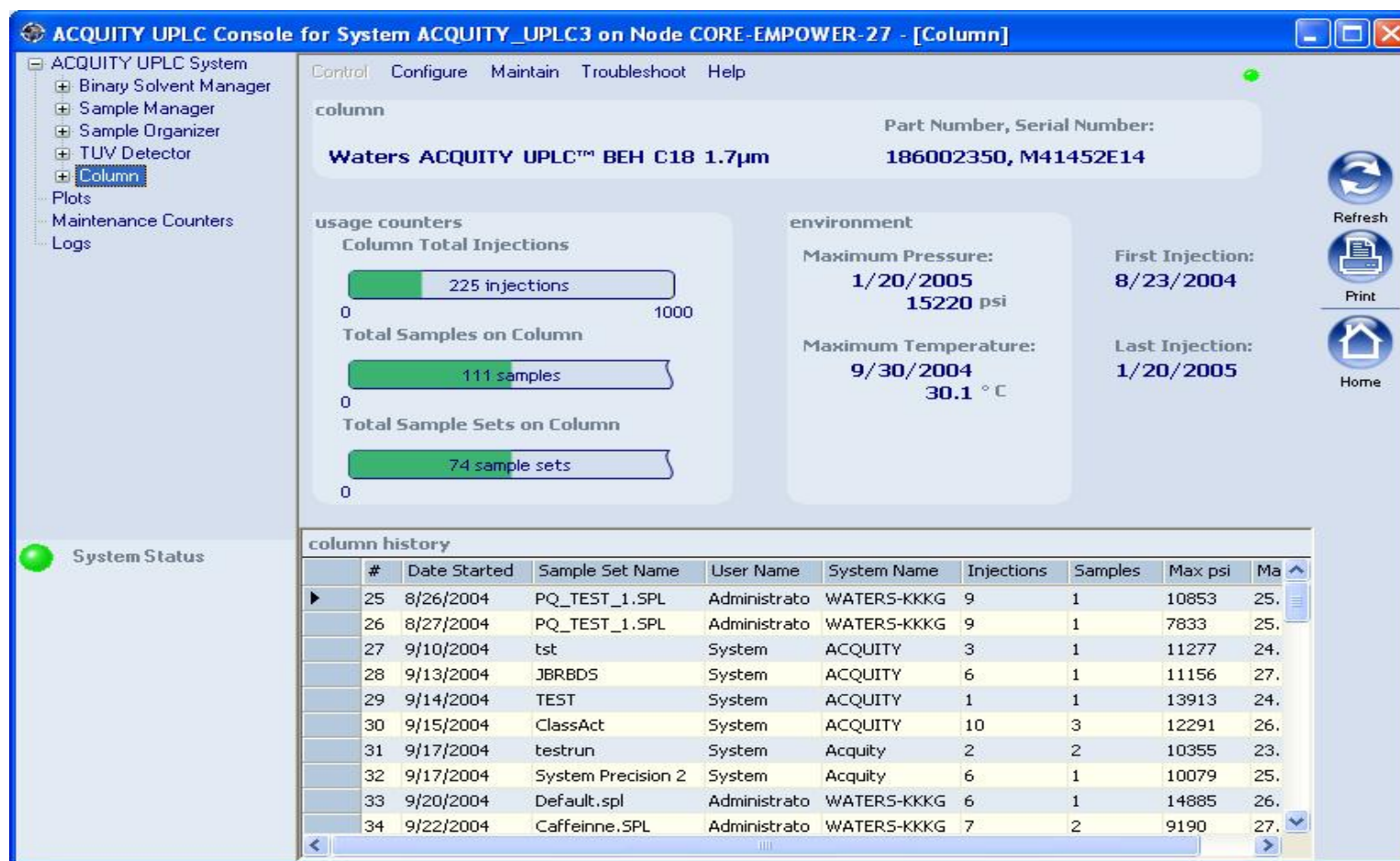
状态:TUV 控制界面

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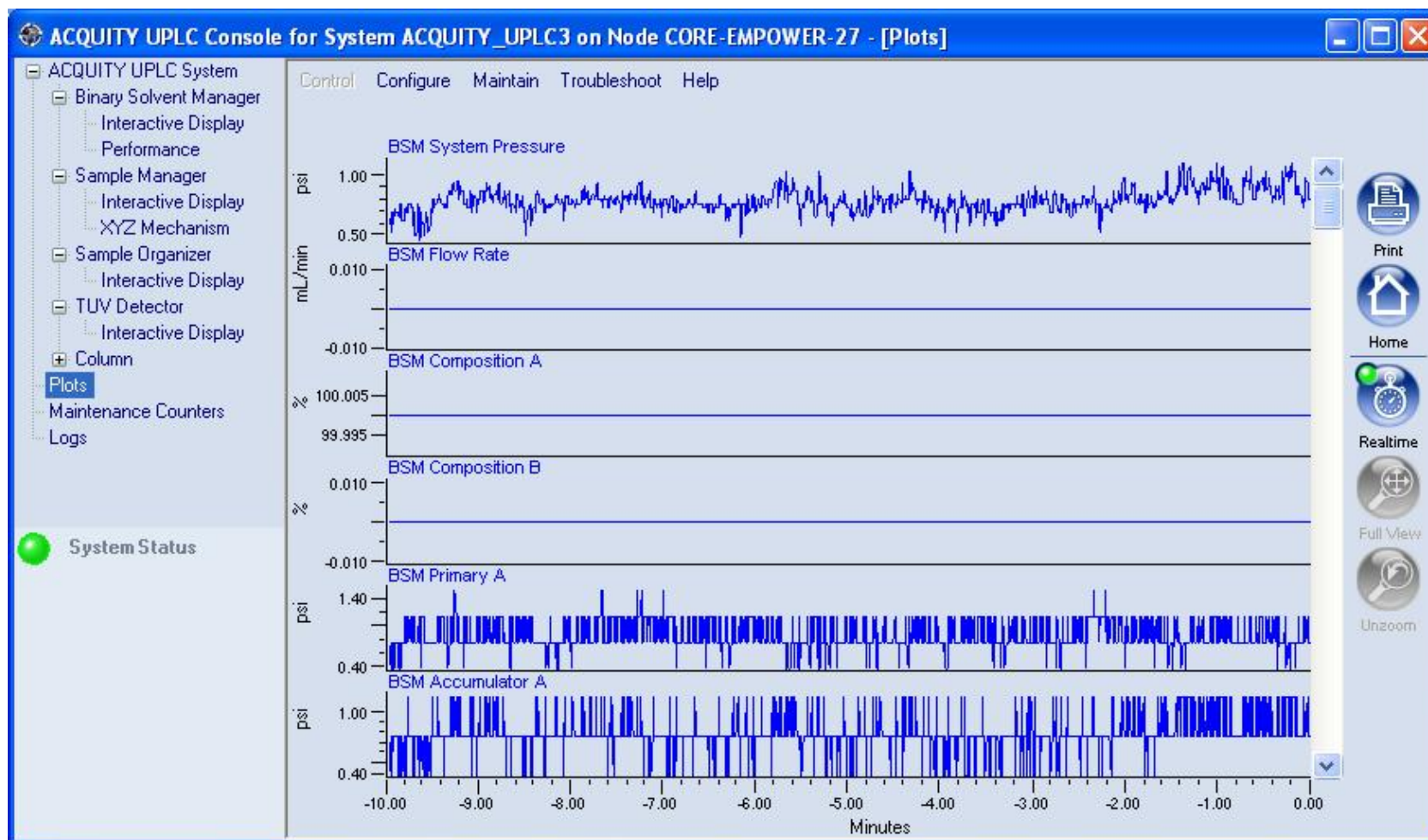
色谱柱信息界面

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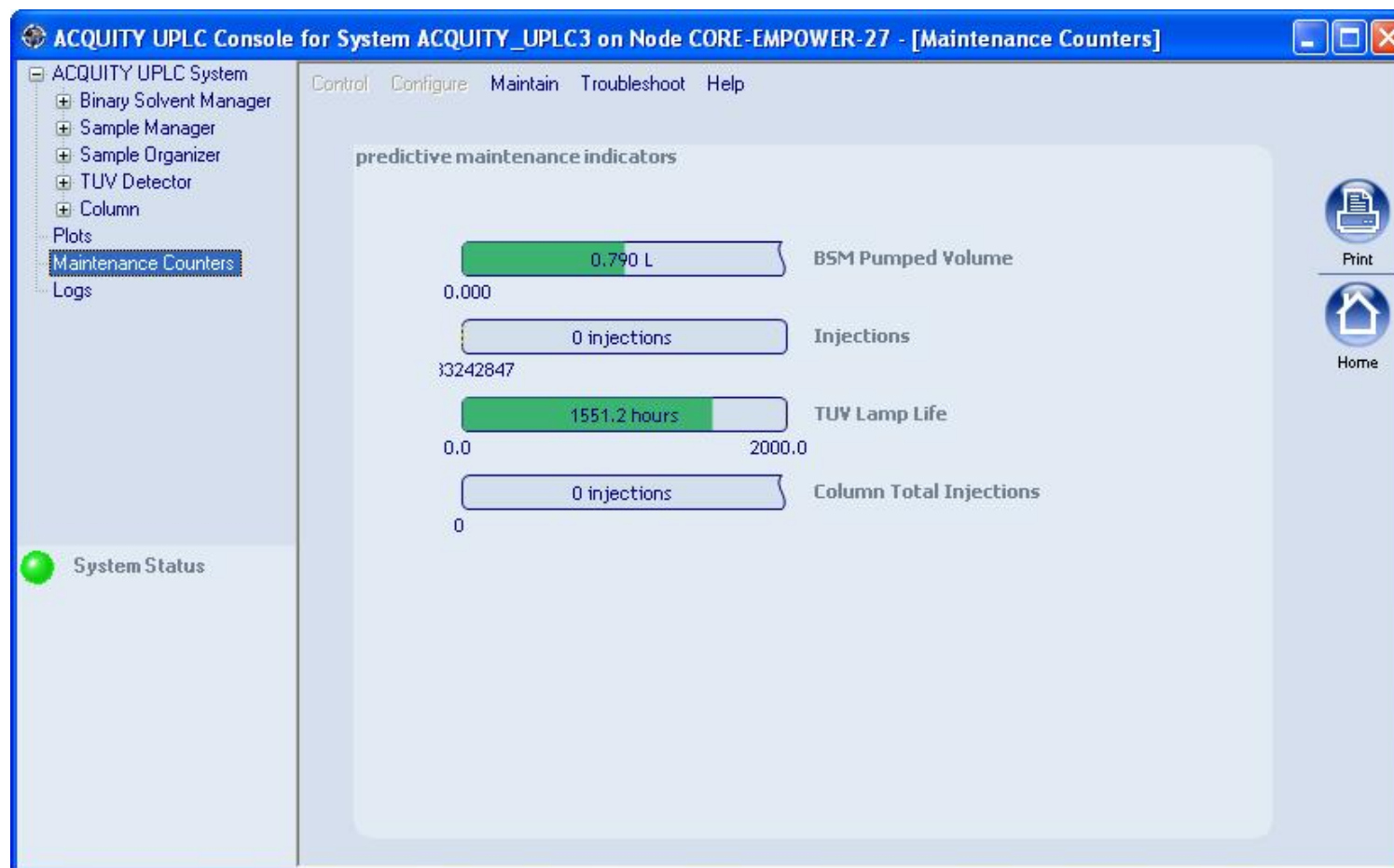
诊断:交互式显示图

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系统信息:维护计数

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系统信息:顶层日志

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ACQUITY UPLC Console for System ACQUITY_UPLC3 on Node CORE-EMPOWER-27 - [Logs]

Control Configure Maintain Troubleshoot Help

Dates: All Content: Errors System or Module: Current System

records

	Date and Time	Severity	Instrument	Message
▶	10/27/2005 10:41 AM	Information	ACQ-SM#A05UP55	eCord connected: SN(M41452E14) Inj. Ct.(225)
	10/27/2005 10:39 AM	Information	ACQ-SM#A05UP55	eCord disconnected: SN(M41452E06) Inj. Ct.(224)
	10/27/2005 10:37 AM	Information	ACQ-SM#A05UP55	eCord connected: SN(M41452E06) Inj. Ct.(224)
	10/27/2005 10:36 AM	Information	ACQ-SM#A05UP55	eCord disconnected: SN(M41452E14) Inj. Ct.(225)
	10/27/2005 10:16 AM	Information	ACQ-SM#A05UP55	eCord connected: SN(M41452E14) Inj. Ct.(225)
	10/26/2005 2:29 PM	Information	ACQ-SM#A05UP55	eCord disconnected: SN(01211517210L92) Inj. Ct.(0)
	10/26/2005 2:01 PM	Information	ACQ-SM#A05UP55	eCord connected: SN(01211517210L92) Inj. Ct.(0)
	10/26/2005 2:00 PM	Warning	ACQ-SM#A05UP55	eCord data invalid
	10/26/2005 9:32 AM	Error	ACQ-SM#A05UP55	Needle volume not calibrated
	10/25/2005 4:36 PM	Information	ACQ-SM#A05UP55	eCord disconnected: SN(01211517210L92) Inj. Ct.(0)
	10/25/2005 9:15 AM	Information	ACQ-SM#A05UP55	eCord connected: SN(01211517210L92) Inj. Ct.(0)

details of current record

eCord connected: SN(M41452E14) Inj. Ct.(225)

ACQUITY UPLC System

- Binary Solvent Manage
 - Interactive Display
 - Performance
- Sample Manager
 - Interactive Display
 - XYZ Mechanism
- Sample Organizer
 - Interactive Display
- TUV Detector
 - Interactive Display
- Column
 - Column QC
 - Batch QC
 - Care and Use
- Plots
- Maintenance Counters
- Logs
- System Status

Refresh

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故障诊断与排除

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