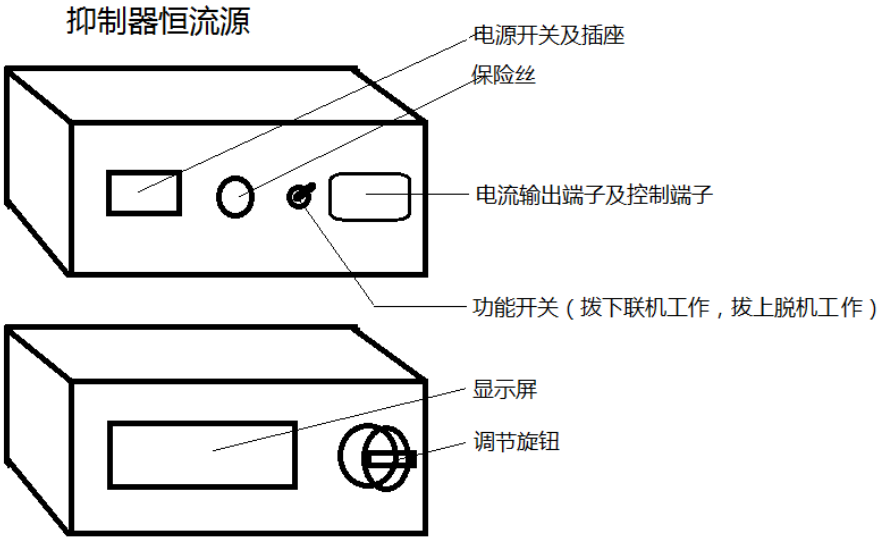
ICS-900 离子色谱仪操作规程

1. 开机

1) 确认淋洗液储量是否满足需要,即测完样后剩余量≥100ml。阴离子淋洗液浓度：4.5mM Na2CO3+0.8mM NaHCO3，建议先配制成浓缩母液(可先配制浓缩 100 倍的母液，即取 4.775g Na2CO3，0.674g NaHCO3 混合，加去离子水定容至 100ml，每次取 20ml 母液稀释至 2L)，然后用母液稀释，以减少误差。阳离子淋洗液浓度：20mM 甲基磺酸（取优级纯 MSA 2.6ml 去离子水稀释至 2L）淋洗液配好后，先摇匀，再真空抽滤后再继续脱气 2 分钟以上。然后沿壁缓缓倒入淋洗液瓶中。（阴离子淋洗液使用水系微孔滤膜过滤；阳离子淋洗液一般无需过滤，若过滤需使用有机系微孔滤膜过滤。）
2) 若仪器有超过一周以上未用，拆下抑制器，活化抑制器。将抑制器上的四接口短接。即将淋洗液入口(ELUENT IN)与淋洗液出口(ELUENT OUT)用黑色直通接头短接、再生液入口(REGEN IN)与再生液出口(REGEN OUT)用灰色直通接头短接。 从抑制器的淋洗液出口(ELUENT OUT)和再生液入口(REGEN IN)分别接上专用的活化接头，用注射器分别注入 5ml 以上的超纯水。
3) 开启氮气瓶总开关，分压表调至 0.2Mpa 左右，淋洗液瓶上的压力表调至 5～10psi(拔出黑色旋钮，顺时针调节至 5psi，将黑色旋钮推回原位锁住)。倒空废液桶中废液，防止其溢出。
4) 打开稳压电源开关，待稳压电源稳定后，再打开 ICS-900 主机电源开关。
5) 启动电脑。待电脑右下角“服务管理器”启动完毕，双击桌面 “Chromeleon 7”，进入工作站，单击工作站左侧导航栏“仪器”进入仪器控制面板。

2. 运行前的准备工作

1) 确认室温是否在 15～30℃之间，若不符，则需开空调，关好门窗，控制室温。
2) 检查软件左侧 “Connected”是否为绿色，若不是，则单击使软件与仪器之间建立起联接。
3) 拉开主机的前盖， 反时针旋松泵头上的废液阀（约拧松 2 圈左右），然后按“灌注”，排除泵头里残留的气泡。约 5 分钟左右，按“关闭”停泵，然后旋紧泵头废液阀。注意不要拧得太紧，防止损坏密封圈。
4) 重新在“泵”模块中按“打开”，待压力上升至 1000psi 时，开启抑制器的外加电源开关，设置电流为 60mA 左右（依分析方法而定，一般设定为 30Ma，具体计算办法附后），将调节钮边上的锁扣向下锁定，将后面的功能开关拨向上方（拨向下方为泵对电源控制）。



5) 在 ICS900 项下单击左上方的“监视基线”后再单击“确认”，进行基线采集，基线波动在 10min 内小于 0.01uS 方可进行做样，（此过程大约 30min）。
6) 准备好需要分析的标液和待测样品溶液。

3. 进样

1) 开泵约 30 分钟左右，基线已平稳，且总电导 Total 和系统压力在正常范围内（阴离子 AS23 系统的总电导值应在 15～30μS 之间，总压力应在 1800±500psi 内）则再次按“监视基线”按钮停止基线的采集。
2) 点击左下侧的“ 数据 ”，在左上侧的文件夹下找到最近做过的序列文件（即样品表），单击选中后，单击最上方的“文件”菜单下的“另存为.”，改名，在保存原始数据前打钩；若仅测样品不做标样（即使用上次的工作曲线），则保留标样数据，否则选中后单击右键菜单下的“删除”键，将不需要的删除。然后在右侧列表中更改或添加样品名称和个数。样品类型：做标样选“校准标准品”；纯水为“空白”；“未知”为待测样。“级别”显示数值为标准曲线的第几个点。然后选定“仪器方法”、“处理方法”，进样量、定容体积或稀释因子通常不需要更改。更改完成后，单击样品表上方的“保存”键重新保存。

A、在数据界面点“创建”项下的“仪器方法”，“选择仪器”不需要改，点下一步；

a) 设置运行时间
b) 编辑泵方法
c) 更改手动进样信息
d) 添加需要注释，完成编辑

B、在数据界面下，双击仪器名称，在仪器方法中修改泵流速；最大、最小压力；进样信息；运行时间等，修改完成后点“保存”，此方法更改后会覆盖原来的方法。（也可以新建文件名，依次编辑 5 个文件，分别为“序列”、“仪器方法”、“处理方法”、“报告模板”及“视图设置”。可通过菜单栏“创建”项下的新建向导来新建。）

3) 序列文件新建好后，再次核对调用的仪器方法是否为该样品的仪器方法。
4) 单击导航栏“仪器”，进入“队列”选项卡，单击“添加…”添加目标序列到当前队列中。
5) 单击“就绪检查”，进行测试前队列的最后检查。若就绪检查报告成功，则单击“开始”，开始进样。
6) 按照提示，先用注射器注入相应的溶液后，再按下方的“确定”键。说明：注射器最后剩余的一小截气泡不要注入，以免引起进样体积误差。依此类推，按照序列表中的顺序在出现提示窗口后依次进样，直至全部结束。注意：标样浓度从低到高，标样之间可不需清洗注射器和进样口；进每一个待测样前均需先清洗注射器和进样口。（准备一杯新制的超纯水，分别用超纯水清洗注射器和定量环 3 次以上）

4. 谱图数据处理

1) 点击导航栏“数据”，找到需要处理的序列文件，将样品表中的标准品“类型”设置为“校准标准品”，将“级别”设置为 01、02、03...等。
2) 双击打开第一个标准品，在导航栏中选择“数据处理”，在 ribbon 栏“数据处理主页”中选中“色谱图”与“处理方法”图标。

   在色谱图下方选择“检测”选项卡，单击“运行 Cobra 向导”，分别设置“积分区域”、“基线噪声范围”、“Cobra 平滑宽度”、“最小峰面积”、“通道和进样类型”。并根据需要在“分组区”增加更多的色谱图处理参数。
3) 在色谱图下方选择“组份表”选项卡，单击“运行组份表向导”，分别设置“时间范围”、“筛选”、“复核”。并根据需要在“分组区”设置“窗口”范围，“标准品方法”，“校准类型”等，在“级别”中输入校准标样的浓度值，在“浓度单位”中设置浓度。
4) 在色谱图下方选择“校准”选项卡，进行“综合校准设置”，并可在“分组区”设置是否启用校准标样等。
5) 设置完成后，保存更改。
6) 在 ribbon 栏“数据处理主页”中选中“色谱图”、“校准图”、“交互结果”即可查看当前峰的校准曲线、校准结果、峰结果等。使用 F4 或 Ctrl+F4 可快捷切换下一个进样或上一个进样。
7) 在导航栏中选择“报告设计器”，即可查看打印报告，或将报告导出为.txt 或.xls 或.pdf 文件。

5. 关机及关机后处理。

1) 分析样品结束后，用淋洗液冲洗流路约二十分钟后，关抑制器外加电流和电源、关泵，关主机电源，最后关压力表、气瓶主阀、气瓶减压阀、稳压电源。
2) 填写实验记录，记录下系统压力、总电导等信息，然后将样品、试剂移出仪器间，清理室内卫生，关好水、电、门窗等。

6. 安全操作注意事项

1) 该仪器必须有专人保管、专人使用。
2) 注意仪器间的洁净，保持无尘。
3) 使用环境：室温，仪器不能直对着空调，无腐蚀性气体。
4) 严格遵守操作规程，出现故障时应作好记录，同时立即向保管人员及科室主任报告，平时使用仪器应作好仪器使用记录及实验记录。
5) 切记：分析柱和保护柱不能用超纯水长时间冲洗或保存，直接使用淋洗液保存即可。
6) 普通的样品，如饮用水、地表水、降水等，进样前需用 0.45μm 以下孔径的微孔水系滤膜过滤，而离子浓度过高或含有机物、过滤金属或重金属浓度较高的样品，如江水、海水、污水或电子五金企业的样品等，进样前需经过其它特殊的前处理方可进样，具体情况可咨询戴安公司的应用工程师。
7) 离子色谱所有管路和接头均为耐酸碱的 PEEK 材料，安装或更换时仅需用手拧紧即可，切忌用扳手拧得过紧，导致管路变形或堵塞。
8) 离子色谱所用的样品瓶、容量瓶等容器的清洗，尽量不要使用洗洁剂和洗液，只需灌满超纯水超声半小时并浸泡 24 小时以上再洗净晾干即可。切忌超声时间过长，导致发热膨胀。

7. 仪器日常维护

1) 仪器日常应保持无尘。
2) 必须保证电源的良好接地。
3) 色谱柱长时间不用，应用淋洗液冲洗约 20 分钟后，从仪器上拆开来并用堵头堵死密封保存，以免其中的液体挥发导致损坏。
4) 抑制器短期不用（一周以上），应用注射器分别从淋洗液出口和再生液入口注入 5 毫升以上的超纯水，然后用堵头堵死密封存放。抑制器再次使用前也应按此方法活化。
5) 定期活化抑制器：分别从抑制器的 Eluent out 和 Regen in 接口处注入 5ml 以上超纯水，以防止抑制器中有沉淀析出。
6) 定期测抑制器的反压，反压不应超过 100psi。反压即为不接电导池和和接电导池的压力差。
7) 定期开机：仪器建议定期使用，若不分析样品，可定期（建议 1～2 周）开机运行 30 分钟后再关机。

8. 系统更换(以阴离子更换为阳离子系统为例)

1) 关闭阴离子系统（关抑制器电流、泵）。
2) 拆下保护柱和分析柱，并用黑色直通连接管路；拆下 ASRS 阴离子抑制器，密封四个接口。
3) 更换装超纯水的淋洗液瓶。

4) 排泵头的气泡。
5) 开泵，清洗管路、电导池至系统电导降至超纯水电导，停泵。
6) 更换阳离子淋洗液,然后开氮气并排泵头的气泡。
7) 开泵，直至总电导率超过 50μS，关泵。
8) 安装保护柱，出口接废液，然后开泵，冲洗保护柱约 5 分钟左右后，关泵。
9) 安装分析柱，出口接废液，然后开泵，冲洗分析柱约 10 分钟左右后，关泵。
10) 活化阳离子抑制器。然后安装抑制器，淋洗液出口（ELUENT OUT）接废液，然后开泵，冲洗抑制器约 2 分钟左右，不需要开抑制器电流，关泵。
11) 安装电导池和抑制器再生液管路，然后开泵，冲洗电导池约 2 分钟左右，不需要开抑制器电流，关泵。
12) 确认管路无漏液，固定好保护柱和分析柱，连接好抑制器的外加电源。

ICS-900 Ion Chromatograph SOP

1. Power on

1) Confirm whether the eluent reserve meets the needs, that is, the remaining amount after the sample is measured ≥ 100ml. Concentration of anion eluent: 4.5mM Na2CO3+0.8mM NaHCO3, it is recommended to prepare a concentrated mother solution (you can prepare a 100-fold concentrated mother solution first, that is, mix 4.775g Na2CO3 and 0.674g NaHCO3, add deionized water to make up to 100ml, Dilute 20ml of mother liquor to 2L each time, and then dilute with mother liquor to reduce errors. Concentration of cationic eluent: 20mM methanesulfonic acid (dilute to 2L with 2.6ml of premium pure MSA deionized water) after the eluent is prepared, shake well, then vacuum filter and then continue to degas for more than 2 minutes. Then pour slowly along the wall into the eluent bottle. (The anion eluent is filtered with an aqueous microporous membrane; the cation eluent generally does not need to be filtered, and an organic microporous membrane is required for filtration.)
2) If the instrument has not been used for more than one week, remove the suppressor and activate the suppressor. Short the four ports on the suppressor. That is, the eluent inlet (ELUENT IN) and the eluent outlet (ELUENT OUT) are short-circuited with a black straight connector, and the regeneration liquid inlet (REGEN IN) and the regeneration liquid outlet (REGEN OUT) are short-circuited with a gray straight connector. From the eluent outlet (ELUENT OUT) and the regeneration liquid inlet (REGEN IN) of the suppressor, connect special activation joints, respectively, and inject more than 5ml of ultrapure water with a syringe.
3) Turn on the main switch of the nitrogen bottle, adjust the partial pressure gauge to about 0.2Mpa, and adjust the pressure gauge on the eluent bottle to 5-10psi (pull out the black knob, adjust it to 5psi clockwise, push the black knob back to the original position to lock live). Empty the waste container to prevent it from overflowing.
4) Turn on the regulated power supply switch, and after the regulated power supply is stable, turn on the power switch of the ICS-900 host.
5) Start the computer. After the "Service Manager" in the lower right corner of the computer is started, double-click "Chromeleon 7" on the desktop to enter the workstation, and click "Instrument" on the left navigation bar of the workstation to enter the instrument control panel.

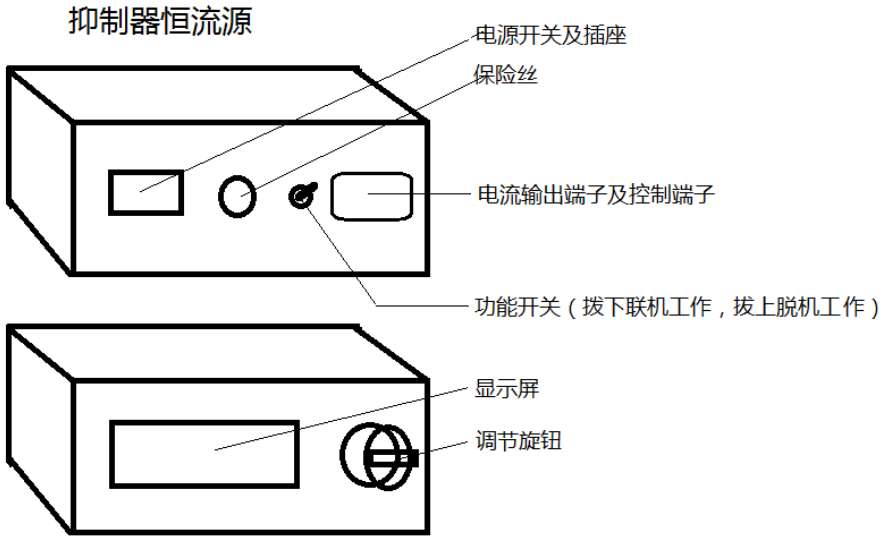
2. Preparations before running

1) Check whether the room temperature is between 15 and 30°C, if not, turn on the air conditioner, close the doors and windows, and control the room temperature.
2) Check whether the “Connected” on the left side of the software is green, if not,

click to establish a connection between the software and the instrument.

3) Open the front cover of the main unit, unscrew the waste valve on the pump head counterclockwise (about 2 turns of loosening), and then press "Fill" to remove the remaining air bubbles in the pump head. After about 5 minutes, press "Close" to stop the pump, and then tighten the waste valve of the pump head. Be careful not to overtighten to prevent damage to the seal.

4) Press "Open" in the "Pump" module again, when the pressure rises to 1000psi, turn on the external power switch of the suppressor, and set the current to about 60mA (depending on the analysis method, generally set to 30Ma, the specific calculation method Attached), lock the lock on the side of the adjustment button downwards, and turn the function switch on the back upwards (turning downward is the control of the pump to the power supply).



5) Under the ICS900 item, click "Monitor Baseline" at the upper left, and then click "Confirm" to collect the baseline. Samples can be made only when the baseline fluctuation is less than 0.01uS within 10 minutes (this process takes about 30 minutes).

6) Prepare the standard solution and sample solution to be analyzed.

3. Injection

a) Turn on the pump for about 30 minutes, the baseline has stabilized, and the total conductance and system pressure are within the normal range (the total conductance value of the anion AS23 system should be between 15 and 30μS, and the total pressure should be within 1800±500psi). Press the "Monitor Baseline" button again to stop the acquisition of the baseline.

b) Click "Data" on the lower left, find the most recent sequence file (ie sample sheet) under the folder on the upper left, click to select it, and then click "Save As." under the "File" menu at the top. ", change the name, and tick before

saving the original data; if only the measured sample is not used as the standard sample (that is, the last working curve is used), the standard sample data will be retained; otherwise, click the “Delete” button under the right-click menu to select Unwanted deletions. Then change or add the sample name and number in the list on the right. Sample type: select "calibration standard" as the standard sample; pure water is "blank"; "unknown" is the sample to be tested. "Level" shows the number of points in the standard curve. Then select "Instrument Method", "Processing Method", injection volume, constant volume or dilution factor usually do not need to be changed. After making changes, click the "Save" button above the sample table to re-save.

A. In the data interface, click "Instrument Method" under "Create", "Select Instrument" does not need to be changed, click Next;

a) Set the run time
b) Edit pump method
c) Change manual injection information
d) Add required comments and complete editing

B. In the data interface, double-click the instrument name, and modify the pump flow rate; maximum and minimum pressure; injection information; running time, etc. in the instrument method. After the modification is completed, click "Save".

(You can also create a new file name and edit five files in sequence, namely "Sequence", "Instrument Method", "Processing Method", "Report Template" and "View Settings". You can create a new file through the "Create" item in the menu bar wizard to create a new one.)

3) After the sequence file is newly created, check again whether the called instrument method is the instrument method of the sample.
4) Click "Instrument" in the navigation bar, enter the "Queue" tab, click "Add..." to add the target sequence to the current queue.
5) Click "Ready Check" for a final check of the pre-test queue. If the readiness check reports a success, click Start to start the injection.
6) According to the prompts, first inject the corresponding solution with a syringe, and then press the "OK" button below. Note: Do not inject a small piece of air bubbles left at the end of the syringe, so as not to cause the injection volume error. And so on, follow the sequence in the sequence table to inject samples in sequence after the prompt window appears, until all the samples are finished. Note: The concentration of standard samples is from low to high, and it is not necessary to clean the syringe and the injection port between the standard samples; the syringe and the injection port need to be cleaned before each sample to be tested is injected. (Prepare a cup of freshly prepared ultrapure water, and wash the syringe and loop with ultrapure water for more than 3 times respectively)

4. Spectral data processing

1) Click "Data" in the navigation bar, find the sequence file to be processed, set the "Type" of the standard in the sample table to "Calibration Standard", and set the "Level" to 01, 02, 03...etc.

2） Double-click to open the first standard, select "Data Processing" in the navigation bar, and select the "Chromatogram" and "Processing Method" icons in the "Data Processing Home Page" of the ribbon column.

Select the "Detection" tab under the chromatogram, click "Run Cobra Wizard", and set the "Integration Area", "Baseline Noise Range", "Cobra Smoothing Width", "Minimum Peak Area", "Channel and Injection Type" respectively ". And add more chromatogram processing parameters in the "grouping area" as needed.

3) Select the "Component Table" tab below the chromatogram, click "Run Component Table Wizard", and set the "Time Range", "Filter", and "Review" respectively. And set the "window" range, "standard method", "calibration type" etc. in the "grouping area" as needed, enter the concentration value of the calibration standard in "level", and set the concentration in "concentration unit".

4) Select the "Calibration" tab below the chromatogram, perform "Comprehensive Calibration Settings", and set whether to enable calibration standard samples in the "Grouping Area".

5) After the setting is complete, save the changes.

6) Select "Chromatogram", "Calibration Chart" and "Interactive Results" in the "Data Processing Home Page" of the ribbon column to view the calibration curve, calibration result, peak result, etc. of the current peak. Use F4 or Ctrl+F4 to quickly switch between the next injection or the previous injection.

7) Select "Report Designer" in the navigation bar to view the printed report, or export the report as a .txt or .xls or .pdf file.

5. Shutdown and post-shutdown processing.

1) After analyzing the sample, flush the flow path with eluent for about 20 minutes, turn off the applied current and power of the suppressor, turn off the pump, turn off the power of the main engine, and finally turn off the pressure gauge, the main valve of the gas cylinder, and the pressure reducing valve of the gas cylinder ,Power Supply.

2) Fill in the experimental records, record the system pressure, total conductance and other information, then remove the samples and reagents from the instrument room, clean the room, and close the water, electricity, doors and windows, etc.

6. Precautions for safe operation

1) The instrument must be kept and used by a special person.
2) Pay attention to the cleanliness between the instruments and keep them free of dust.
3) Use environment: room temperature, the instrument cannot be directly facing the air conditioner, and there is no corrosive gas.
4) Strictly abide by the operating procedures, make records in case of failures, and immediately report to the custodian and the department director, and keep records of the use of the instruments and experiments when using the instruments in peacetime.
5) Remember: the analytical column and the guard column cannot be washed or stored with ultrapure water for a long time, and can be stored directly with the eluent.
6) Ordinary samples, such as drinking water, surface water, precipitation, etc., need to be filtered with a microporous water filter membrane with a pore size of 0.45 μm or less before injection, and the ion concentration is too high or contains organic matter, filter metal or heavy metal concentration. Samples, such as river water, sea water, sewage or samples from electronic hardware companies, etc., need to undergo other special pretreatment before sample injection. For specific conditions, please consult the application engineers of Dionex.
7) All the pipes and joints of the ion chromatography are made of acid and alkali resistant PEEK materials. When installing or replacing, they only need to be tightened by hand. Do not use a wrench to tighten them too much, which will cause the pipes to be deformed or blocked.
8) For the cleaning of containers such as sample bottles and volumetric flasks used in ion chromatography, try not to use detergents and lotions. Just fill with ultrapure water for half an hour and soak for more than 24 hours before washing and drying. Do not use ultrasound for too long, which may cause heat and swelling.

7. Routine maintenance of the instrument

1) The instrument should be kept dust-free on a daily basis.
2) The power supply must be well grounded.
3) If the chromatographic column is not used for a long time, after rinsing with the eluent for about 20 minutes, disassemble it from the instrument and seal it with a plug to prevent the liquid in it from volatilizing and causing damage.
4) If the suppressor is not used for a short period of time (more than a week), inject more than 5 ml of ultrapure water from the eluent outlet and the regeneration liquid inlet with a syringe, and then seal it with a plug for storage. The suppressor should also be activated in this way before being used again.
5) Periodically activate the suppressor: inject more than 5ml of ultrapure water from

the Eluent out and Regen in ports of the suppressor respectively to prevent precipitation in the suppressor.

6) Periodically measure the back pressure of the suppressor, the back pressure should not exceed 100psi. The back pressure is the pressure difference between the unconnected conductivity cell and the connected conductivity cell.
7) Periodic startup: The instrument is recommended to be used regularly. If the sample is not analyzed, it can be turned on regularly (1 to 2 weeks is recommended) and run for 30 minutes before shutting down.

8. System replacement (take the replacement of anion into a cation system as an example)

1) Shut down the anion system (shut off suppressor current, pump).
2) Remove the guard column and analytical column, and connect the pipeline with black straight through; remove the ASRS anion suppressor, and seal the four ports.
3) Replace the eluent bottle filled with ultrapure water.
4) Drain the air bubbles from the pump head.
5) Turn on the pump, clean the pipeline and conductivity cell until the conductivity of the system drops to the conductivity of ultrapure water, and stop the pump.
6) Change the cation eluent, then turn on the nitrogen gas and drain the air bubbles of the pump head.
7) Turn on the pump until the total conductivity exceeds 50μS, then turn off the pump.
8) Install the guard column, connect the waste liquid to the outlet, then turn on the pump, rinse the guard column for about 5 minutes, and then turn off the pump.
9) Install the analytical column, connect the waste liquid to the outlet, then turn on the pump, rinse the analytical column for about 10 minutes, and then turn off the pump.
10) Activate the cation suppressor. Then install the suppressor, the eluent outlet (ELUENT OUT) is connected to the waste liquid, then turn on the pump, and flush the suppressor for about 2 minutes. It is not necessary to turn on the suppressor current and turn off the pump.
11) Install the conductivity cell and suppressor regeneration liquid pipeline, then turn on the pump and flush the conductivity cell for about 2 minutes. It is not necessary to turn on the suppressor current and turn off the pump.
12) Confirm that there is no leakage in the pipeline, fix the guard column and analytical column, and connect the external power supply of the suppressor.