Agilent GCMSD 操作规程

1. 打开氦气钢瓶减压阀。保证钢瓶出口压力在合适的范围（0.4-0.6mpa）。检查是否需要更换 GC 进样垫（绿色， 红色耐高温或低流失进样垫），衬管，色谱柱。保证 MS 放空阀拧紧，机械泵油位及颜色正常。
2. 接通 GC 和 MS 电源，如果长时间没有开机。需要手推 MS 侧板。等仪器自检通过，进入仪器控制界面。如果通讯有故障。请检查网络是否正常。如果通讯正常，可以将 GC 的电源关闭，重新启动 GC。MS 需要点击 reset 键（具体位置见章末）。自检通过后再次启动软件。
3. 系统初始化：（主要目的：抽真空）
   a) 设定离子源及四级杆温度
   b) 柱流量不要高于 2ml/min (标准型高真空泵)
   c) 推荐开机两小时后再开始调谐，测定进样。如果条件允许，长期开机可以保持良好的真空。节省开机等待时间，降低背景例如水峰等干扰。进一步提高灵敏度
4. 在仪器控制界面，编辑方法，运行方法。方法的默认路径为 C:\MSDchem\1\method\. 根据实验室的具体情况。路径可以在 D 盘等。数据在 Data 文件夹下。如果有自动进样器，可以编辑序列批量进样。序列路径在 Sequence 下。

编辑一个完整的方法（Edit Entire Method）

- 方法信息（Method Information）：
  - ✧ 描述性文字
- 采集参数（Instrument/ Acquisition）：
  - ✧ 进样口：进样模式（分流，不分流等），温度，压力，载气等
  - ✧ 色谱柱：恒流，恒压，流量，柱信息等
  - ✧ 炉温：恒温，程序升温等
  - ✧ 自动进样塔：洗针程序
  - ✧ MS：调谐文件，溶剂延迟，质量范围，sampling, 阈值或 SIM 分组时间，停留时间等
- 数据分析参数（Data Analysis）：
  - ✧ 谱图优化：鼠标右键，左键及左右键的功能
  - ✧ 积分优化：自动积分，设参数做积分（化学工作站积分&RTE 积分），手动积分
  - ✧ 建立校正表：第一个浓度标样，New Calibration Table，输入名称、含量、目标离子、限定离子

    第二个浓度标样：Add new Level，输入含量

第三个浓度标样：Add new Level，输入含量

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第一个浓度标样第 2 针数据：Recalibrate，选择级别 1，平均或取代

第二个浓度标样第 2 针数据：Recalibrate，选择级别 2，平均或取代

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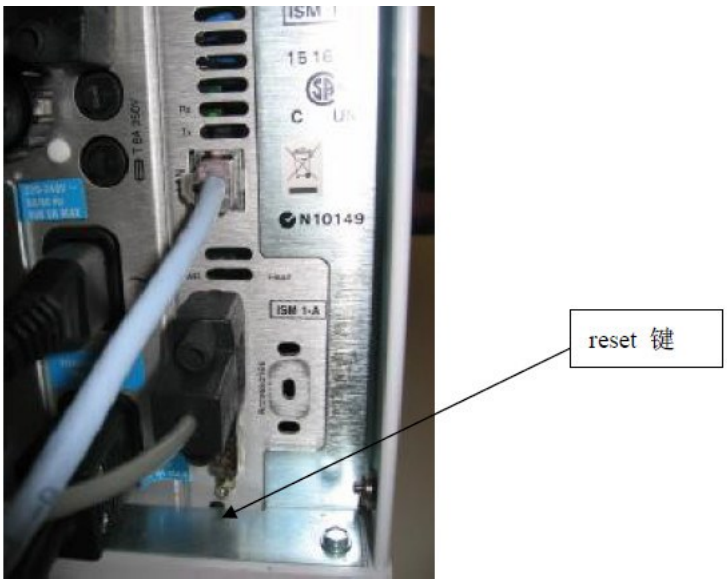
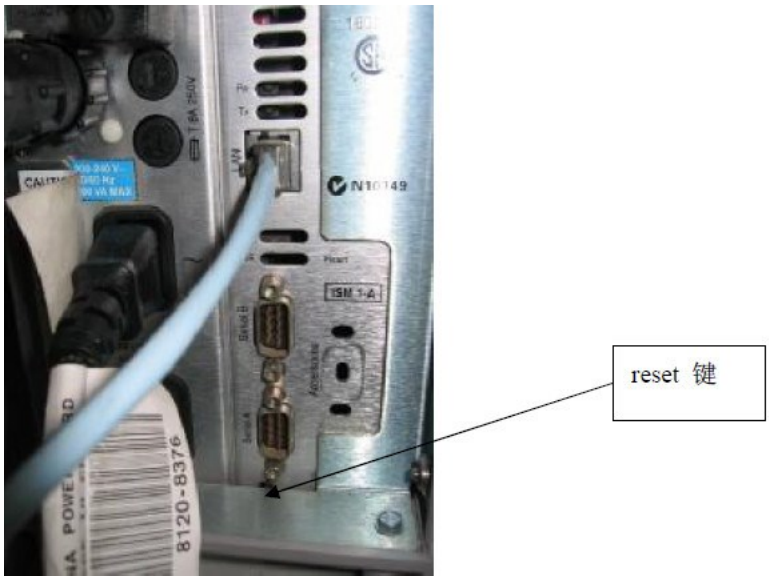
校正设置：浓度单位、曲线/原点

✧ 设定报告：百分比报告，谱库检索报告，定量报告，用户自建报告、输出目的地（屏幕，打印机或保存为文件）

5975MSD reset 键



5973MSD reset 键



Agilent GCMSD SOP

1. Open the helium cylinder pressure reducing valve. Ensure that the cylinder outlet pressure is in the appropriate range (0.4-0.6mpa). Check if the GC pads (green, red, high temperature or low bleed pads), liners, columns need to be replaced. Make sure that the MS vent valve is tightened and the oil level and color of the mechanical pump are normal.
2. Power up the GC and MS, if not powered on for an extended period of time. The MS side panel needs to be pushed by hand. After the instrument passes the self-test, enter the instrument control interface. If there is a communication failure. Please check if the network is normal. If the communication is normal, you can power off the GC and restart the GC. The MS needs to click the reset button (see the end of the chapter for the exact location). Start the software again after passing the self-test.
3. System initialization: (main purpose: vacuumize)
   a) Set the ion source and quadrupole temperature
   b) The column flow should not be higher than 2ml/min (standard high vacuum pump)
   c) It is recommended to start tuning and measure the injection after two hours of power-on. If conditions permit, a good vacuum can be maintained after long-term startup. Save boot time and reduce background interference such as water peaks. Further increase in sensitivity
4. In the instrument control interface, edit the method and run the method. The default path for the method is C:\MSDchem\1\method\. It depends on the specific situation of the laboratory. The path can be in the D drive and so on. The data is under the Data folder. If you have an autosampler, you can edit sequence batch injections. Sequence paths are under Sequence.

Edit Entire Method

- Method Information:
  - ✧ Descriptive text
- Instrument/ Acquisition:
  - ✧ Injection port: injection mode (split, splitless, etc.), temperature, pressure, carrier gas, etc.
  - ✧ Column: constant flow, constant pressure, flow, column information, etc.
  - ✧ Furnace temperature: constant temperature, temperature program, etc.

✧ Autosampler Tower: Needle Wash Program

✧ MS: tune files, solvent delays, mass ranges, sampling, threshold or SIM grouping times, dwell times, etc.

■ Data Analysis:

✧ Spectrum optimization: functions of the right mouse button, left button and left and right buttons

✧ Integration optimization: automatic integration, set parameters for integration (ChemStation integration & RTE integration), manual integration

✧ Create calibration table: the first concentration standard sample, New Calibration Table, enter name, content, target ion, limiting ion

Second concentration standard: Add new Level, enter the amount

The third concentration standard: Add new Level, enter the amount

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1st concentration standard 2nd pin data: Recalibrate, select level 1, average or replace

Second concentration standard 2nd pin data: Recalibrate, select level 2, average or replace

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Calibration Settings: Concentration Units, Curve/Origin

■ Report setting: percentage report, library search report, quantitative report, user-created report, output destination (screen, printer or save as file)