Dionex ICS-6000
Ion Chromatography System

Dual EGC Mode Installation

065760 Revision 02 • May 2020

thermoscientific

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Revision History:

Revision 01, April, 2018, Original Publication.

Revision 02, February 2020 – Updated ICS-6000 System Dual EGC Mode Start-Up Procedure, Vacuum Kit Installation, and Dual EGC Mode Re-Start Procedure (Section 1).

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

 **SAFETY**	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
 **WARNING**	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
 **CAUTION**	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument but will not cause injury.***
 **NOTE**	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

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1. Installation

1.1 ICS-6000 System Dual EGC Mode Start-up, 1-mm Column

NOTE

Thermo Scientific recommends sanitizing the entire system (without installing the columns at a system pressure above 3000 psi) with at least 2 hours of 100 mM KOH at 0.1 mL/min for 1 mm columns using the KOH eluent generator cartridge.

NOTE

To ensure stable baseline and low background noise, it is crucial to have sufficient removal of the hydrogen and oxygen gas formed with the EGC generated eluents. For 1-mm system, connect the vent of the Dionex RFIC Dual EGC Degasser to the Vacuum Port located at the back of the Dionex DP module.

NOTE

Only turn on the EGC power when the system pressure reaches above 3000 psi. This step is very important to ensure best system performance.

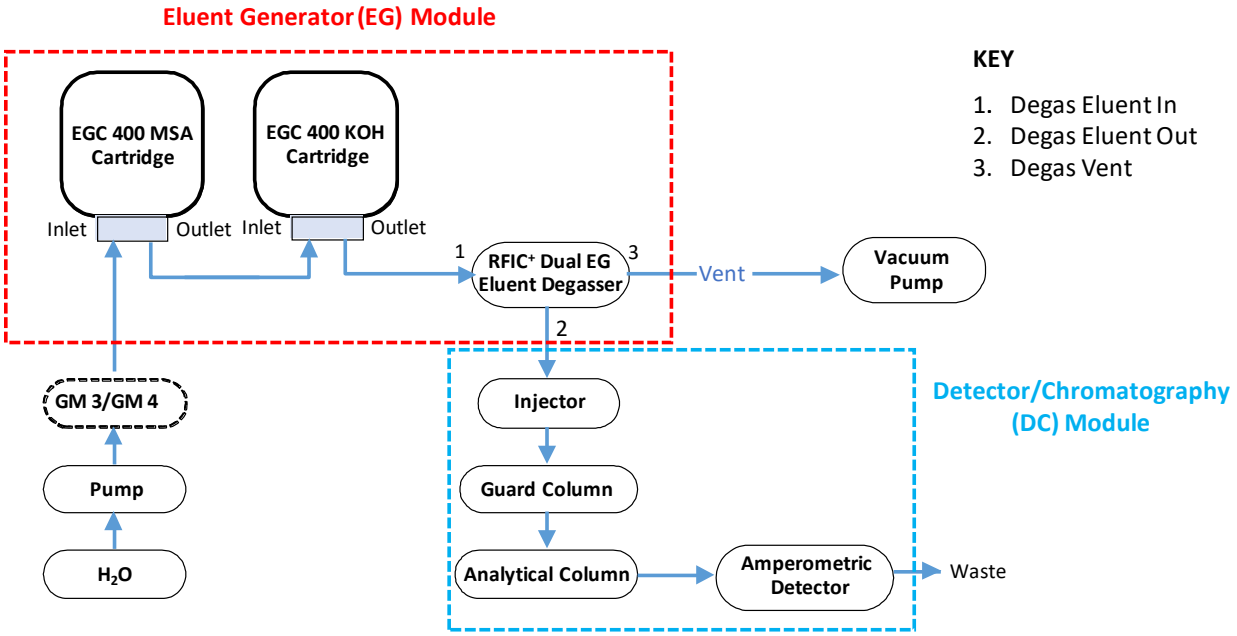
NOTE

To achieve optimum sensitivity, use Au on PTFE disposable electrodes with 1 mil gasket (P/N 30397) for ED detection (carbohydrate quadruple waveform), and use gasket for pH-Ag/AgCl reference electrode (P/N 072162) in the reference chamber of the ED cell.

NOTE

In a properly working system, the electrochemical detection (ED) background for the Dionex CarboPac column QA and most applications is typically 25-45nC.

Figure 1 Plumbing Schematic for ICS-6000 System Dual EGC Mode, 1-mm Column



1.1.1 ICS-6000 System Dual EGC Mode Start-Up Procedure, 1-mm Column

Figure 2 Overview of ICS-6000 System Dual EGC Mode, 1-mm Column

1. Fill a 2 L eluent reservoir bottle with ASTM filtered, Type I (18-megohm) degassed deionized water. Connect the reservoir to the eluent inlet line of the pump. Keep the water blanketed under an inert gas (helium or nitrogen).
2. Prime the pump as instructed by the system operation manual.
3. Remove the vent plug on the top of the Dionex EGC 400 MSA cartridge (please refer to Dionex Eluent Generator Cartridges Manual Document No. 065081 for details about preparation of the Dionex EGC cartridges).
4. Install the Dionex EGC 400 MSA cartridge on the right side (labeled “System 2”) of the panel in the EG module.
5. Connect the pump outlet to the INLET port of the Dionex EGC 400 MSA cartridge and direct the OUTLET port of the cartridge to waste.
6. Turn on the pump and pump DI water through the Dionex EGC 400 MSA cartridge for 15 minutes at the flow rate of 1 mL/min (prime mode).

7. Connect the OUTLET port of the Dionex EGC 400 MSA cartridge to a PEEK backpressure tubing (Item # 22181-20031). Direct the backpressure tubing to waste.
8. Set the pump flow rate to 0.1 mL/min.
9. On the E-Panel, under Eluent Generator Tab, set the Eluent Polarity to Acidic Eluents. Set MSAprog to 100 mM. Turn on the Dionex EGC power.
10. Condition the Dionex EGC 400 MSA cartridge for 30 min.
11. Turn off the pump.
12. Remove the PEEK backpressure tubing from the OUTLET port of the Dionex EGC 400 MSA cartridge.
13. Remove the vent plug on the top of the Dionex EGC 400 KOH cartridge.
14. Install the Dionex EGC 400 KOH cartridge on the left side of the panel (labeled “System 1”) in the EG module.
15. Connect the OUTLET port of the Dionex EGC 400 MSA cartridge to the INLET port of the Dionex EGC 400 KOH cartridge, and direct the OUTLET port to waste.

Figure 3 Dionex EGC 400 MSA Cartridge in EG Module of ICS-6000 System Dual EGC Mode, 1-mm Column

16. Turn on the pump and pump DI water through the Dionex EGC 400 KOH cartridge for 15 minutes at the flow rate of 1 mL/min.
17. Connect the OUTLET port of the Dionex EGC 400 KOH cartridge to a PEEK backpressure tubing (Item # 22181-20031). Direct the backpressure tubing to waste.
18. Set the pump flow rate to 0.1 mL/min.
19. On the E-Panel, under Eluent Generator Tab, set the Eluent Polarity to Basic Eluents. Set KOH_{prog} to 100 mM. Turn on the Dionex EGC power.
20. Condition the Dionex EGC 400 KOH cartridge for 30 min.
21. Remove the backpressure tubing from the OUTLET port of the Dionex EGC 400 KOH cartridge.
22. Connect the OUTLET port of the Dionex EGC 400 KOH cartridge to the ELUENT IN port of the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951). (Refer to Section 1.2.1 for instructions on connecting the vent of the Dionex RFIC$^+$ Dual EG Eluent Degasser to the vacuum system.)

Figure 4 Dionex EGC 400 KOH Cartridge and Dionex RFIC⁺ Dual EG Eluent Degasser in the EG Module of ICS-6000 System Dual EGC Mode, 1-mm Column

23. Direct the ELUENT OUT port of the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951) to waste.
24. With EGC power OFF, turn on the pump at the flow rate of 0.2 mL/min. Flush the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951) for 15 min.
25. Turn off the pump.
26. Remove the PEEK backpressure tubing (Item # 22181-20031) from the ELUENT OUT port of the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951).
27. Connect the ELUENT OUT of the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951) to P port of the injection valve.
28. Install the column to C port of the injection valve. Direct the OUTLET of the column to waste.

Figure 5 View of DC Compartment, with Only the Separator Column Installed (Without the Guard Column).

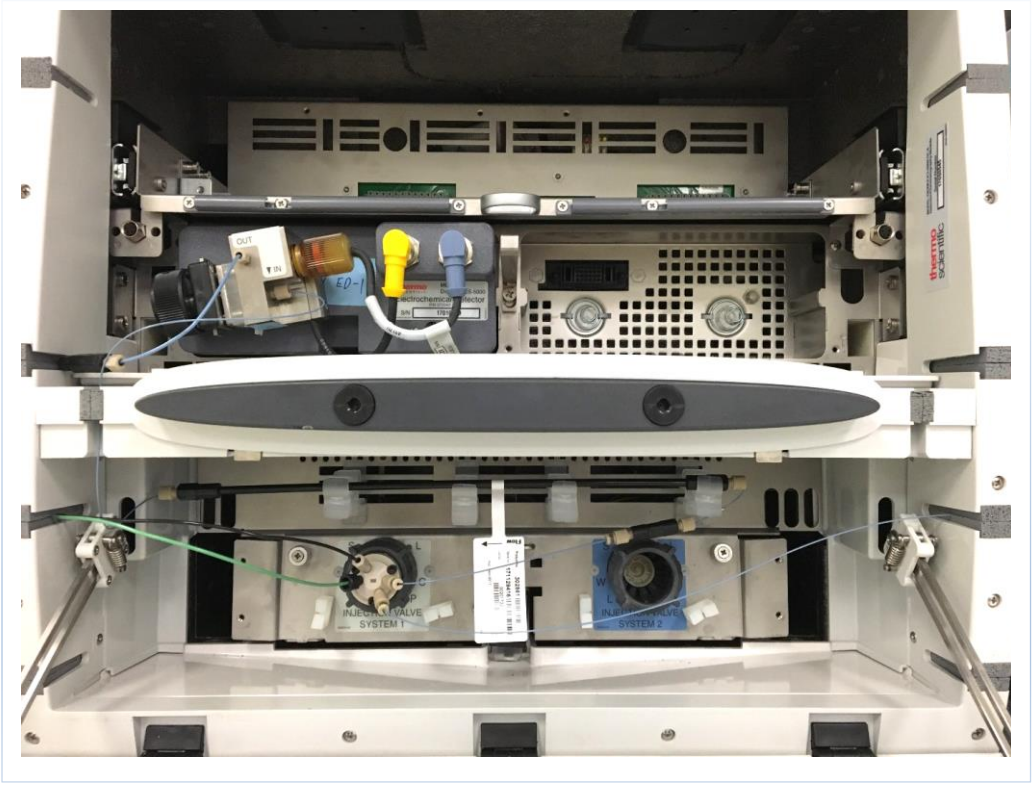
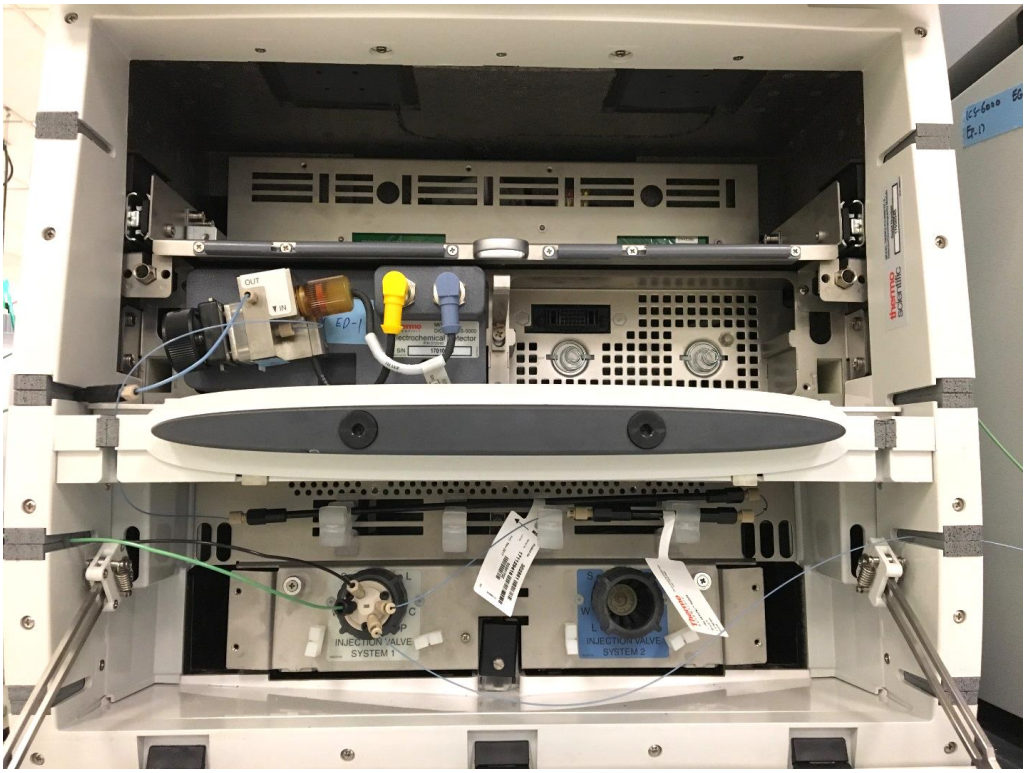


Figure 6 View of DC Compartment, with Both the Guard and the Separator Column Installed.



29. Turn on the pump.
30. After the system pressure reach 3000 psi, with Eluent Polarity set to Basic Eluents, set $KMSA_{prog}$ and KOH_{prog} to the desired concentrations for the applications. Turn on the Dionex EGC power for 60 min.

IMPORTANT

Install backpressure tubing (P/N 22181-20038 for 500 psi back pressure, P/N 22181-20039 for 1000 psi backpressure) between the ELUENT OUT of the Dionex RFIC⁺ Dual EG Eluent Degasser and the C port of the injection valve if necessary, to achieve a system pressure of 3000 psi or above. Please refer to Document No. 155052-01 enclosed with the separation column for flow diagram.

31. Connect the OUTLET of the column to the CELL IN of the ED cell, and direct the CELL OUT to waste.
32. Confirm that the pH is >11. With the pH within this range, turn on the cell using the carbohydrate standard quad waveform. Let the system equilibrate for 30-45 min.

IMPORTANT

To prevent backpressure tubing (P/N 22181-20038 for 500 psi back pressure, P/N 22181-20039 for 1000 psi backpressure, P/N 22181-20031 backpressure tubing) from clogging, for future use, please rinse the tubing with DI water after each use.

NOTE

To achieve optimum sensitivity, use Au on PTFE disposable electrodes with 1 mil gasket (P/N 30397) for ED detection (carbohydrate quadruple waveform), and use gasket for pH-Ag/AgCl reference electrode (P/N 072162) in the reference chamber of the ED cell.

1.1.2 ICS-6000 System Dual EGC Mode Vacuum Kit Installation, 1-mm Column

NOTE

To ensure stable baseline and low background noise, it is crucial to have sufficient removal of the hydrogen and oxygen gas formed with the EGC generated eluents. Connect the vent of the Dionex RFIC⁺ Dual EG Eluent Degasser to the Vacuum Port located at the back of the Dionex DP module.

A. With DP module power off, remove the plug from the vacuum port located at the back of the DP module.
B. Screw a barbed fitting (Item # 047799) onto the vacuum port (Figure 7).
C. Connect one end of the PharMed (Item # 063267) tubing to the barbed fitting (Figure 7).
D. Connect the other end of the PharMed (Item # 063267) tubing to the barbed fitting (P/N 047799) on the cap of the 250 mL glass bottle (liquid trap) (Figure 9).
E. Locate one of the gas tubing labeled “degas vent” located at the back of the EG module (Figure 10). Slide the gas tubing onto the barbed fitting (Item # 056754) installed on the cap of the bottle (Figure 9).
F. Connect the other end of the “degas vent” tubing to the fitting that is located at the vent of Dionex RFIC⁺ Dual EG Eluent Degasser (Item # 22181-60951).
G. Make sure all the connections are properly tightened.
H. Turn on the power of the DP module.
I. Go to Commands (F8) on the Chromeleon Console, select Pump_1, right click on the Properties panel, and check Expert.
J. Under Properties tab, got to Degasser Vacuum, the Value should read OK. This indicates the completion of the vacuum installation.
K. If the Value reads Not OK, check for leaks at all the connections for vacuum. Check the status of the vacuum pump located inside the DP module to ensure that it functions properly.

Figure 7 Vacuum Tubing Connection at the back of the DP Module of ICS-6000 System Dual EGC Mode, 1-mm Column

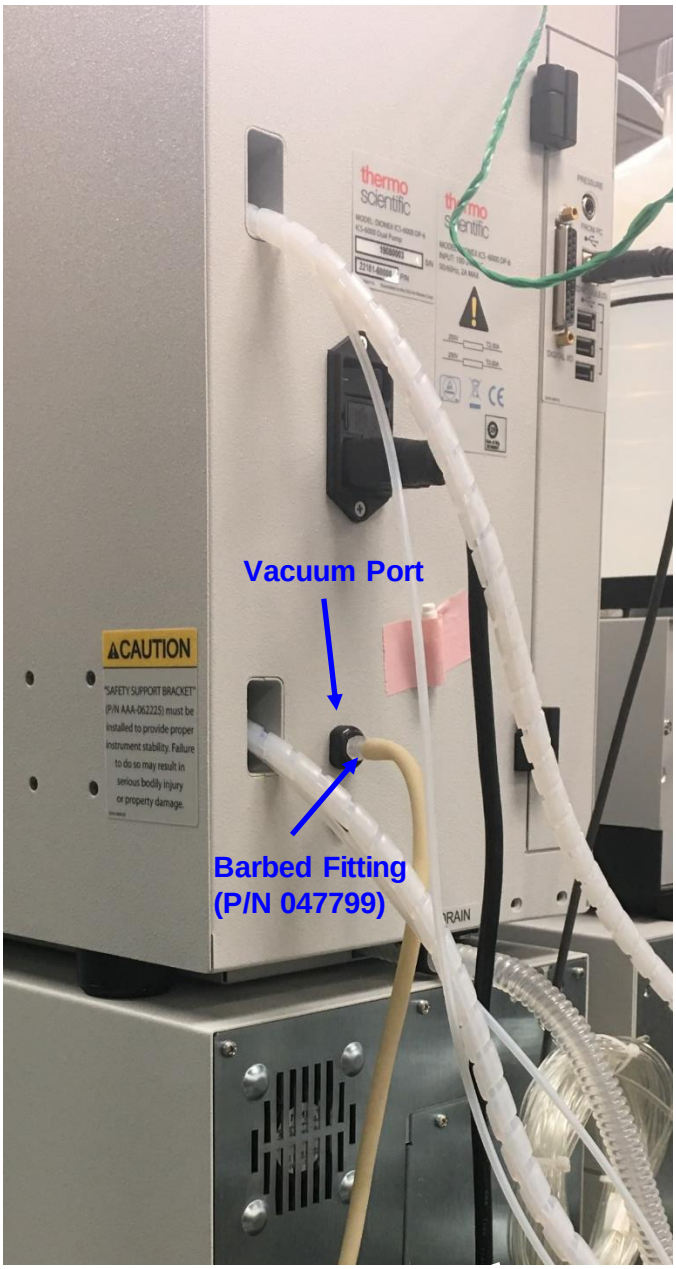


Figure 8 250 mL Bottle Assembly (Fluid Trap)

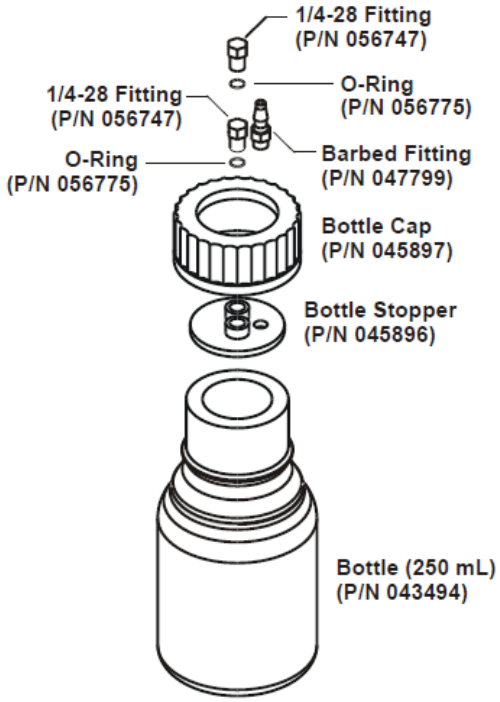
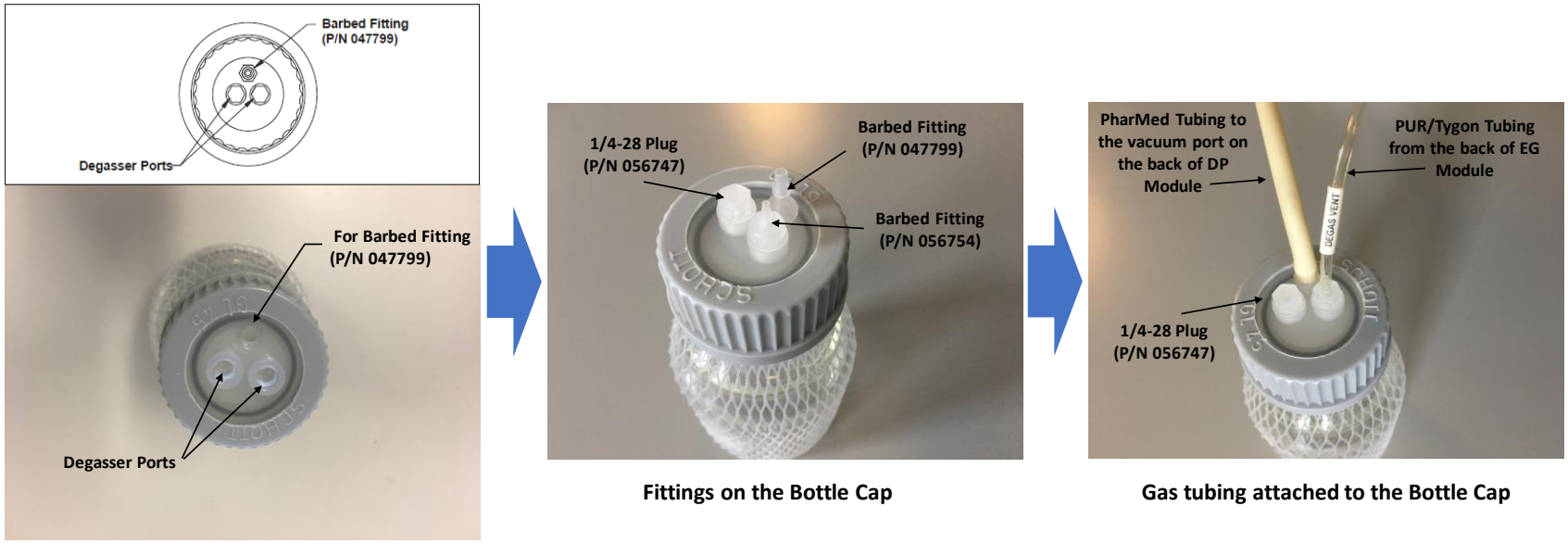


Figure 9 Assembly and Tubing Connections of the Fluid trap for the Vacuum System of ICS-6000 System Dual EGC Mode, 1-mm Column

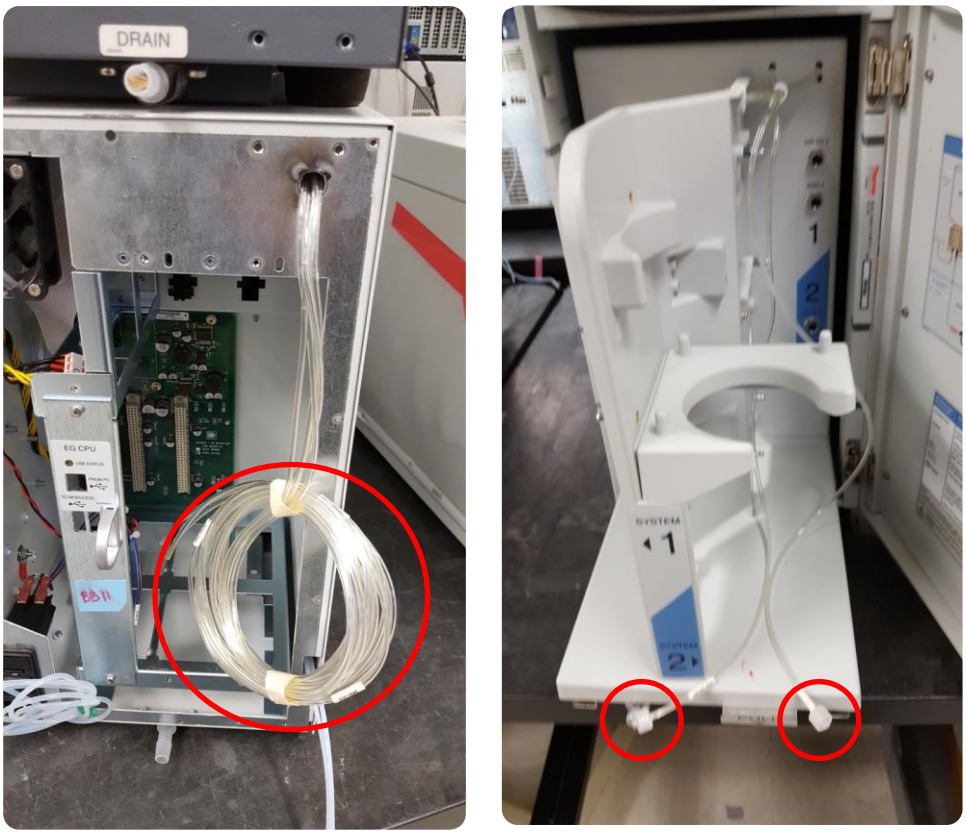


250 mL Bottle Assembly (Fluid Trap)

Fittings on the Bottle Cap

Gas tubing attached to the Bottle Cap

Figure 10 PUR/Tygon Tubing on EG Module of Dual EGC 1-mm System



Rear view of EG module **Front view of EG module**

1.1.3 ICS-6000 System Dual EGC Mode Re-Start Procedure, 1-mm Column

NOTE

The systems under Dual EGC mode are designed to be operated non-stop for months at a time. If the system has been shut down for more than 5 days, the system should be re-started using the following procedure (applied to both analytical and capillary systems).

A. Disconnect all the components after the Dionex EGC 400 KOH cartridge from the system.
B. Direct the OUTLET of the Dionex EGC 400 KOH cartridge to waste.
C. Set the flow rate to 0.2 mL/min. Turn the pump and EGC power on. With Eluent Polarity set to Basic Eluents, set $KMSA_{prog}$ to 5 mM and KOH_{prog} to 5 mM. Allow the system to run for 20 min.
D. Turn the pump off.
E. Connect the OUTLET of the Dionex EGC 400 KOH cartridge to the ELUENT IN of the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951). Direct the ELUENT OUT of the Dionex RFIC$^+$ Dual EG Eluent Degasser to waste.
F. Set the flow rate to 0.2 mL/min and turn the pump on for 15 min. Keep EGC power off.
G. Turn the pump off.
H. Connect the ELUENT OUT of the Dionex RFIC$^+$ Dual EG Eluent Degasser to P port of the injection valve.
I. Disconnect the outlet of the column from the ED cell. Direct the outlet of the column to waste.
J. Set the flow rate to recommended flow rate of the column (e.g. 0.063 mL/min). After the system pressure reaches 3000 psi, turn on EGC power.
K. With Eluent Polarity set to Basic Eluents, set $KMSA_{prog}$ and KOH_{prog} to the desired concentrations for the applications. Turn on the Dionex EGC power for 60 min.
L. Connect the outlet of the separator column to the ED cell.
M. Direct the outlet of the ED cell to waste.
N. Turn the pump and EGC power on. Set to the required application condition.
O. When the pH reading of the ED detector shows pH > 11, turn the ED cell on.
P. Allow the system to equilibrate for 30-45 minutes prior to collecting data.

1.1.4 ICS-6000 System Dual EGC Mode Dual Channel, 1-mm Column

Figures 11 show the schematics of an ICS-6000 System in Dual EGC Mode with Dual Channel configurations. Please follow the start-up and vacuum kit installation procedures described in Sections 1.1.2 to set up each individual channel and Figure 12 for tubing connection on the liquid trap.

Figure 11 Analytical (1-mm) / Analytical (1-mm) Dual Channel

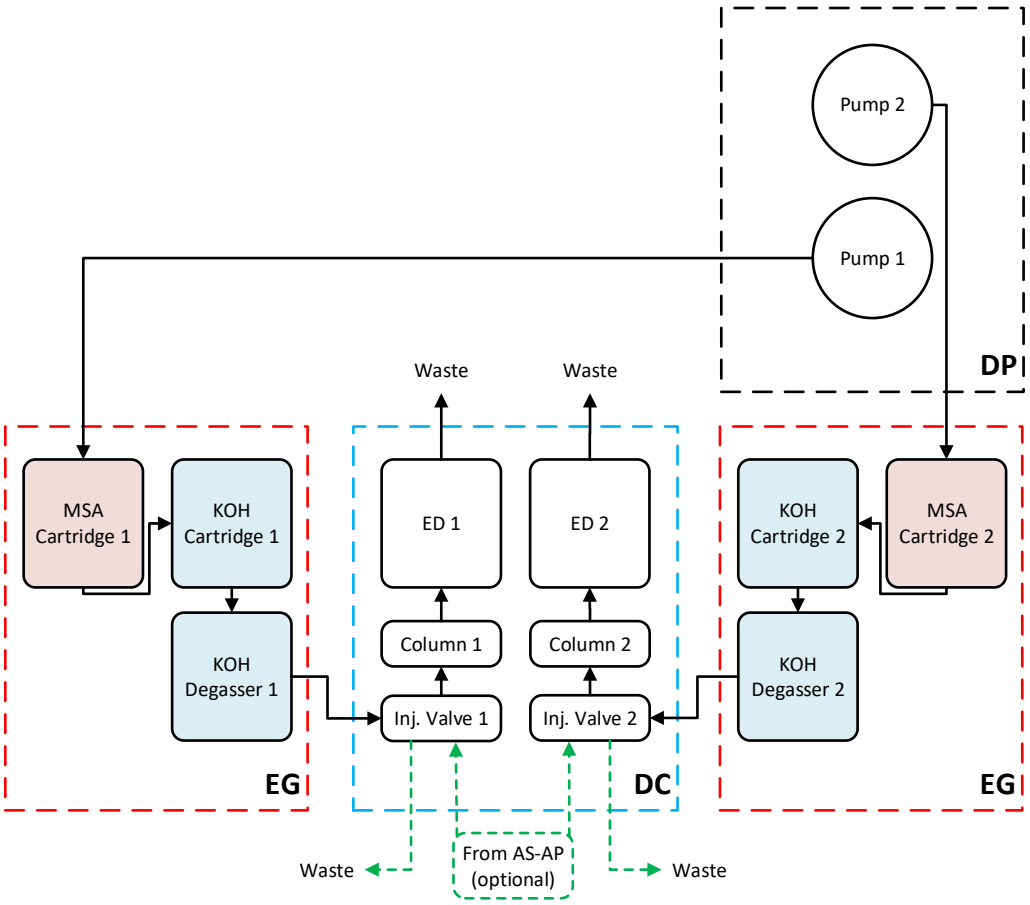
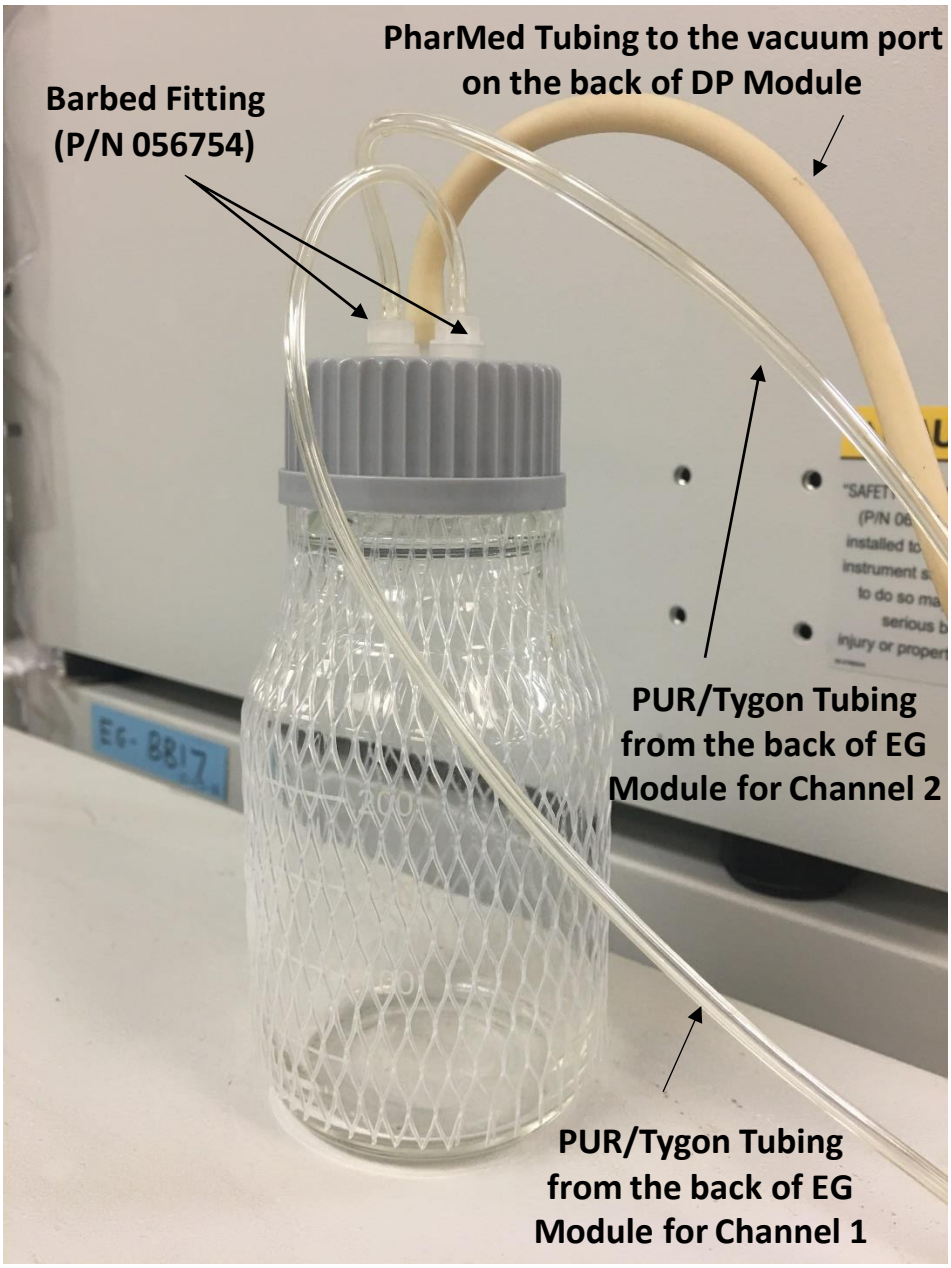


Figure 12 Vacuum Tubing Connection on the Liquid Trap for ICS-6000 System Dual EGC Mode with 1-mm/1-mm Dual Channels



1.2 ICS-6000 System Dual EGC Mode Start-up, 0.4-mm Column

NOTE

Thermo Scientific recommends sanitizing the entire system (without installing the columns at a system pressure above 3000 psi) with at least 2 hours of 100 mM KOH at 0.02 mL/min for 0.4 mm columns using the KOH eluent generator cartridge.

NOTE

To ensure stable baseline and low background noise, it is crucial to have sufficient removal of the hydrogen and oxygen gas formed with the EGC generated eluents. Connect the vents of the Dionex RFIC Capillary EGC MSA Degasser and the Dionex RFIC Capillary Eluent Degasser to the Vacuum Port located at the back of the Dionex DP module. Make sure all connections and fittings for the vacuum degas are vacuum tight before using the system.

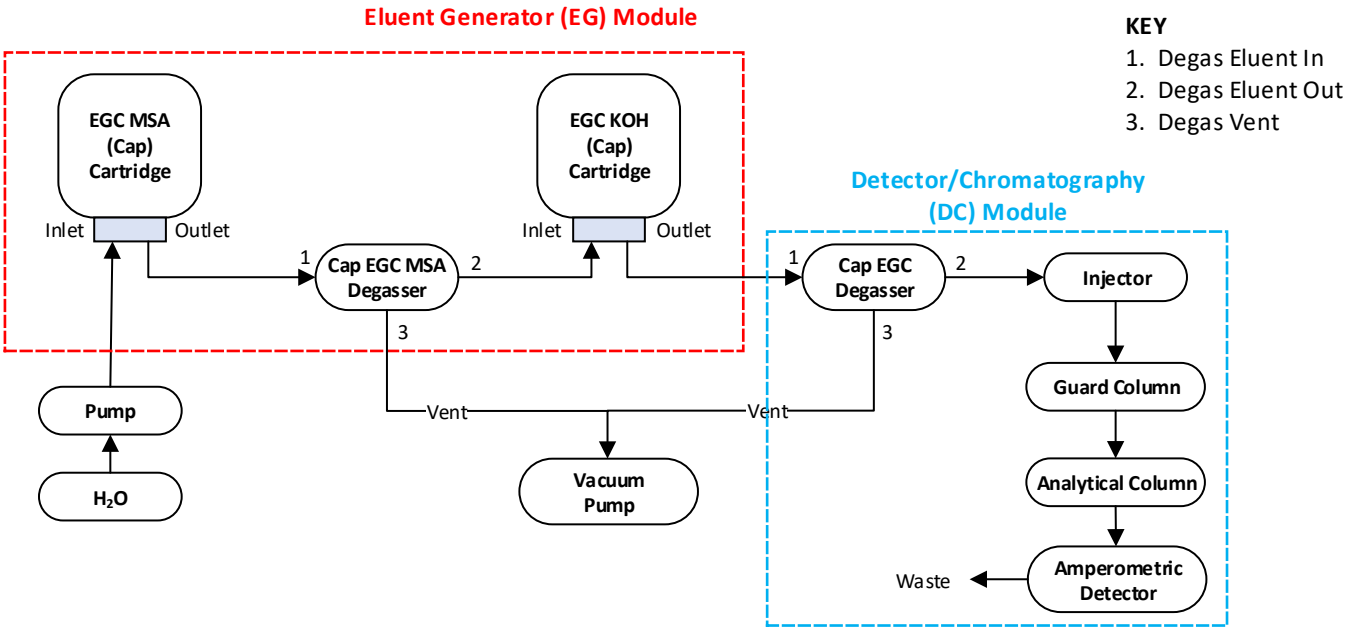
NOTE

Only turn on the EGC power when the system pressure reaches above 3000 psi. This step is very important to ensure best system performance.

NOTE

In a properly working system, the electrochemical detection (ED) background for the Dionex CarboPac column QA and most applications is typically 25-45nC.

Figure 13 Plumbing Schematic for ICS-6000 System Dual EGC Mode, 0.4-mm Column



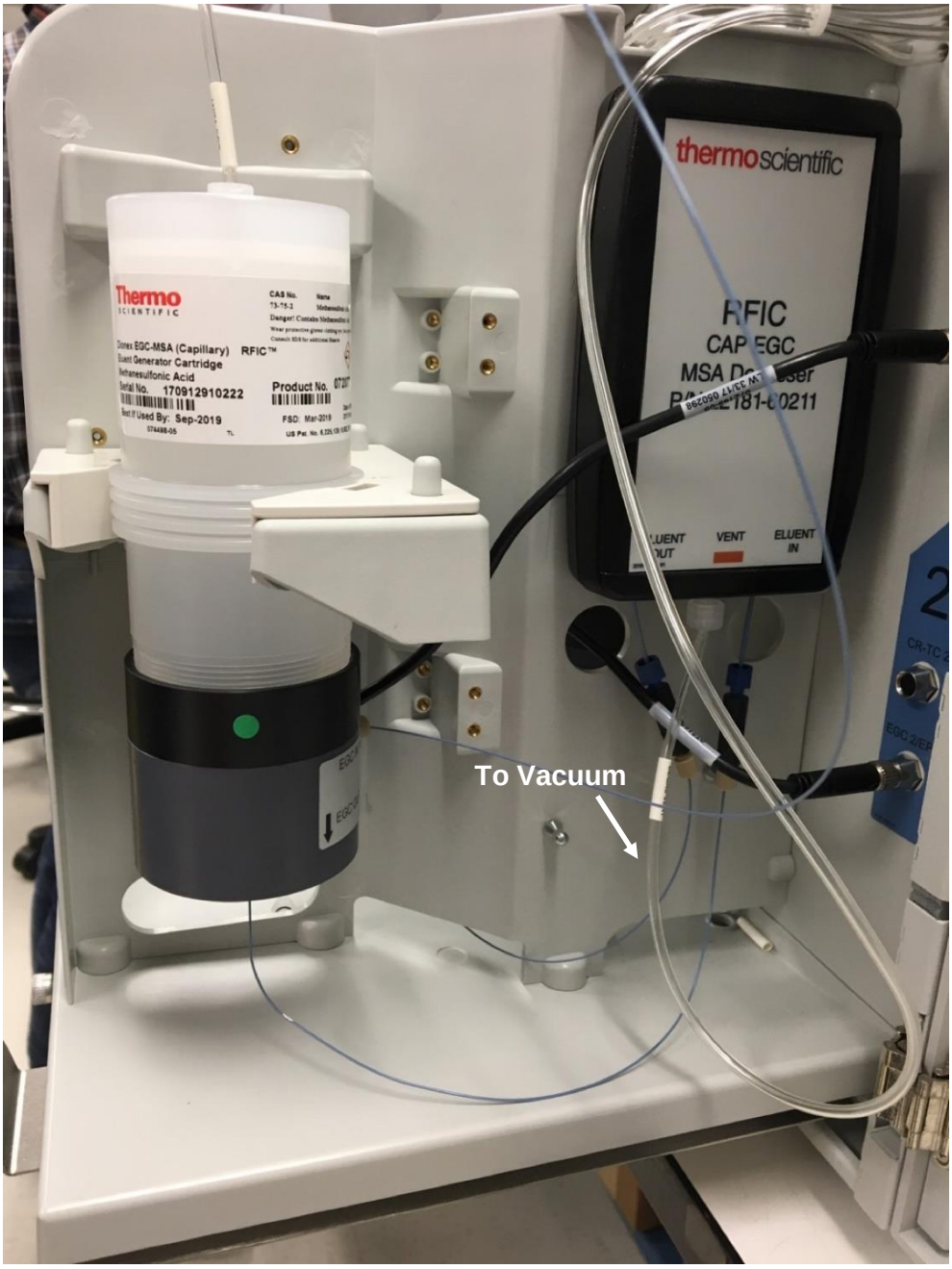
1.2.1 ICS-6000 System Dual EGC Mode Start-up, 0.4-mm Column

Figure 14 Overview of ICS-6000 System Dual EGC Mode, 0.4-mm Column

1. Fill a 2 L eluent reservoir bottle with ASTM filtered, Type I (18-megohm) degassed deionized water. Connect the reservoir to the eluent inlet line of the pump. Keep the water blanketed under an inert gas (helium or nitrogen).
2. Prime the pump as instructed by the system operation manual.
3. Remove the vent plug on the top of the Dionex EGC-MSA (Capillary) cartridge (please refer to Dionex Eluent Generator Cartridges Manual Document No. 065081 for details about preparation of the Dionex EGC cartridges).
4. Connect the pump outlet to the EGC IN port of the Dionex EGC-MSA (Capillary) cartridge and direct the EGC OUT port of the cartridge to waste.
5. Turn on the pump and pump DI water through the Dionex EGC-MSA (Capillary) for 15 minutes at the flow rate of 0.1 mL/min (prime mode).
6. Connect the EGC OUT port of the Dionex EGC-MSA (Capillary) cartridge to a PEEK backpressure tubing (Item # 22181-20032). Direct the backpressure tubing to waste.
7. Set the pump flow rate to 0.020 mL/min.
8. On the E-Panel, under Eluent Generator Tab, set the Eluent Polarity to Acidic Eluents. Set MSAprog to 100 mM. Turn on the Dionex EGC power.
9. Condition the Dionex EGC-MSA (Capillary) for 30 min.
10. Turn off the pump.
11. Remove the PEEK backpressure tubing from the EGC OUT port of the Dionex EGC-MSA (Capillary).

12. Connect the EGC OUT port of the Dionex EGC-MSA (Capillary) cartridge to the ELUENT IN port of the Dionex RFIC Capillary EGC MSA Degasser (Item # 22181-60211). Direct the ELUENT OUT to waste. (Refer to Section 1.2.2 for instructions on connecting the vent of the Dionex RFIC Capillary EGC MSA Degasser to the vacuum system.)

Figure 15 Dionex EGC-MSA (Capillary) Cartridge and Dionex RFIC Capillary EGC MSA Degasser in EG Module of ICS-6000 System Dual EGC Mode, 0.4-mm Column

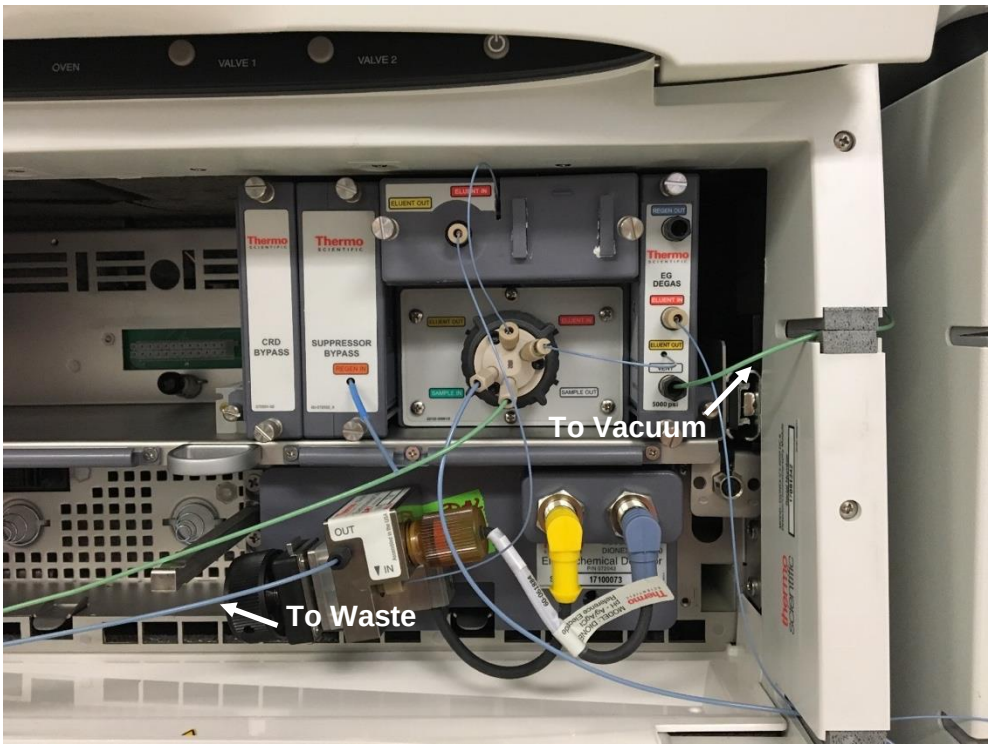


13. Set the pump flow rate to 0.05 mL/min and turn the pump on for 10 min.
14. Remove the vent plug on the top of the Dionex EGC-KOH (Capillary) cartridge.
15. Connect the ELUENT OUT port of the Dionex RFIC Capillary EGC MSA Degasser (Item # 22181-60211) and the EGC IN port of the Dionex EGC-KOH (Capillary) cartridge. Direct the EGC OUT port of the cartridge to waste.

Figure 16 Dionex EGC-KOH (Capillary) Cartridge in EG Module of ICS-6000 System Dual EGC Mode, 0.4-mm Column

16. Turn on the pump and pump DI water through the Dionex EGC-KOH (Capillary) for 15 minutes at the flow rate of 0.1 mL/min.
17. Connect the EGC OUT port of the Dionex EGC-KOH (Capillary) cartridge to a PEEK backpressure tubing (Item # 22181-20032). Direct the backpressure tubing to waste.
18. Set the pump flow rate to 0.020 mL/min.
19. On the E-Panel, under Eluent Generator Tab, set the Eluent Polarity to Basic Eluents. Set KOHprog to 100 mM. Turn on the Dionex EGC power.
20. Condition the Dionex EGC-KOH (Capillary) for 30 min.
21. On the E-Panel, turn the Dionex EGC power OFF and keep the pump running at 0.02 mL/min for 10 min.
22. Turn off the pump.
23. Remove the PEEK backpressure tubing from the EGC OUT port of the Dionex EGC-KOH (Capillary) cartridge.
24. Connect the EGC OUT port of the Dionex EGC-KOH (Capillary) cartridge to the ELUENT IN port of the Dionex RFIC Capillary Eluent Degasser (Item # 22181-60202). (Refer to Section 1.2.2 for instructions on connecting the vent of the Dionex RFIC Capillary Eluent Degasser to the vacuum system.)

Figure17 View of IC Cube in DC Module of ICS-6000 System Dual EGC Mode, 0.4-mm Column



25. Connect the ELUENT OUT port of the Dionex Capillary RFIC Eluent Degasser (Item # 22181-60202) to a PEEK backpressure tubing (Item # 22181-20032). Direct the backpressure tubing to waste.
26. With EGC power OFF, turn on the pump at the flow rate of 0.020 mL/min. Flush the Dionex RFIC Capillary Eluent Degasser (Item # 22181-60202) for 15 min.
27. Turn off the pump.
28. Remove the PEEK backpressure tubing (Item # 22181-20032) from the ELUENT OUT port of the Dionex RFIC EGC Capillary Eluent Degasser (Item # 22181-60202).
29. Connect the ELUENT OUT of the Dionex RFIC Capillary Eluent Degasser (Item # 22181-60202) to 2 port of the injection valve.
30. Install the column to 3 port of the injection valve. Direct the OUTLET of the column to waste.
31. With EGC power OFF, turn on the pump and pump DI water at the flow rate of 0.010 mL/min for 15 min.
32. With Eluent Polarity set to Basic Eluents, set KMSAprog and KOHprog to the desired concentrations for the applications. Turn on the Dionex EGC power for 60 min.
33. Connect the OUTLET of the column to the CELL IN of the ED cell, and direct the CELL OUT to waste.
34. Confirm that the pH is >11. With the pH within this range, turn on the cell using the carbohydrate standard quad waveform. Let the system equilibrate for 30 min.

NOTE

To achieve optimum sensitivity, use Au on PTFE disposable electrodes with 1 mil gasket (P/N XXXXXX) for ED detection (carbohydrate quadruple waveform), and use gasket for pH-Ag/AgCl reference electrode (P/N 072162) in the reference chamber of the ED cell.

1.2.2 ICS-6000 System Dual EGC Mode Vacuum Kit Installation Procedure, 0.4-mm Column

NOTE

To ensure stable baseline and low background noise, it is crucial to have sufficient removal of the hydrogen and oxygen gas formed with the EGC generated eluents. Connect the vents of the Dionex RFIC Capillary EGC MSA Degasser and the Dionex RFIC Capillary Eluent Degasser to the Vacuum Port located at the back of the Dionex DP module. Make sure all connections and fittings for the vacuum degas are vacuum tight before using the system.

A. With DP module power off, remove the plug from the vacuum port located at the back of the DP module.
B. Cut a 12-inch (300 mm) length of the yellow polyurethane air tubing. Slide the ¼-28 x 1/8 tan fitting bolt (Item # 052267) onto the air tubing, then slide the yellow Tefzel ferrule (Item # 048949) onto the tubing.
C. Connect this fitting to the vacuum port on the DP module.
D. Connect the other end of the tubing to the stainless-steel tee.
E. Connect the one end of the tee to one of the tubing labeled “EGC vent” installed in the DP module.
F. Connect the other end of one “EGC vent” tubing to the fitting that is located at the vent of Dionex RFIC Capillary EGC MSA Degasser (Item # 22181-60211).
G. Connect a second 12-inch piece of polyurethane air tubing to the remaining end of the stainless-steel tee.
H. Slide the ¼-28 × 1/8 tan fitting bolt (Item # 052267) onto the air tubing, then slide the yellow Tefzel ferrule (Item # 048949) onto the tubing.
I. Connect this fitting to the ¼-28 union (Item # 039296).
J. To connect the gas vent line for the Dionex RFIC EGC Capillary Eluent Degasser (Item # 22181-60202), slide the ¼-28 × 1/16 fitting bolt (Item # 052230) onto the green PEEK tubing (0.030 in. i.d.), and slide the two-piece ferrule (Item # 062511) onto the tubing, connect this green tubing to the ¼-28 union (Item # 039296) to connect the gas vent line to the vacuum.
K. Make sure all the connections are properly tightened.
L. Turn on the power of the DP module.
M. Go to Commands (F8) on the Chromeleon Console, select Pump_1, right click on the Properties panel, and check Expert.
N. Under Properties tab, got to Degasser Vacuum, the Value should read OK. This indicates that the completion of the vacuum installation.
O. If the Value reads Not OK, check for leaks at all the connections for vacuum. Check the status of the vacuum pump located inside the DP module to ensure that it functions properly.

Figure 7 Vacuum Setup at DP Module of ICS-6000 System Dual EGC Mode, 0.4-mm Column

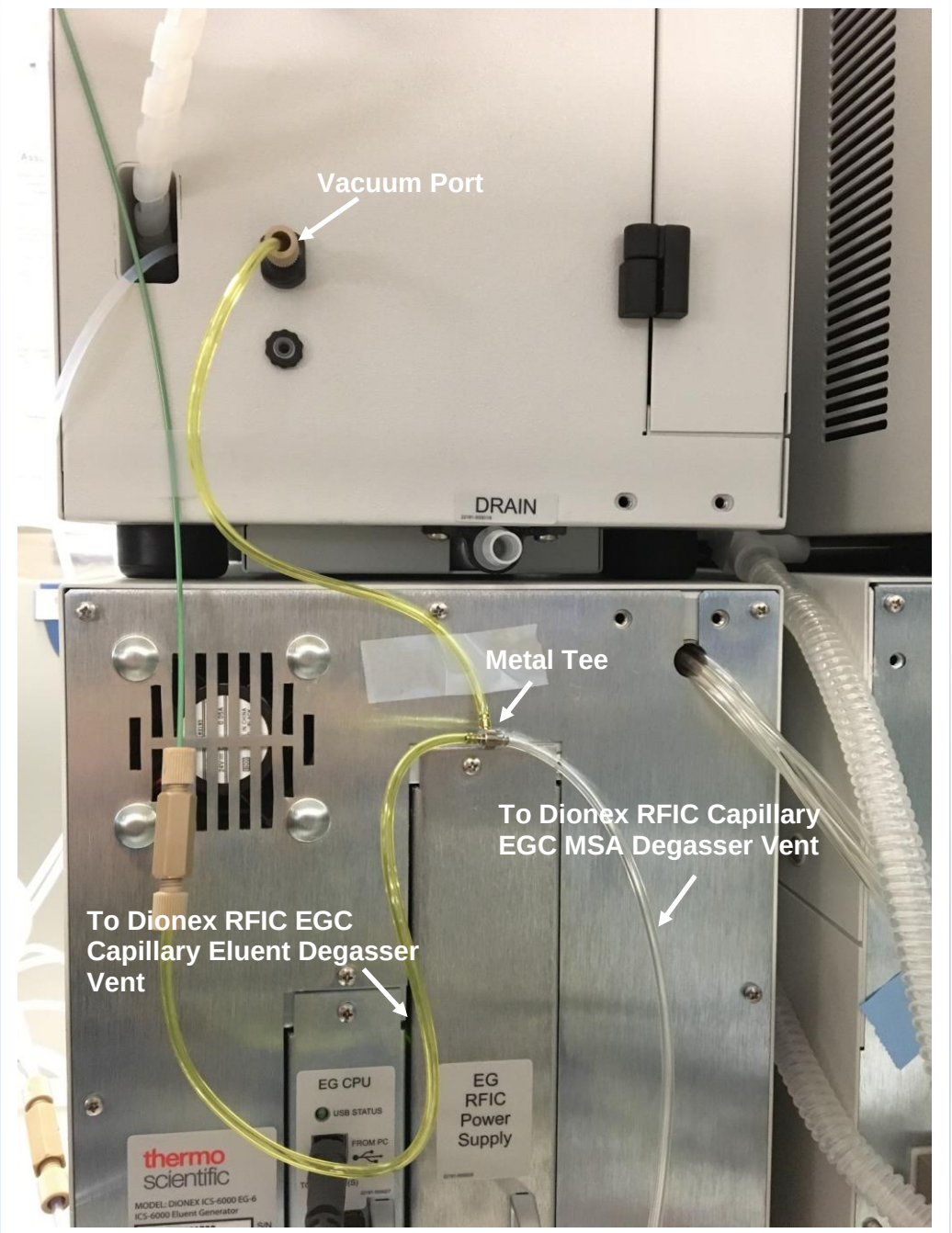
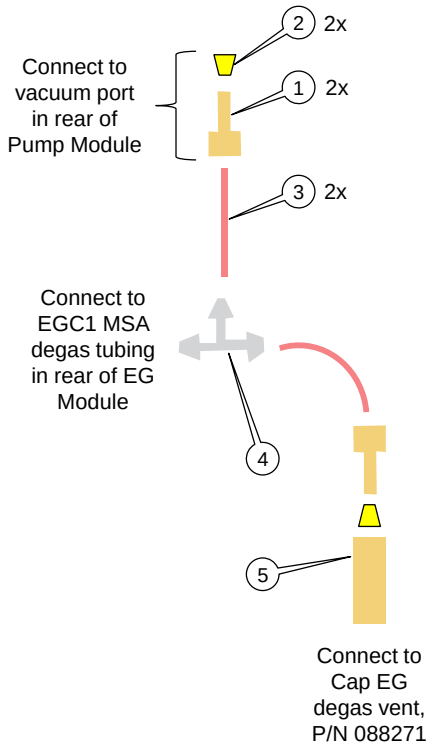


Figure 8 Vacuum Line Connections for Dual EGC Mode, 0.4-mm Column



#	Item #	Description	Qty.	UOM
1	052267	FTG,NUT,FLNGLS,1/4-28X1/8 PK	3	Ea
2	048949	FTG,FER,FLNGLS,1/8 TFZL	3	Ea
3	030091	TUBING,AIR,062X125 PU,YEL	10	Ft
4	035690	FTG,BARB,TEE,1/16. NAT,PP	9	Ea
5	039296	FTG,UNION,1/4-28,ENDS,02-P1	2	Ea

1.2.3 ICS-6000 System Dual EGC Mode Re-Start Procedure, 0.4-mm Column

NOTE

The systems under Dual EGC mode are designed to be operated non-stop for months at a time. If the system has been shut down for more than 5 days, the system should be re-started using the following procedure (applied to both analytical and capillary systems).

A. Disconnect all the components after the Dionex EGC-KOH (Cap) cartridge from the system.
B. Direct the EGC OUT of the Dionex EGC-KOH (Cap) cartridge to waste.
C. Set the flow rate to 0.02 mL/min. Turn the pump and EGC power on. With Eluent Polarity set to Basic Eluents, set $KMSA_{prog}$ to 50 mM and KOH_{prog} to 20 mM. Allow the system to run for 30 min.
D. Turn the EGC power off.
E. Keep the pump on at 0.02 mL/min for 10 min.
F. Turn the pump off.
G. Connect the OUTLET of the Dionex EGC-KOH (Cap) cartridge to the ELUENT IN of the Dionex RFIC capillary eluent degasser (Item # 22181-60202). Direct the ELUENT OUT of the Dionex RFIC capillary eluent degasser to waste.
H. Set the flow rate to 0.02 mL/min and turn the pump on for 10 min. Keep EGC power off.
I. Turn the pump off.
J. Connect the ELUENT OUT of the Dionex RFIC Capillary Eluent Degasser (Item # 22181-60202) to 2 port of the injection valve.
K. Install the CarboPac PA200 (0.4 x 50 mm) guard column and the CarboPac PA200 (0.4 x 250 mm) separator column to 3 port of the injection valve. Direct the OUTLET of the column to waste.
L. Set the flow rate to 0.01 mL/min With EGC power off, pump DI water through the system for 10 min.
M. Turn the pump off.
N. Connect the outlet of the separator column to the ED cell.
O. Direct the outlet of the ED cell to waste.
P. Turn the pump and EGC power on. Set to the required application condition.
Q. When the pH reading of the ED detector shows pH > 11, turn the ED cell on.
R. Allow the system to equilibrate for 30-45 minutes prior to collecting data.

1.2.4 ICS-6000 System Dual EGC Mode Dual Channel, 0.4-mm Column

Figure 20 Capillary (0.4-mm) / Capillary (0.4-mm) Dual Channel Flow Diagram

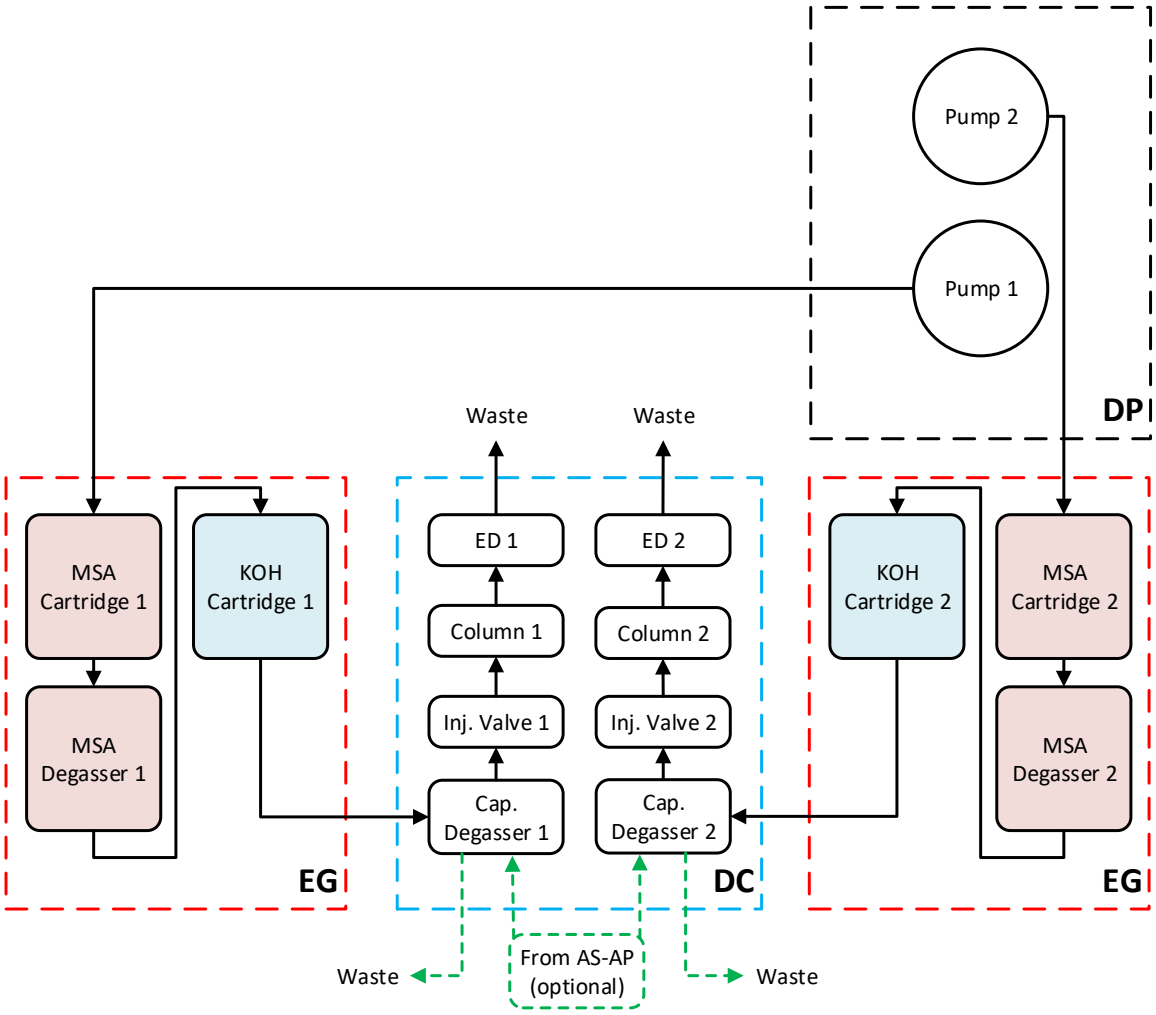
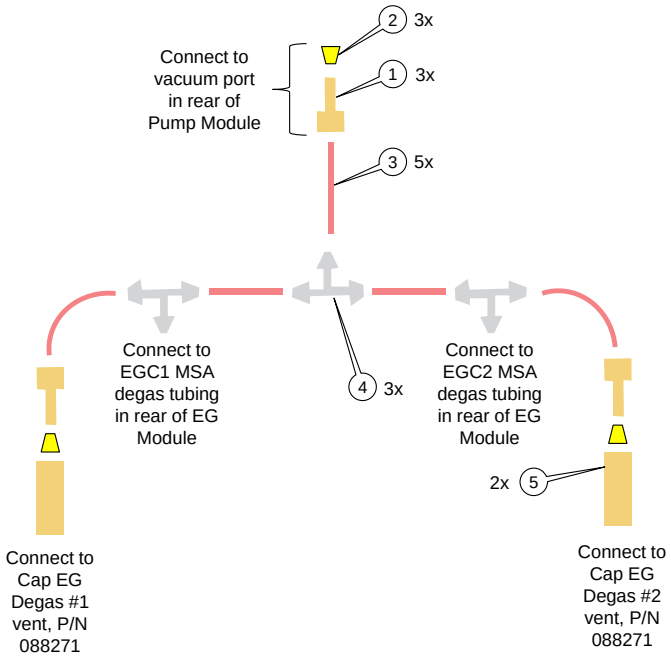


Figure 21 Dual Channel Dual EGC Mode Vacuum Connection, 0.4-mm Column



#	Item #	Description	Qty.	UOM
1	052267	FTG,NUT,FLNGLS,1/4-28X1/8 PK	3	Ea
2	048949	FTG,FER,FLNGLS,1/8 TFZL	3	Ea
3	030091	TUBING,AIR,062X125 PU,YEL	10	Ft
4	035690	FTG,BARB,TEE,1/16. NAT,PP	9	Ea
5	039296	FTG,UNION,1/4-28,ENDS,02-P1	2	Ea

1.3 ICS-6000 System Dual EGC Mode Hybrid Dual Channel, 1-mm/0.4-mm Column

Figure 22 Analytical (1-mm) / Capillary (0.4-mm) Dual Channel Flow Diagram

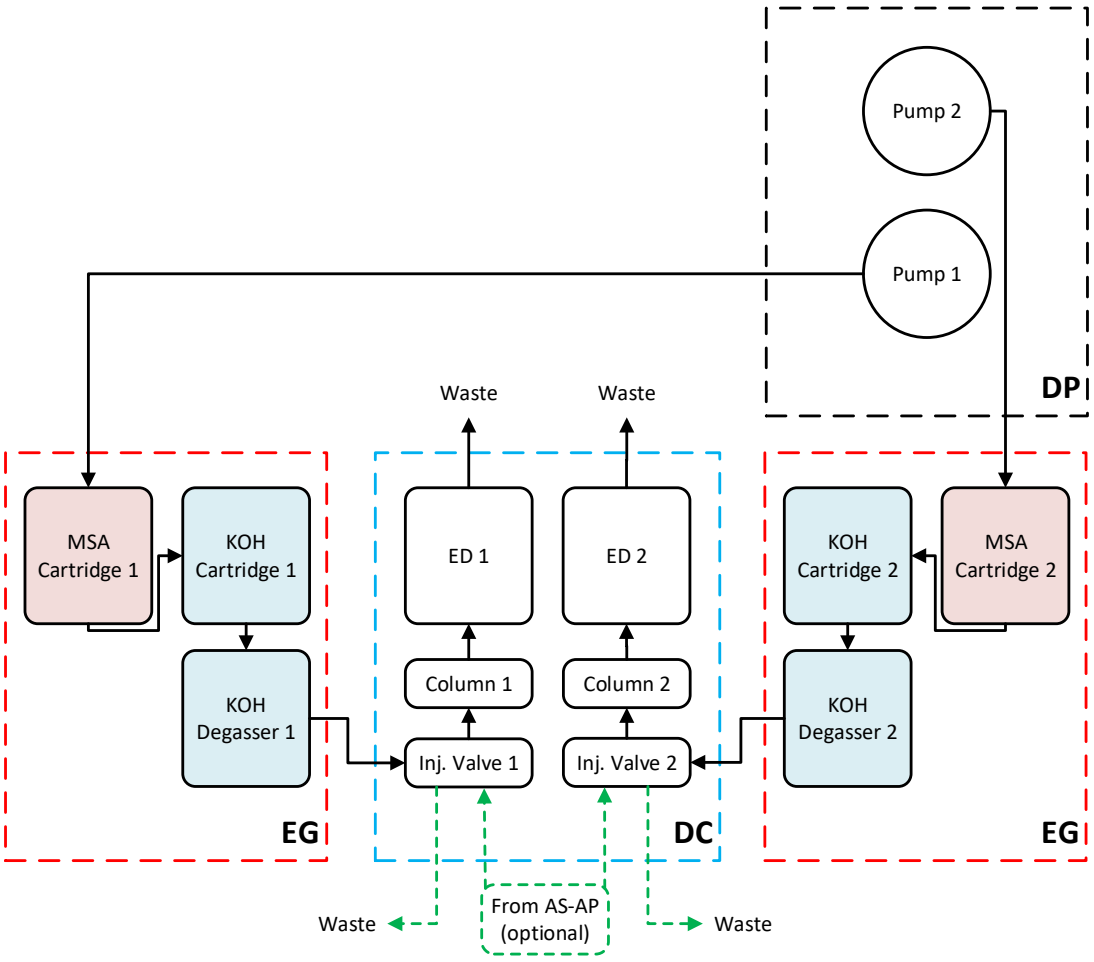
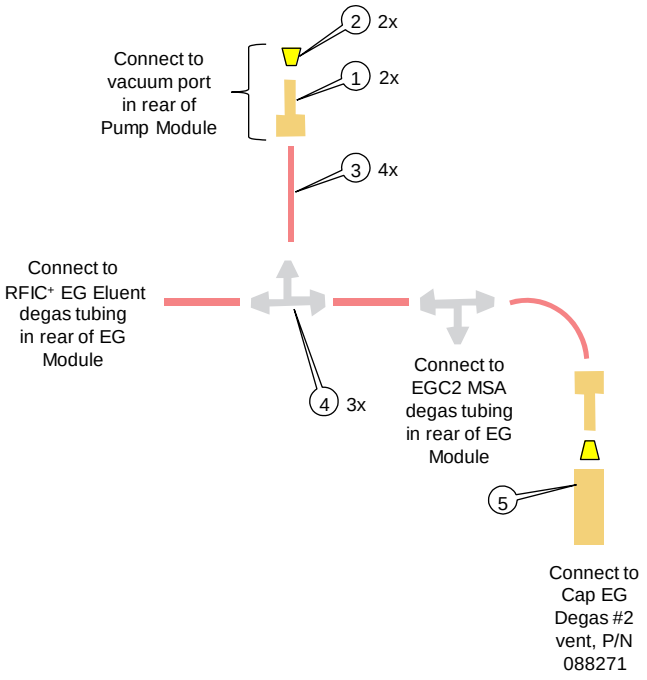


Figure 9 Dual Channel Hybrid Dual EGC Mode, Vacuum Connection



#	Item #	Description	Qty.	UOM
1	052267	FTG,NUT,FLNGLS,1/4-28X1/8 PK	3	Ea
2	048949	FTG,FER,FLNGLS,1/8 TFZL	3	Ea
3	030091	TUBING,AIR,062X125 PU,YEL	10	Ft
4	035690	FTG,BARB,TEE,1/16. NAT,PP	9	Ea
5	039296	FTG,UNION,1/4-28,ENDS,02-P1	2	Ea

2. Chromeleon 7.2 User Guide

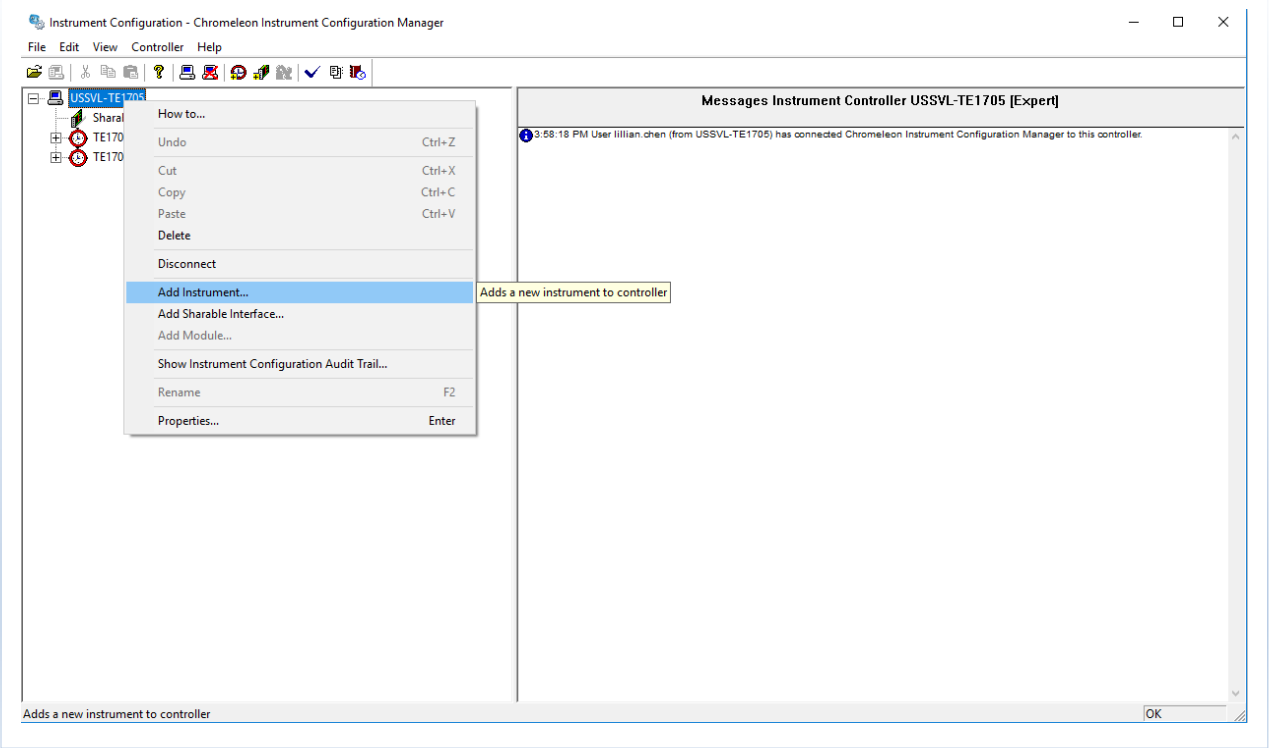
The purpose of this User Guide is to help you set up configuration and instrumental method in Chromeleon 7 while using Dionex ICS-6000 System Dual EGC Mode. The content of this document focuses on the settings related to Dual EGC Mode only. For more information on installation of Chromeleon 7, please refer to the Chromeleon 7 Chromatography Data System Installation Guide (Software Version 7.2.7, November 2017).

2.1 Set up configuration for Dual EGC Mode

2.1.1 Create an Instrument

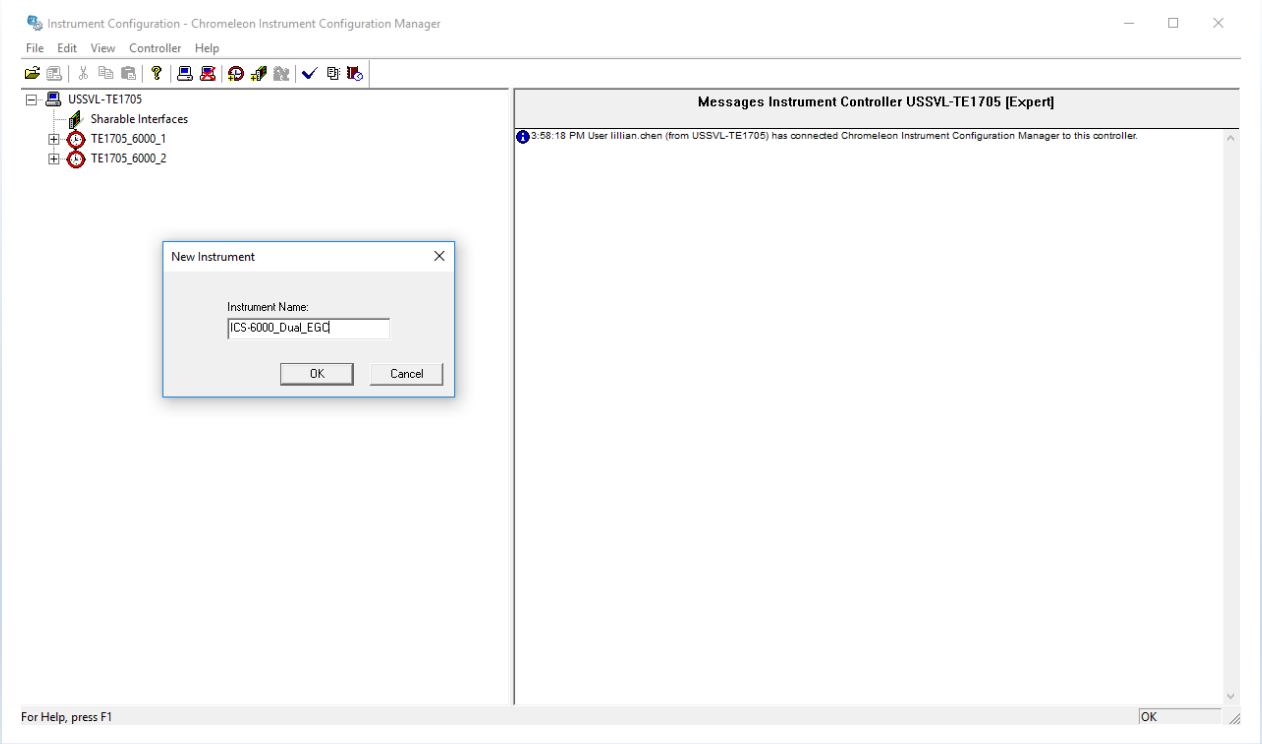
A. On the **Chromeleon Configuration**, right click inside the instrument panel, click **Add Instrument**.

Figure 10 Instrument Configuration: Adding an Instrument



B. In the pop-up window, type in the name of the **New Instrument**.

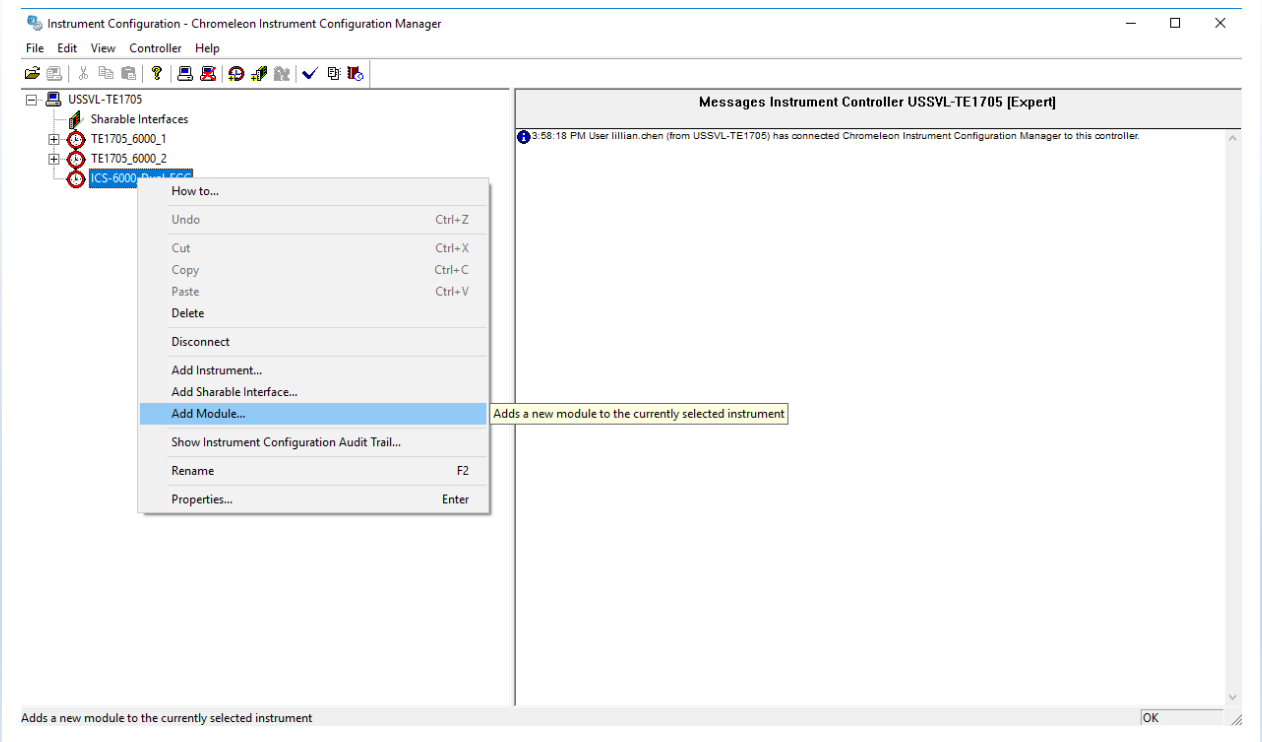
Figure 11 Instrument Configuration: Naming a New Instrument



2.1.2 Add Modules for the Instrument

A. Select the **New Instrument** by clicking on the name of the Instrument, then right click and select **Add Module** to enter the pop-up window of "**Add module to instrument**".

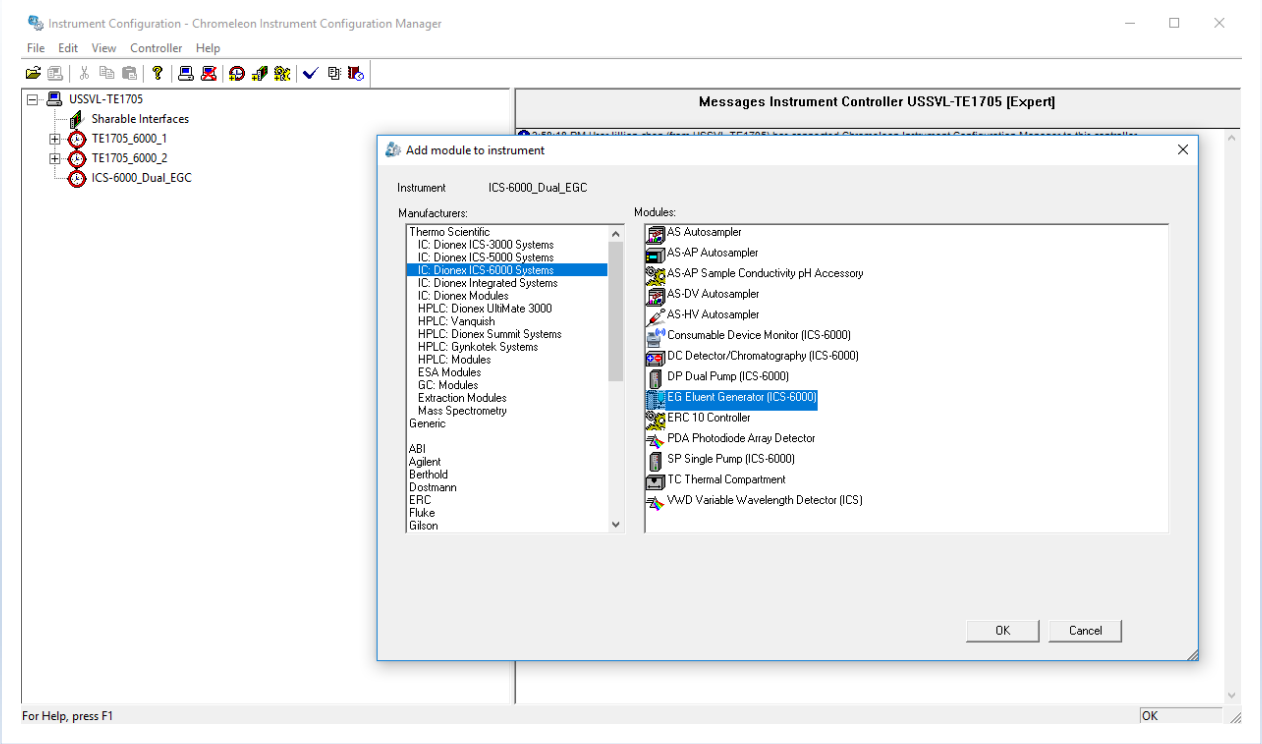
Figure 12 Instrument Configuration: Add Modules



B. In the "**Add module to instrument**" window, select **IC: Dionex ICS-6000 Systems**. In the Modules column, select the modules (e.g. DP or SP, DC, EG, AS-AP).

C. Follow the instruction in Chromeleon 7 Chromatography Data System Installation Guide (Software Version 7.2.7, November 2017) to add the modules.

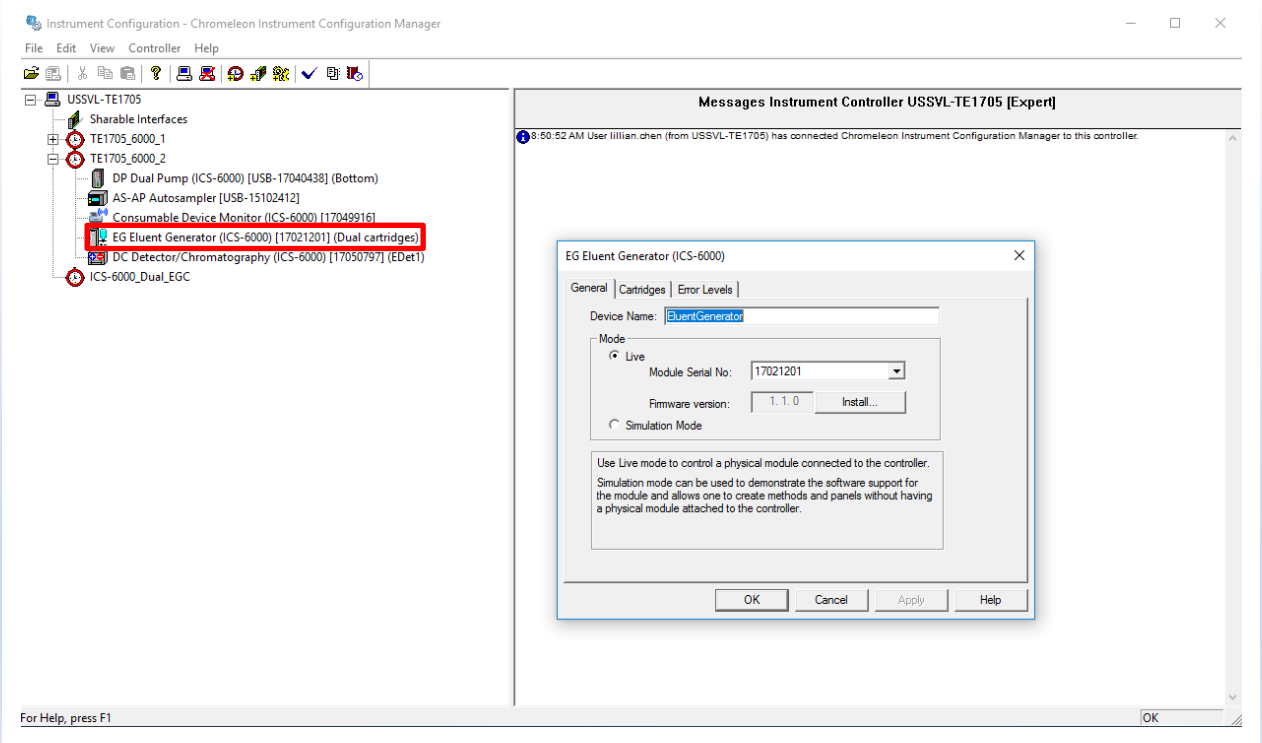
Figure 13 Instrument Configuration: Add EG module



2.1.3 Configure the Eluent Generator Module

A. On the pop-up window of "**EG Eluent Generator (ICS-6000)**", Under **General** tab, select the **Module Serial Number** of the module.

Figure 14 Instrument Configuration: Configure EG module



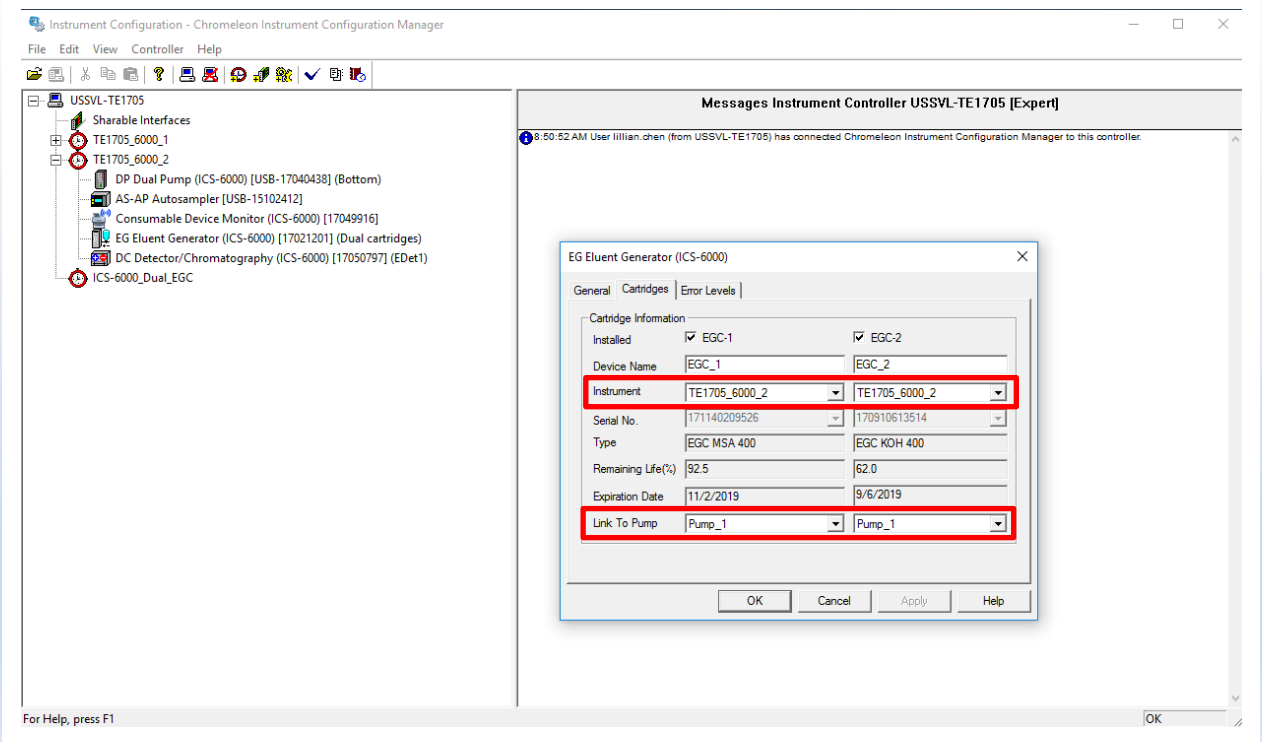
B. Under **Cartridges** tab, click to check both the **EGC-1** and **EGC-2** check boxes.

C. Go to **Instrument**, and click the drop down selection. Select the same ICS-6000 instrument for both the **EGC-1** and **EGC-2**.

D. Go to **Link To Pump**, and click the drop down selection. Select the pump that is plumbed to for both the **EGC-1** and **EGC-2** (the **EGC-1** and **EGC-2** are required to be linked to the same pump).

E. Click **OK** to save the settings.

Figure 15 Instrument Configuration: Configure EGC-1 and EGC-2



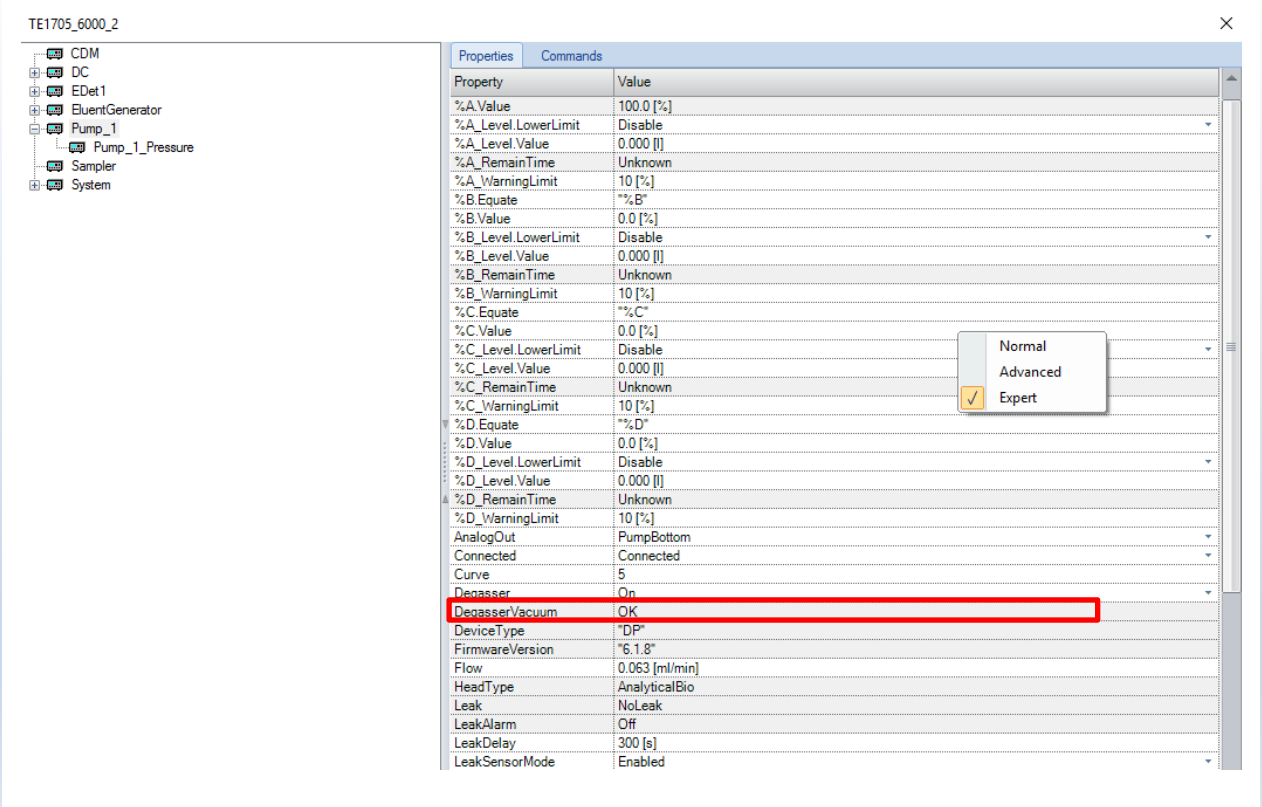
F. After saving the settings, the text of **Dual cartridges** is shown next to the serial number of the EG module on the **Instrument Configuration** main page.

2.1.4 Set Degasser Malfunction Error Levels in the Dual Pump/Single Pump Module

To ensure stable baseline and low background noise, it is crucial to have sufficient removal of the hydrogen and oxygen gas formed with the EGC generated eluents. To For 1-mm system, connect the vent of the Dionex RFIC$^+$ Dual EG Eluent Degasser to the Vacuum Port located at the back of the Dionex DP module. For 0.4-mm system, connect the vents of the Dionex RFIC Capillary EGC MSA Degasser and the Dionex RFIC Capillary Eluent Degasser to the Vacuum Port located at the back of the Dionex DP module. Make sure all connections and fittings for the vacuum degas are vacuum tight before using the system.

To check the status of the system vacuum, go to **Command** (F8) , at the pop-up window, right click and check **Expect**. Under **Properties** tab, in **Property** column, find **Degasser Vacuum**, the value should be **OK**. If it shows Not OK, check if there is any leak in the connection of the vacuum system, or check the status of the vacuum pump inside the DP or SP module.

Figure 16 Commands: Degasser Vacuum Status

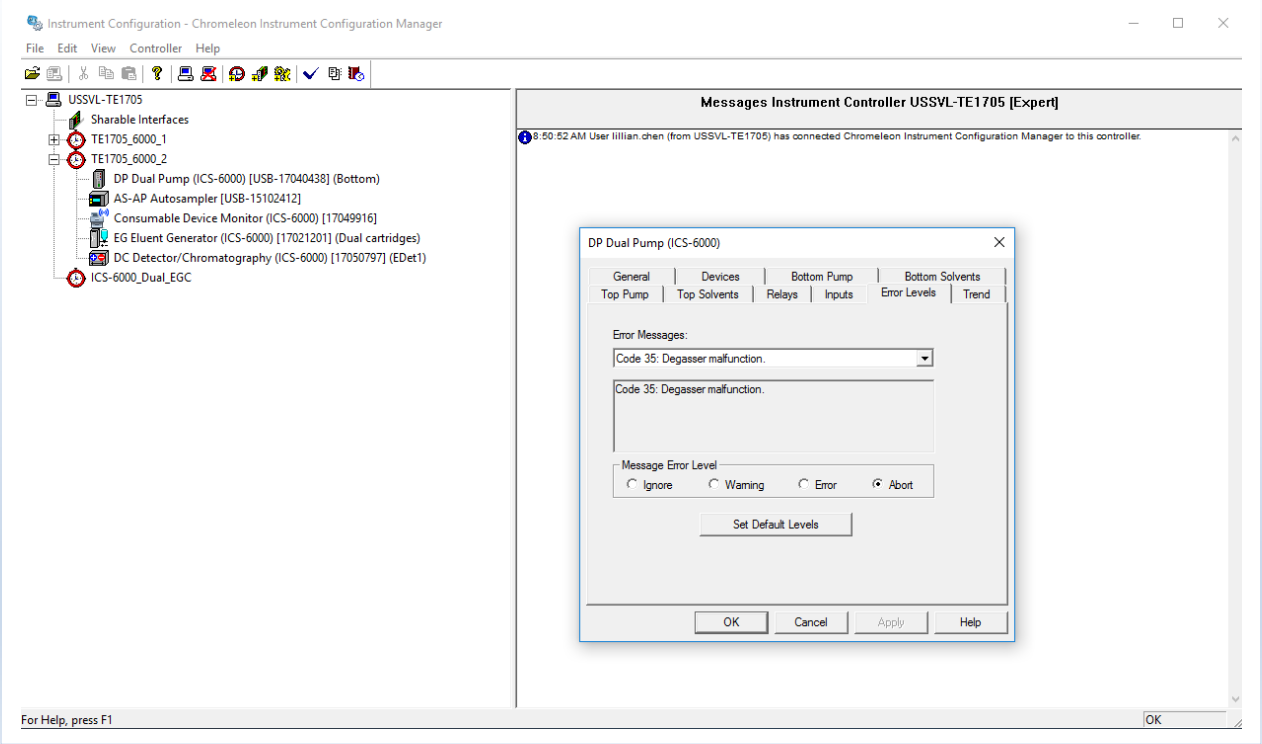


A. On the pop-up window of "**DP Dual Pump (ICS-6000)**" or "**SP Single Pump (ICS-6000)**", Under **Error Levels** tab, go to **Error Messages**, and click the drop-down selection and select **Code 35: Degasser malfunction**.

B. Go to **Message Error Level**, click to check **Abort** check box.

C. Click **OK** to save the setting.

Figure 17 Instrument Configuration: Set Message Error Level for Degasser Malfunction



2.1.5 Set Pressure Limit in the Dual Pump/Single Pump Module

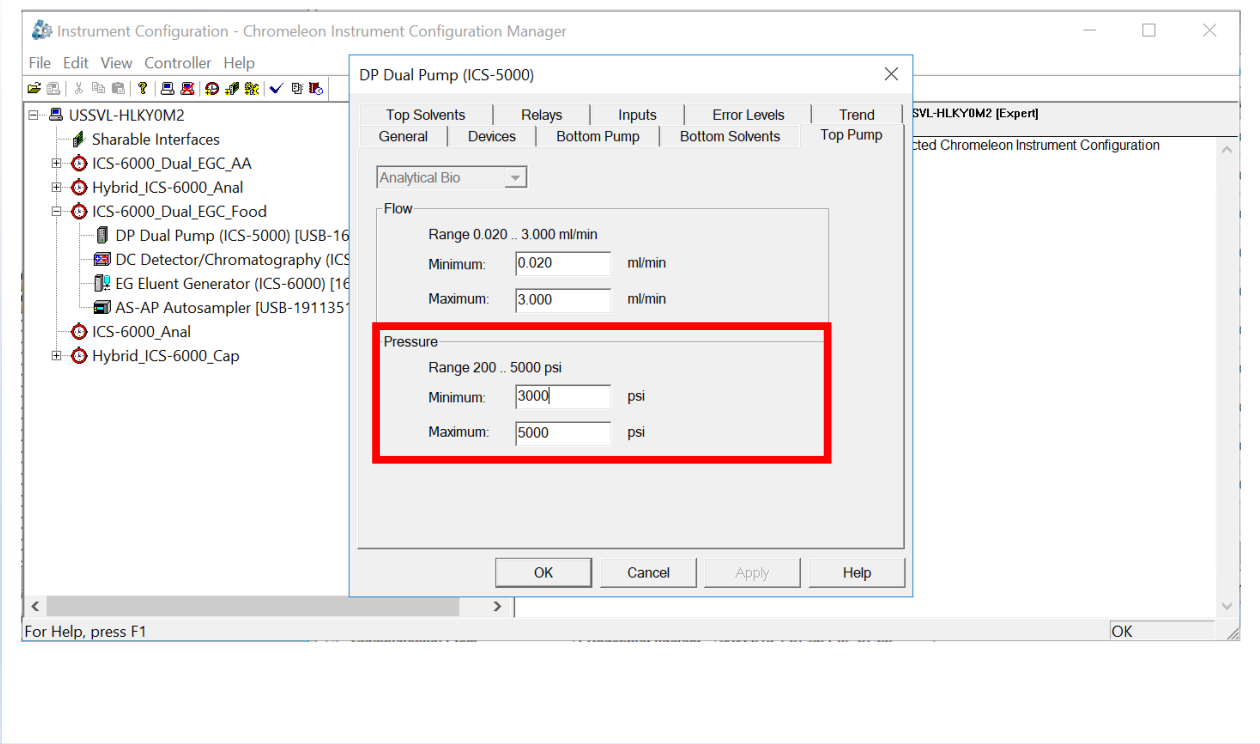
To ensure sufficient removal of the hydrogen and oxygen gas formed with the EGC generated eluents, backpressure applied at the outlet of the Dionex RFIC^{+} Dual EG Eluent Degasser (Item # 22181-60951)must be above 2500 psi. To prevent gas from breaking through, in the case of dropped backpressure due to leaks at the connections downstream, it's recommended to increase the setting of minimum pressure of the pump.

A. On the pop-up window of "**DP Dual Pump (ICS-6000)**" or "**SP Single Pump (ICS-6000)**", go to **Bottom Pump** or **Top Pump** tab, depending on which pump (**bottom pump** or **top pump**) is plumbed to both the **EGC-1** and **EGC-2**.

B. Go to **Pressure**, and type 3000 psi in the **Minimum** box.

C. Click **OK** to save the setting.

Figure 18 Instrument Configuration: Set Minimum Limit for Pump Pressure



2.2 E-panels under Dual EGC Mode

A. After saving the settings in **Instrument Configuration**, start the **Chromeleon Console**.

B. Under the **Home** tab, the cartoon of two cartridges with concentration settings is shown on the EG module (Figure 33).

C. Figure 34 shows the **Eluent Generator** tab.

Figure 19 Chromeleon Console: Home e-Panel of Dual EGC Mode

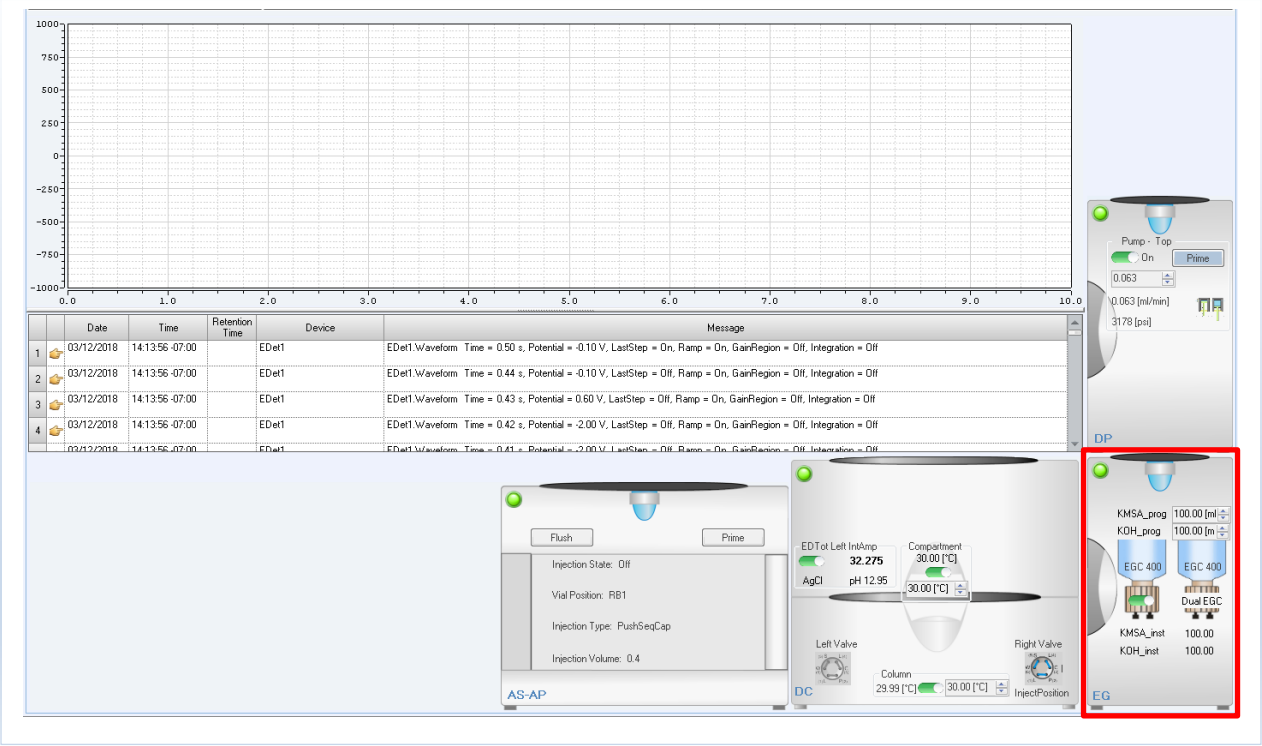
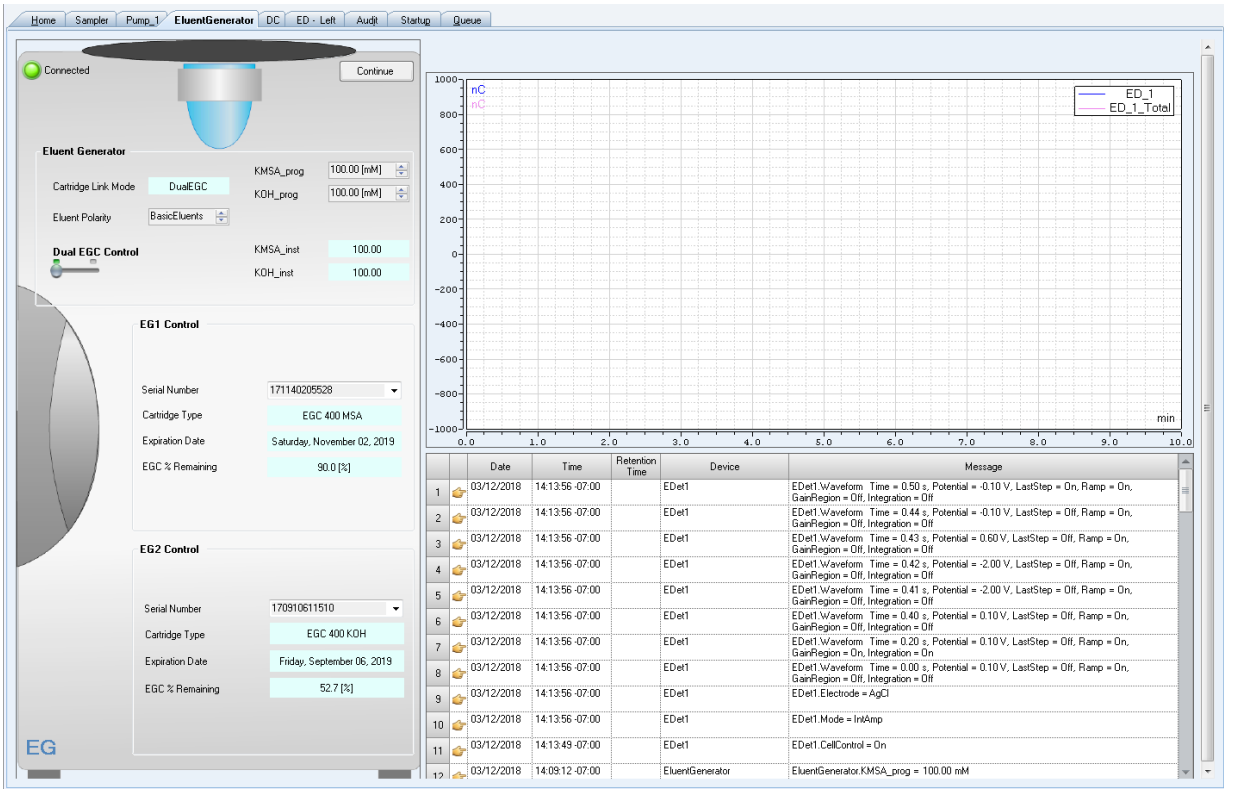


Figure 20 Chromeleon Console: Eluent Generator e-Panel of Dual EGC Mode

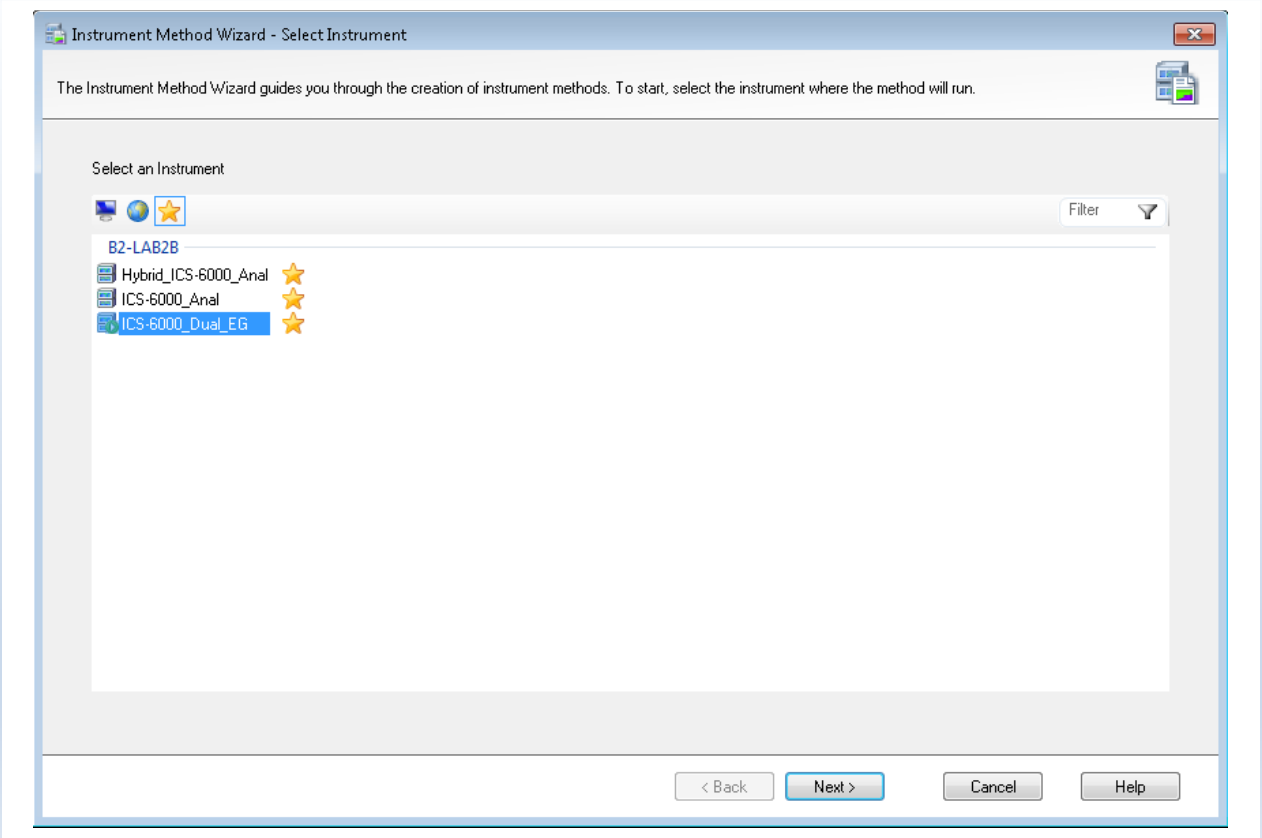


2.3 Create an Instrumental Method for Dual EGC Mode

A. In **Chromeleon Console**, on the Windows taskbar, click **Create** > **Instrument Method**, to open **Instrument Method Wizard**.

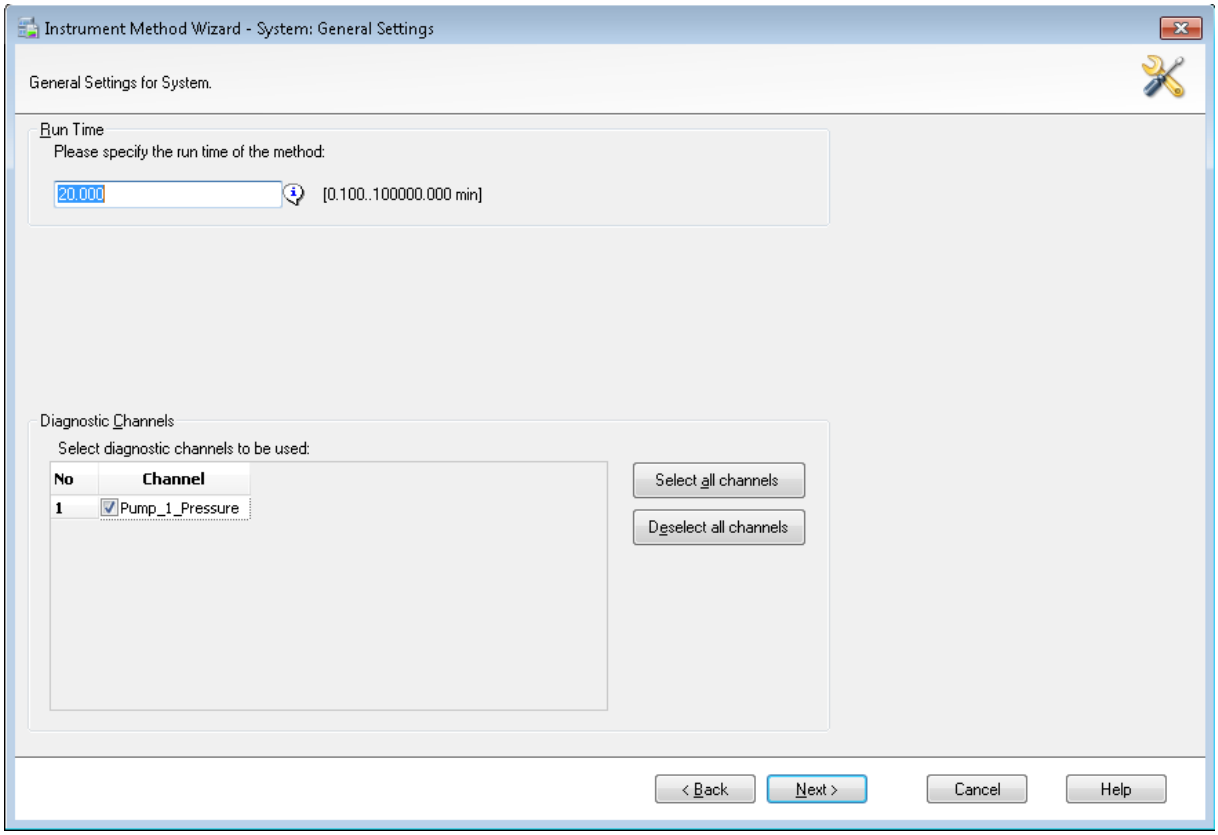
B. On the **Instrument Method Wizard**, select the **Instrument** created from **Instrumental Configuration**. Click **Next**.

Figure 21 Chromeleon Console: Instrument Method Wizard – Select Instrument



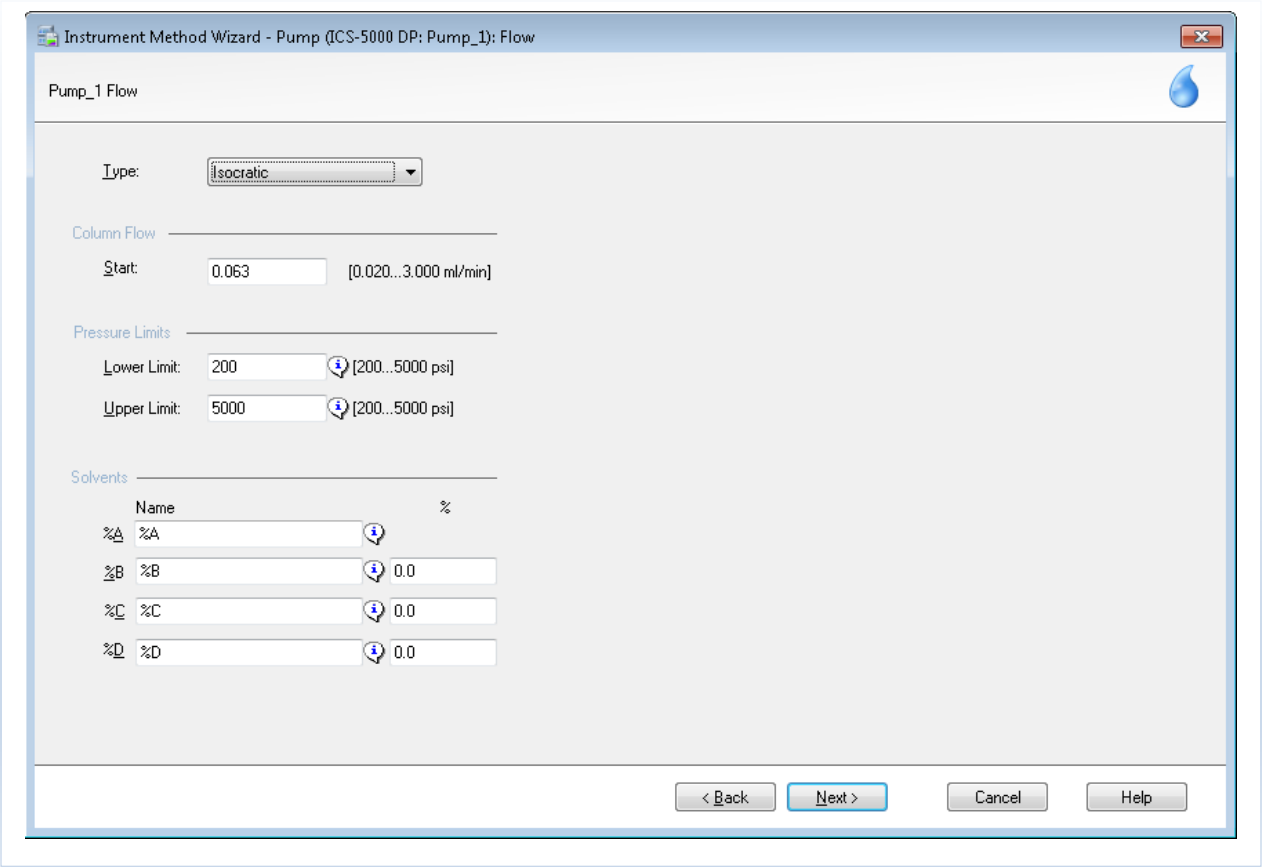
C. On the **System General Settings** page, type in the **run time** of the method. Click **Next**.

Figure 22 Chromeleon Console: Instrument Method Wizard – System General Settings – Run Time



D. On the **Pump Flow** page, type in the **flow rate** of the method. The standard flow rate for 1-mm column and 0.4-mm column are 0.063 and 0.010 mL/min, respectively.

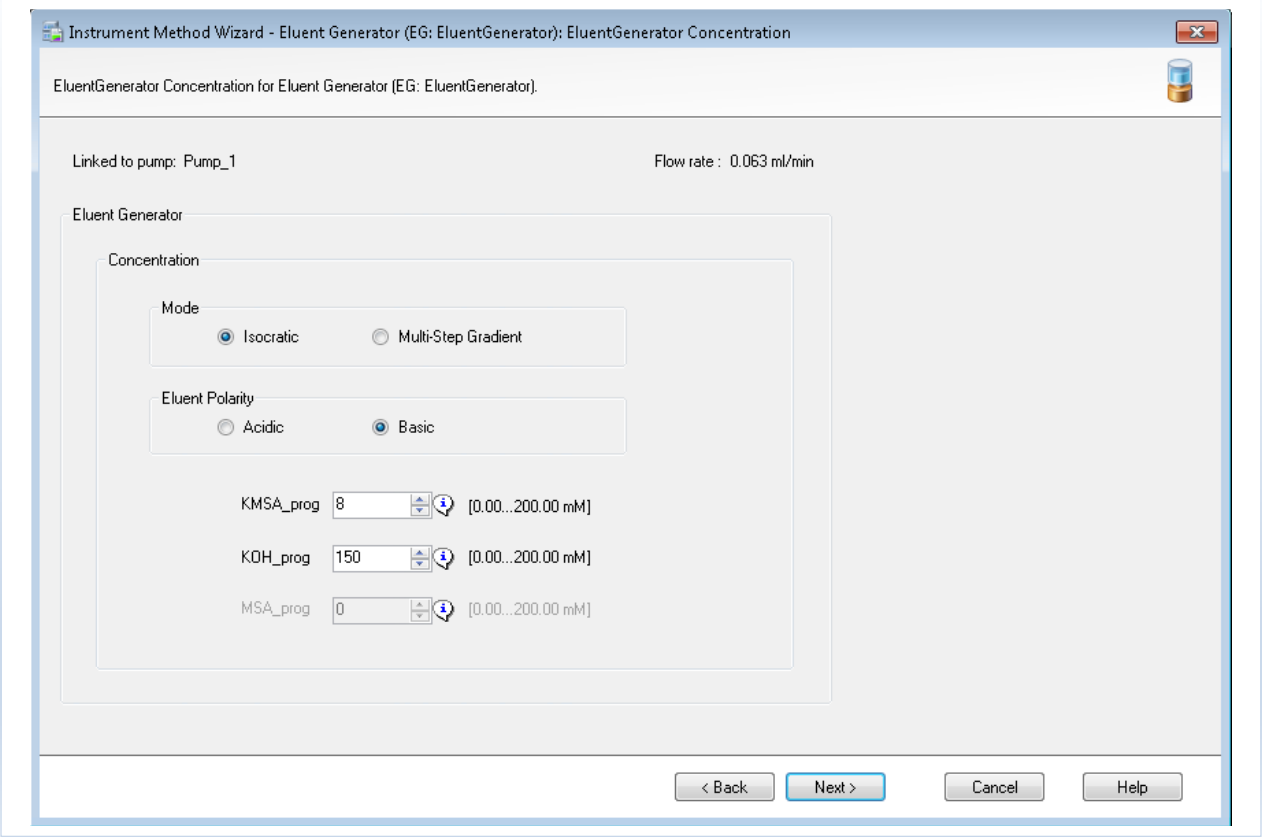
Figure 23 Chromeleon Console: Instrument Method Wizard – Pump Flow



E. On the **Eluent Generator Concentration** page, select the Concentration **Mode**.

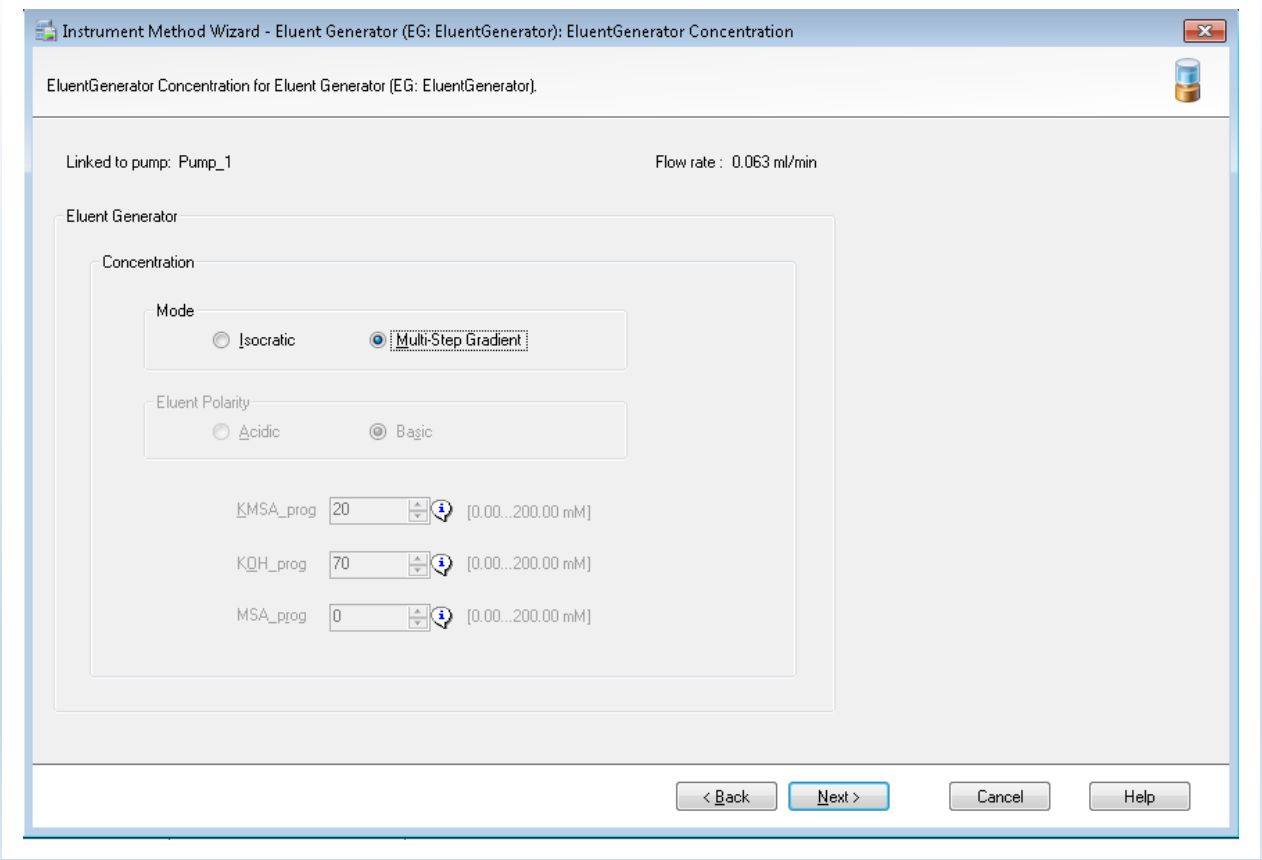
   a. For **isocratic** method, select **Isocratic**. Go to **Eluent Polarity** and select **Basic**. Type in the desired concentration of **KMSA_prog** and **KOH_prog**. Click **Next** to enter the settings for autosampler or detector.

Figure 24 Chromeleon Console: Instrument Method Wizard – Eluent Generator Concentration: Select Isocratic Mode



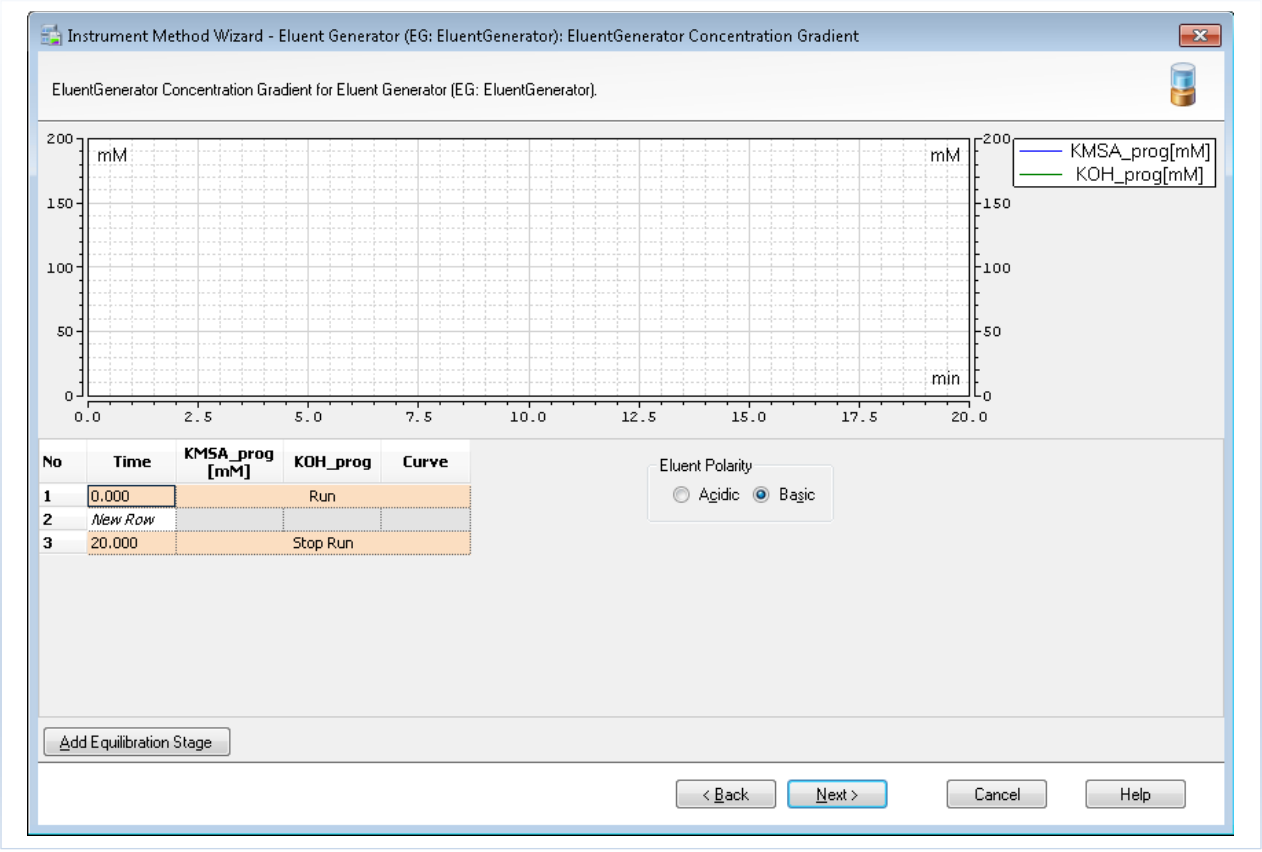
b. For **gradient** method, select **Multi-Step Gradient**. Click **Next**.

Figure 25 Chromeleon Console: Instrument Method Wizard – Eluent Generator Concentration: Select Multi-Step Gradient Mode



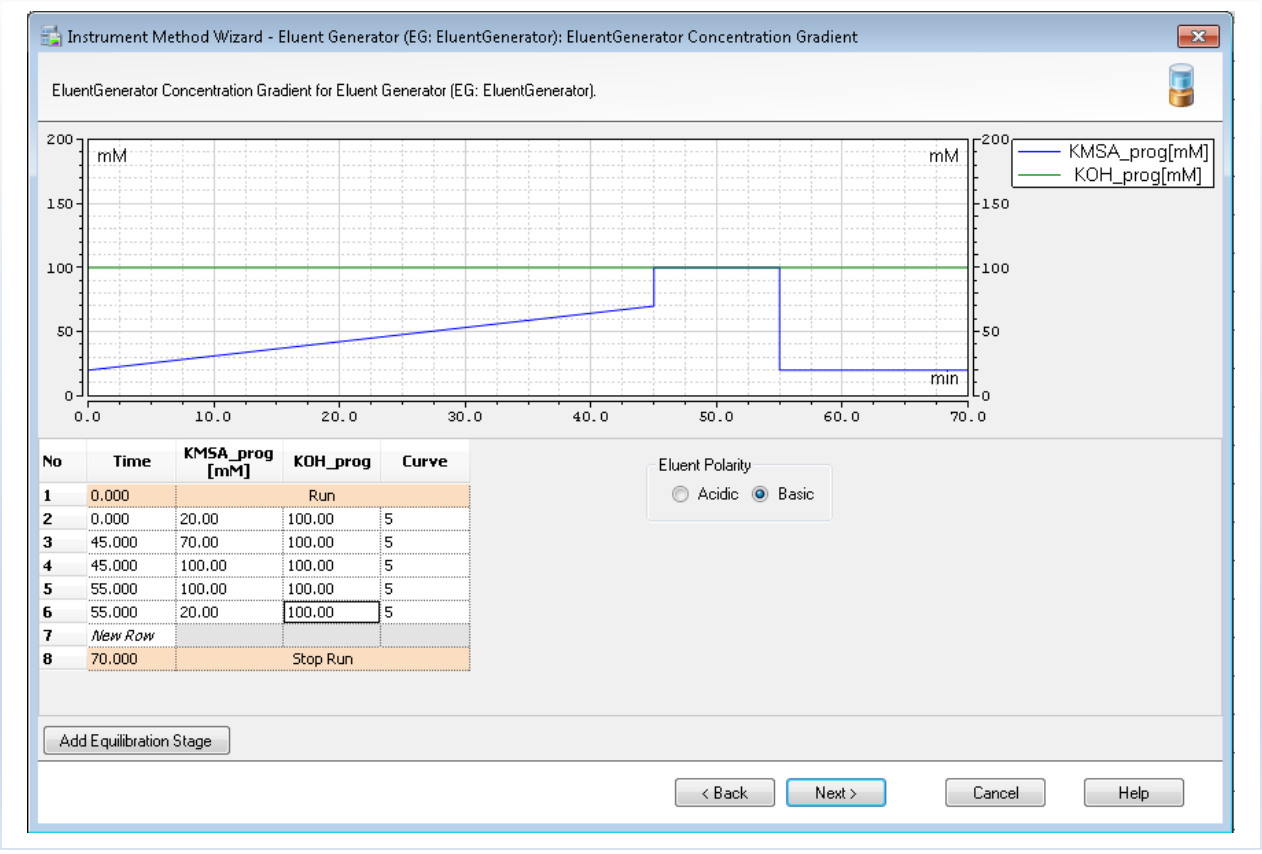
i. On the **Eluent Generator Concentration Gradient** page, go to **Eluent Polarity**, select **Basic**.

Figure 26 Chromeleon Console: Instrument Method Wizard – Eluent Generator Concentration Gradient: Select Eluent Polarity



ii. On the parameter **table**, enter the time, concentration, and curve for the gradient condition. Click **Next** to enter the settings for autosampler or detector.

Figure 27 Chromeleon Console: Instrument Method Wizard – Eluent Generator Concentration Gradient: Enter gradient parameters



No	Time	KMSA_prog [mM]	KOH_prog	Curve
1	0.000	Run		
2	0.000	20.00	100.00	5
3	45.000	70.00	100.00	5
4	45.000	100.00	100.00	5
5	55.000	100.00	100.00	5
6	55.000	20.00	100.00	5
7	*New Row*			
8	70.000	Stop Run		

3. Troubleshooting

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex ICS-6000 system Dual EGC Mode. For more information on problems that originate with the Ion Chromatograph (IC), refer to the Troubleshooting Guide in the appropriate operator's manual. Remember that some of the problems may be related to parts of your experimental protocol (sample contamination, imprecision during sample transfer, etc.). The following text should help you to locate and eliminate problems traceable to the carbohydrate hardware and chemistries. It also provides a selection of cleanup and reconditioning procedures that have been found effective by many users.

NOTE

For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

3.1 High Back Pressure

3.1.1 Finding the Source of High System Pressure

Column pressure, (after subtracting the system pressure) for the Dionex CarboPac PA200 Analytical Column should be close to the pressure listed in the QAR when using the test chromatogram conditions.` If a Dionex CarboPac guard and analytical column are both installed, column pressure will increase by approximately 20% over the pressure listed in the QAR for the column. If the total system pressure is much higher than expected, it is advisable to determine the cause of the high system pressure.

A. Make sure that the pump is set to the correct eluent flow rate. Higher than recommended eluent flow rates will cause higher pressure. If necessary, measure the pump flow rate by collecting the DI H_2O eluent for a specified time at operating pressure, and measure the collected volume using an analytical balance. This data (weight/time) will give actual flow rate.
B. Determine which part of the system is causing the high pressure. High pressure could be due to plugged or constricted tubing, an injection valve with a clogged port, a column bed support clogged with particulates, or a clogged detector cell.

   To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the Dionex EGC 400 MSA cartridge for 1-mm system (the Dionex EGC (Cap) MSA cartridge for 0.4-mm system), and turn the pump on. Watch the pressure; it should not exceed 200 psi. (unless a backpressure coil has been installed between the pump outlet and the injection valve in which case, first disconnect the eluent line from the pump to the backpressure coil). The total pressure with the Dionex EGC 400 MSA cartridge, Dionex EGC 400 KOH cartridge and Dionex RFIC$^+$ Dual EG Eluent Degasser for 1-mm system (the Dionex EGC (Cap) MSA cartridge, Dionex RFIC capillary EGC MSA degasser, Dionex EGC-KOH (Cap) cartridge, and Dionex RFIC EGC capillary eluent degasser for 0.4-mm system) connected should be <1000 psi. Continue adding system components (backpressure coil (if present), injection valve, column(s), and detector) one by one, while monitoring the system pressure. The pressure should increase by the sum of the measured pressures of the individual guard and analytical columns (see product QAR) when the CarboPac Guard and Analytical columns are connected.

C. A Dionex High-Pressure In-Line Filter positioned between the Pump and Eluent Generator (or injection valve if and EGC is not installed) should be installed to prevent particulates from blocking the system

3.1.2 Replacing Column Bed Support Assemblies for 1 mm Column

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

A. Disconnect the column from the system.
B. Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.
C. Remove the bed support. Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you do not scratch the walls of the end fitting. Discard the old bed support assembly.
D. Place a new bed support assembly (provided with each 1-mm column) into the end fitting. Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Drop the bed support assembly into the end fitting, making sure that the bed support assembly is centered at the bottom of the end fitting. Wrap the end fitting gently on a hard surface to reorient the bed support assembly as necessary in order to properly situate the bed support assembly in the end fitting.

CAUTION

If the column tube end is not clean when inserted into the end fitting, particulate matter may obstruct a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.

E. While holding the column in an inverted configuration, tighten the end fitting back onto the column. Tighten it finger-tight, then an additional 1/4 turn (3.5 lbf-in). Tighten further only if leaks are observed. It is important you do not overtighten the end fitting or you will strip the threads on the column!
F. Reconnect the column to the system and resume operation.

3.1.3 Filter Samples

Samples containing particulate material may clog the column inlet bed support. Filter samples through a 0.45 µm filter prior to injection.

3.2 High Background

While it may be possible to obtain reasonable performance even with elevated levels of detection background according to some requirements, high background frequently brings about an increased size of gradient artifacts and can be accompanied by a presence of ghost peaks. Detection sensitivity may also change suddenly when the detection background is too high. A background >50 nC with 8 mM KMSA/150 mM KOH at 0.0.063 mL/min for the 1-mm system and 0.010 mL/min for 0.4-mm system and 30°C using the quadruple waveform indicates one of the following possibilities:

A. Incorrect detection parameters.
Verify that Ag/AgCl is specified as a reference electrode. Check all values of waveform in program against those in the Disposable Electrode Manual.
B. Compromised working electrode surface.
Briefly install a new working electrode and check the background as above. If the

reading remains > 50 nC, remove the new electrode within 30 minutes and continue testing for column or system contamination. Otherwise continue with your work with the new electrode installed.

C. Insufficient degassing vacuum.
Verify that the vacuum applied to both the Dionex RFIC$^+$ Dual EG Eluent Degasser (Item # 22181-60951) are above 27-inch Hg for 1-mm system (the Dionex RFIC Capillary EGC MSA Degasser (Item # 22181-60211) and the Dionex RFIC Capillary Eluent Degasser (Item # 22181-60202) for 0.4-mm system).
In the case that the vacuum doesn't work properly, after the vacuum is restored, install the restriction tubing in place of the column. With EGC power OFF, turn on the pump to wash the system of the residual gas for 2 h. After that, install the column and turn on the pump with EGC power OFF, keep the pump running for 15 min before turning EGC power back on.

D. Column contamination: Remove the column set from the system first and replace it with a length of 0.001" i.d. PEEK tubing, generating a pressure drop over 2000 psi. If the background reading improves after the column is removed from the system, go to Appendix A, "CarboPac PA200 Column Care" in Product Manual of CarboPac PA200 Columns for Dual EGC ICS-6000 (Document No. 065734).

E. Water contamination: Prepare eluents using a fresh ultra-pure water from another source. If the background is reduced, investigate the source of contamination in the original source of water.

F. System Contamination: If the background remains high even with fresh water and without the column, carry out the 2 M sodium hydroxide rinse. In a properly working system, the electrochemical detection (ED) background for the Dionex CarboPac PA200 QAR eluent is 25-40nC. If the background is much higher, determine the cause of high background.

G. Minor leak at the ED cell. Due to the low flow rate, the minor leak at the ED cell might not set off the leak alarm. The minor leak can cause increased background and noise. Verify that there is no leak at the ED cell. If leaking around the 1-mil gasket is observed, remove the gasket, carefully dry with lint free cloth the wet ED surface and replace with new 1-mil gasket.

3.2.1 Preparation of Eluents

A. The Dionex CarboPac PA200 1 mm and 0.4 mm columns are not compatible with manually prepared eluents. The use of a Dionex EGC 400 MSA cartridge and a Dionex EGC 400 KOH cartridge is required.

B. Make sure that the deionized water used has a specific resistance of 18.2 MΩ-cm or greater.

3.2.2 A Contaminated Guard or Analytical Column

A. Remove the columns from the system.

B. Install a back-pressure coil that generates approximately 2000 psi and continue to pump eluent. If the background decreases, the column(s) is (are) the cause of the high background.

C. To eliminate downtime, clean or replace the analytical column at the first sign of column performance degradation. Clean the column as instructed in, "Appendix A, Dionex CarboPac PA200 Column Care" in Product Manual of CarboPac PA200 Columns for Dual EGC ICS-6000 (Document No. 065734).

3.3 Poor Resolution

One of the unique features of Dionex CarboPac columns is the fast equilibration time in gradient applications from the last eluent (high ionic strength) to the first eluent (low ionic strength). The actual equilibration time depends on the ratio of the strongest eluent concentration to the weakest

eluent concentration and application flow rate. Typically, equilibration times range from 15 to 20 minutes for both 1-mm and 0.4-mm columns.

If increased separation is needed for early eluting peaks, reduce the initial eluent concentration.

Due to different system configurations, the gradient profile may not match the gradient shown in example applications in the product column manual. Gradient conditions can be adjusted to improve resolution or to adjust retention times either by changing the gradient timing or by changing the initial and/or final eluent concentration.

A. Keep the eluent concentrations constant and adjust the gradient time. This is the simplest way to compensate for total system differences if resolution is the problem.
B. Change the initial and/or final eluent concentration and adjust the gradient time. This approach requires more time to develop and more knowledge in methods development work. Its advantage is that it allows a method to be tailored for a particular application, where selectivity, resolution, and total run time are optimized. Be aware poor peak resolution can be due to any or all of the following factors.

3.3.1 Loss of Column Efficiency

A. Check to see if headspace has developed in the guard or analytical column. This is usually due to improper use of the column such as exposing it to high pressures. Remove the column's inlet end fitting (see Section 5.1.2, "Replacing Column Bed Support Assemblies" in Product Manual of CarboPac PA200 Columns for Dual EGC ICS-6000 (Document No. 065734)). If the resin does not fill the column body all the way to the top, the column must be replaced.
B. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.0025" for both 1 mm and 0.4 mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks. Only use precut tubing of the same type.
C. If tubing is not connected properly from the inlet and outlet of the column, it can cause low efficiency. When installing Dionex CarboPac columns, it is recommended to turn off the pump while connecting the column inlet and the column outlet to the detector. This will avoid any slippage of the ferrule under high pressure conditions which can cause low peak efficiencies.

3.3.2 Shortened Retention Times

NOTE

Even with adequate system and column efficiency, resolution of peaks will be compromised if analytes elute too fast.

A. Check the flow rate. See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using an analytical balance.
B. Check to see if the eluent compositions and concentrations are correct. An eluent that is too concentrated will cause the peaks to elute faster.
C. Column contamination can lead to a loss of column capacity. Highly retained contaminant ions will occupy a portion of the anion exchange sites limiting the number of sites available for retention of analyte ions. Refer to "Appendix A, Dionex CarboPac PA200 Column Care" in Product Manual of CarboPac PA200 Columns for Dual EGC ICS-6000 (Document No. 065734), for recommended column cleanup procedures.

NOTE

Possible sources of column contamination are impurities in injected samples and in the deionized water used for eluents or components of the sample matrix. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

D. After cleaning the column, follow the system re-start procedure BEFORE reinstalling the column in the system and let it equilibrate with eluent for about 30 minutes directing the column effluent to waste. Then connect the column to the electrochemical detector cell. The column is equilibrated when consecutive injections of the standard result in reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should have been eluted from the column.

NOTE

For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

3.3.3 Loss of Resolution for Early Eluting Peaks

If poor resolution or efficiency is observed for early eluting peaks compared to the later eluting peaks, check the following:

A. Improper eluent concentration may be the problem if retention time is less than expected. Check the flow rate of the pump, as pump flow rate will affect the eluent concentration in an RFIC-EG system. Ensure the Eluent Generator is set to the correct eluent concentration.
B. Column overloading may be the problem. Reduce the amount of sample ions being injected onto the analytical/capillary column by either diluting the sample or injecting a smaller volume onto the column.
C. Sluggish operation of the injection valve may be the problem due to partially plugged port faces. Refer to the valve manual for instructions.
D. Improperly swept out volumes anywhere in the system prior to the guard and analytical/capillary columns may be the problem. Swap components, one at a time, in the system prior to the analytical/capillary column and test for early eluting peak resolution after every system change.

3.3.4 Spurious Peaks

A. The columns may be contaminated. If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system, the retention times for the analytes will then decrease and spurious, inefficient (broad) peaks may show up at unexpected times. Clean the column as indicated in “Column Care” in Product Manual of CarboPac PA200 Columns for Dual EGC ICS-6000 (Document No. 065734).

NOTE

For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

B. The injection valve may need maintenance. When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. This will occur when the injection valve needs to be cleaned or re-torqued (see injection valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the

chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

3.3.5 No Peaks, Poor Peak Area Reproducibility or Too Small Peak Areas

A. Check the position and filling levels of sample vials in the auto sampler.
B. Check the transfer line volume.
C. Check injector needle-height setting.
D. Check if there is any bubble trapped in the sample syringe of the auto sampler.
E. Check each line of the schedule for proper injector parameters. Revert to full loop and column appropriate sample loop size.
F. Service the injection valve (check for leaks, Tefzel fragments, or sediments inside the valve).

3.3.6 Large Baseline Dip in the Chromatogram

A large baseline dip appearing later in the chromatogram is usually caused by oxygen in the sample injected. The 'oxygen dip' is normal and can be reduced in magnitude with higher KOH concentration in the eluent.

3.3.7 Unidentified Peaks Appear with Expected Analyte Peaks

During a potassium methanesulfonate/ potassium hydroxide gradient, a number of small peaks may appear. These peaks are usually due to trace contaminants in the water supply. The contaminants accumulate on the column during the isocratic section of the chromatogram and are released, frequently as irregular baseline deformations or sharp spikes, with the increasing eluent strength.

If extraneous peaks are observed even after the water supply is excluded as a possible cause, clean the auto sampler lines and sample loop. The auto sampler should be cleaned using the following protocol:

A. Disconnect the column and detector cell from the auto sampler.
B. Set the pump to 100% deionized water.
C. Place the following solutions in the auto sampler and inject in sequence. Use 0.4 µL and 0.1 µL full loop injections for 1 mm and 0.4 mm systems respectively:
    1. 1 M NaOH
    2. Deionized water
    3. IPA
    4. Deionized water
    5. 1 M HCl
    6. Deionized water

3.4 Decreased Detection Sensitivity

Always confirm the loss of response by performing at least one injection of the system suitability standard mix as described in Section 4.1 in Product Manual of CarboPac PA200 Columns for Dual EGC ICS-6000 (Document No. 065734). This is to make sure that a decreased level of response is not being caused by system problems.
Any decrease in detection sensitivity means that the working electrode surface has been affected. The operator should install a new working electrode. Spare gold working electrodes should always be available in order to avoid unnecessary delays.

Exceptions:

Check the pH reading. If the value is out of range or >13.2, install a new reference electrode and then install a new gold working electrode. The system cleanup is not necessary. The decrease in

sensitivity was caused by a gold-oxide-buildup on the electrode surface because the reference potential was too high.

Peak heights will increase with increasing eluent concentrations. This is due to improvement of the kinetics in the electrode detection related to ionic strength and pH effects. If you run the same standard at 1 mM and at 12 mM, peak heights will increase at 12mM. Hence you will see a decrease in peak heights whenever going to lower eluent concentrations as well.
After installing a new working electrode (with or without the complete system cleanup), confirm the normal detection sensitivity. Carry out a test with a reference standard. Should the response be too low, immediately remove the new working electrode from the system.

3.5 Excessive Gradient Rise

The magnitude of the gradient rise can be minimized by running high eluent strengths during the times when the system is not in use for sample or standard analysis. This will keep the column conditioned, free from carbonate buildup, and ready for analysis.

A. Make sure the status of vacuum is OK (following the instruction in Section 1.2 for installation of the vacuum kit).
B. Make sure the gradient rise is not caused by the system and/or detector cell.
C. Set column temperature to 40 °C and wash the guard and column with 100 mM KMSA/100 mM KOH for at least four hours (preferably overnight). Run a blank gradient at 30 °C and if necessary repeat the cleanup with 100 mM KMSA/100 mM KOH wash at 40 °C.

3.6 Reconditioning or Replacement of the Gold (disposable) Electrodes or Replacement of the Reference Electrode

Refer to Product Manual for Disposable Electrodes Doc. No. 065040, Dionex ICS-5000 Ion Chromatography System Manual Doc. No. 065342 and User's Compendium for Electrochemical Detection Doc. No. 065340 for any help necessary with electrochemical detection, working and reference electrodes.