Ion chromatography

Combustion ion chromatography with a Dionex Integrion HPIC system using Chromeleon 7 CDS software

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Goal
To update Technical Note 72211 with installation and configuration instructions for a combustion ion chromatography (CIC) system consisting of a Nittoseiko Automated Combustion Furnace (AQF) model AQF-2100H interfaced with a Thermo Scientific™ Dionex™ Integrion™ High-Performance Ion Chromatography (HPIC) system, controlled by Thermo Scientific™ Chromeleon™ 7 Chromatography Data System (CDS) software

Introduction
Ion chromatography is the method of choice for determining ionic components, such as fluoride, chloride, and sulfate. However, not all samples—for example, solids, intractable solids, powders, semisolids, non-aqueous solutions, and gases including liquefied petroleum gas (LPG)—are suitable in their original state for conventional IC analysis. Sample preparation is needed to extract the analytes or remove the interfering matrix, which is often costly and time-consuming. In automated (pyrohydrolytic) combustion IC, samples are pyrolyzed in an oxidizing atmosphere. The resultant vapors are absorbed in an aqueous solution and then introduced directly into the IC system for analysis. All halogen- and organosulfur-containing compounds from the sample are converted to total halogens and total sulfur and measured as their corresponding halides and sulfate.

This automated method is highly sensitive, easy to use, and less prone to operator variations. It saves time and produces less hazardous waste than other sample preparation procedures, such as acid digestion or extraction with an organic solvent. To operate a combustion IC system with the Nittoseiko Analytech (Nittoseiko Analytech Co., formerly known as Mitsubishi Chemical Analytech) combustion system previously required both the Mitsubishi Analytech software and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software (Technical Note TN72211[1]). This Technical Note updates Technical Note 72211 with a single controlling software, Chromeleon CDS version 7.3 or above. A new driver was developed to control the Nittoseiko AQF-2100H combustion system through the AQF-2100 ePanel, Chromeleon Custom Variables, instrument method, and sequence programs.

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Equipment

The combustion IC system described in this note includes the following:

- Dionex Integrion HPIC system (P/N 22153-60305) (Firmware 1.7.0 and later; Dionex Integrion Pump Wellness virtual module: Firmware 6.2.0 and later), including:

  - Eluent generation capability

  - Thermo Scientific™ Dionex™ Integrion™ IC Conductivity Detector, P/N 22153-62034

  - Column oven temperature control

  - Thermostatted detector compartment

  - Tablet control (optional)

  - Consumables device monitoring capability (optional)

- Thermo Scientific™ Dionex™ AXP Auxiliary Pump (optional, used for pre-concentration)

- Nittoseiko Automatic Combustion Unit Model AQF-2100H (SYSCON version 3.15 or higher), comprising:

  - Electric furnace, HF-210 (Firmware 1.09 or higher, P/N NS-FN1HAG)

  - Gas absorption unit, GA-211 (Firmware 1.07 or higher, P/N NS-AQ3GA2)

  - External solution selector, ES-211 (optional, used for external calibrations, P/N NS-AQ3ES2)

  - Sample changer options

    - Automatic boat controller, ABC-210 (required for most applications) (P/N NS-BL1ABL)

    - Liquid autosampler, ASC-250L (Firmware 1.31 or higher, optional, P/N NS-SC1L50)

    - Liquid and solid autosampler, ASC-270LS (Firmware 1.03 or higher, optional, described in this technical note, P/N NS-SC7LS)

    - Gas injector module, GI-260 for gas and liquefied petroleum gas (LPG) samples (optional, P/N NS-GI2LL)

      - Combustion monitor (Firmware 1.0 or higher, optional, P/N NS-AQ3CM)

- Computer, including:

  - At least five USB ports for (3) computer peripherals, (1) Dionex Integrion HPIC system, and (1) Nittoseiko AQF-2100H system

  - USB or RS232 cables for each AQF-2100 module and the Dionex Integrion HPIC system. One additional USB or RS232 cable with USB converter is needed to connect the combustion system to the computer.

Software

Chromeleon CDS version 7.3 with CIC driver (available free of charge with the purchase of CIC system)

Reagents

- Degassed ASTM Type I deionized (DI) water, >18 MΩ·cm resistivity

- Ultra-high purity (UHP) grade argon gas

- UHP grade oxygen gas

- 5.0 mg/L sodium phosphate (in DI water) for determining the constant volume parameter

- High-purity hydrogen peroxide, 30%, stabilized, Suprapur™ (EMD Millipore), (Fisher Scientific P/N M1072980250), used to prepare absorption solution for total bromine, total iodine, and total sulfur (application-specific)

Process overview

Samples are introduced into the AQF-2100H by one of the auto sample changer options. The samples are oxidized by O_2 at high temperature, the vapors are sparged through the absorbing solution in the Nittoseiko GA-211 module, and the solution is transferred to the IC system for analysis. Pyrolysis (combustion) occurs between 800 and 1,100 °C.

Using phosphate and hydrogen peroxide

Phosphate is not detected in a combusted sample and therefore is suitable as an internal standard in the absorbing solution to calibrate the analytical results. Hydrogen peroxide is added in the absorbing solution to oxidize hydrogen sulfite resulting from the incomplete oxidation of sulfur, which will be quantified as sulfate in aqueous solution.

Setting up and installing the Dionex Integrion HPIC instrument

The Dionex Integrion HPIC system, RFIC model, is configured with a conductivity detector, eluent generation, consumables monitoring, and temperature control of the column oven and detector compartment. This system can operate at pressures up to 5,000 psi with eluent generation. To set up this application:

1. Plumb the Dionex Integrion HPIC system based on the intended application (refer to Figure 9, 10, or 11).

2. Connect the USB cable from the Dionex Integrion HPIC system to the computer.

3. Connect the RS232 or USB cables from the Nittoseiko GA-211, ES-211, and sample changer modules to the Nittoseiko HF-210 module.

4. Connect the USB cable or RS232 with USB converter cable from the Nittoseiko HF-210 module to the computer.

5. Connect the power cables and turn on all the modules.

Electronically configuring the CIC modules in the Chromeleon CDS software

To configure the IC system:

1. Start the Chromeleon Instrument Controller program.

2. Select *Configure Instruments* to open the Chromeleon Instrument Configuration Manager.

3. Right-click on the local computer name.

4. Select *Add an Instrument.*

5. Enter an appropriate name (for example: IntegrionCIC).

6. Add the following modules to this instrument configuration: Integrion HPIC system, Integrion HPIC Pump (Wellness), and Nittoseiko AQF-2100H, as detailed below.

Integrion HPIC system module

1. Select *Add a Module, IC: Dionex Integrated Modules, and Integrion HPIC System* (Figure 1). A window with multiple tabs will open automatically (Figure 2).
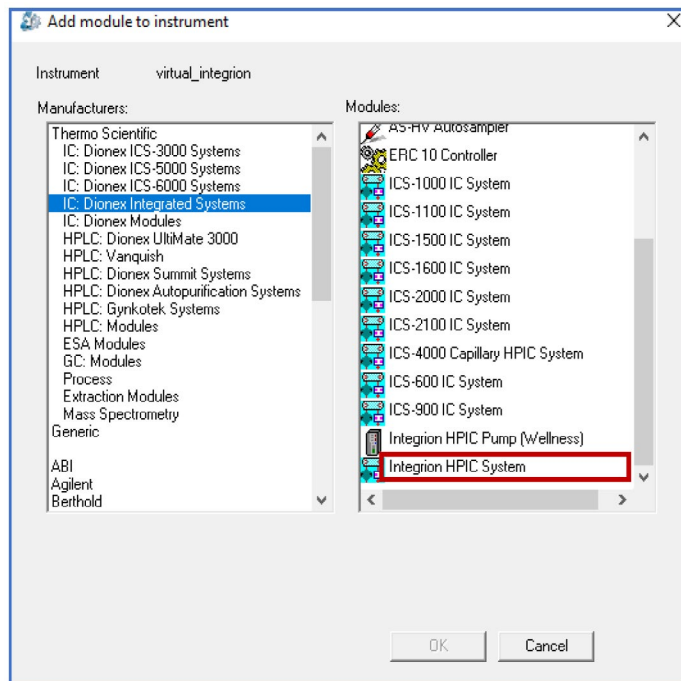

Figure 1. Adding Integrion HPIC system module to configuration

2. Select the Model Serial No. on the General tab. The Chromeleon CDS software will automatically detect all electrolytic devices, detectors (Figure 2), pump degasser, and seal wash pump (not shown). Chromeleon 7 CDS manages and controls the Nittoseiko AQF-2100H system and the Dionex Integrion HPIC system. TTL cables are no longer needed but remain an option.
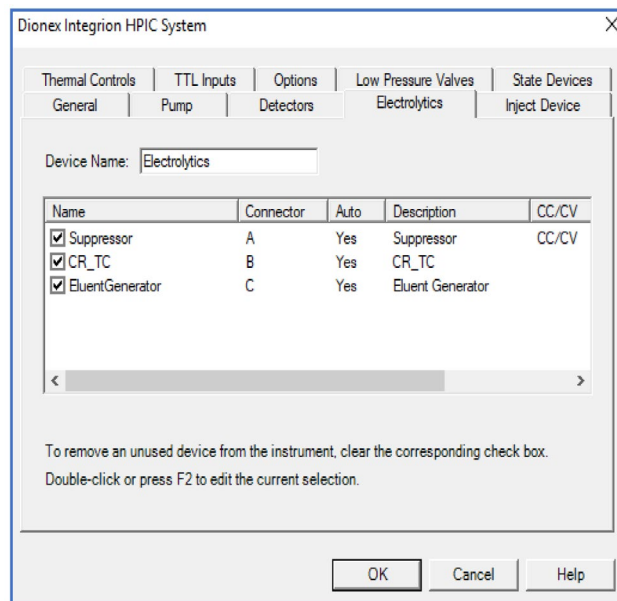

Figure 2. Automatic detection of electrolytic devices

3. Use the Inject Device tab to assign control of the Integrion injection valve.

Mode	Assign control of Integrion injection valve
Direct injection using the GA-211 injection valve	Assignment is not necessary
Direct injection using the Integrion injection valve	Assign to Integrion control
Preconcentration	Assign to Integrion control

Integrion HPIC pump (Wellness) module

To add pressure monitoring capabilities, it is necessary to add another module. To do this:

1. Right-click and select *Add a Module, IC: Dionex Integrated Modules, Integrion HPIC Pump (Wellness) module*

2. Select the USB address to link the module to the configuration.

3. Select the Devices tab and click on the Pressure Signal(s) box (Figure 3).
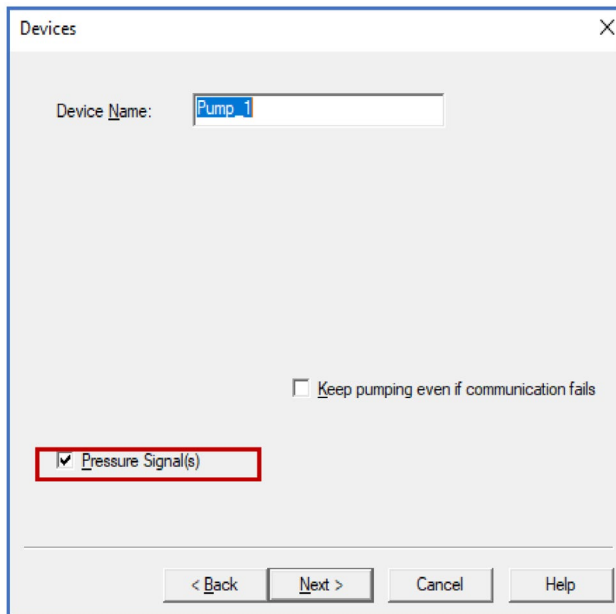
Figure 3. Adding Integrion HPIC pump wellness module and activating pressure signal monitoring

Nittoseiko AQF-2100H module

The ASC-270LS sample changer is used in this technical note. To add the combustion system modules:

1. Right-click and select *Add a Module, Nittoseiko Analytech, and the AQF-2100* icon (Figure 4).
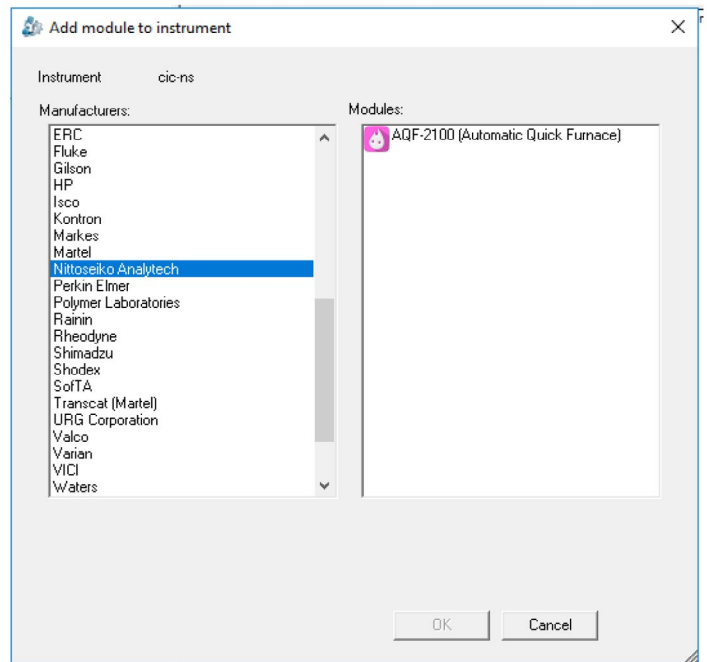

Figure 4. Adding Nittoseiko Analytech AQF-2100H module

2. Select the General tab. Click on *Search* to find the COM channels and select the COM for the combustion system (often COM4) to link the module to the configuration (Figure 5).
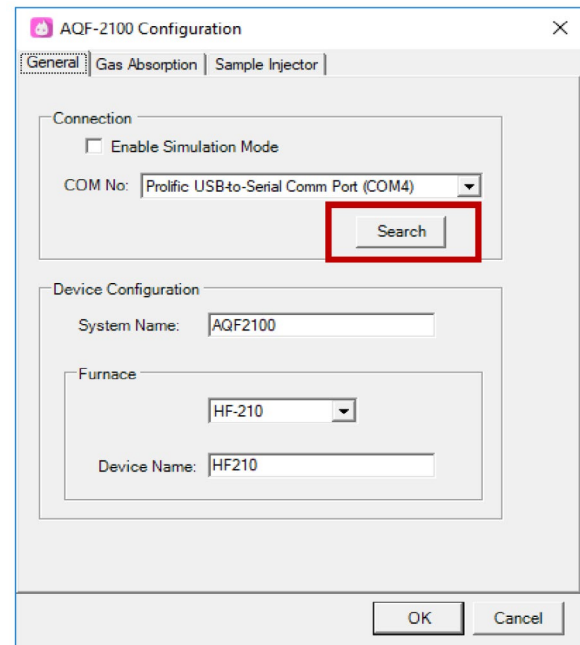

Figure 5. General tab. Activating the connection by selecting Search and selecting COM channel on the drop-down list

3. In the lower part of the General tab, select the furnace module (Figure 6).
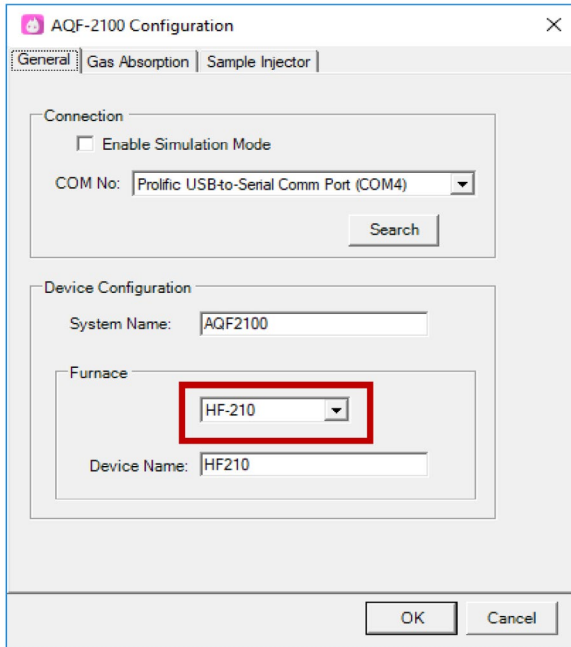

Figure 6. General tab. Select the furnace type.

4. Select the Gas Absorption tab. Select the absorption tube volume (Figure 7).
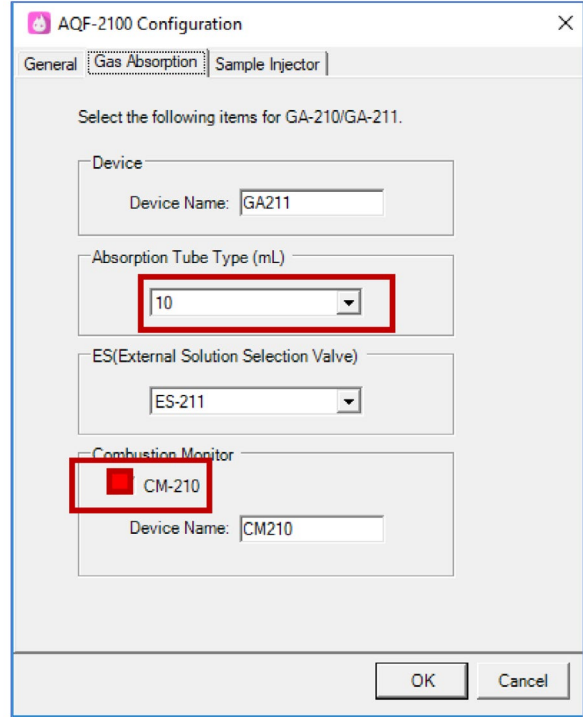

Figure 7. Gas Absorption tab. Select the Absorption Tube Type and check the CM-210 box.

5. Select the Sample Injector tab. Select Sample Injector, Tray Type, and Syringe Size (Figure 8).
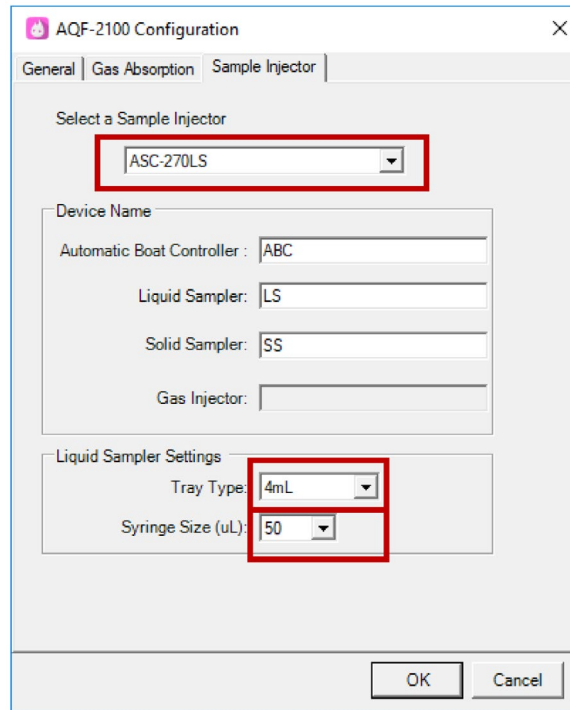

Figure 8. Sample Injector tab. Select the Sample Injector, Tray Type, and Syringe Size.

Schematics

Direct injection using the injection valve in the Nittoseiko GA-211 module

This is the recommended configuration. In this configuration, the sample loop is installed on the Nittoseiko GA-211 injection valve. When the sample is ready for IC analysis, the Nittoseiko GA-211 pump loads the sample loop from the absorption tube. The schematic is shown in Figure 9. This setup is recommended for IC applications with pressures up to 5,000 psi.

Direct injection using the injection valve in the Dionex Integrion system

In this configuration, the sample loop is installed on the Dionex Integrion injection valve, instead of using the injection valve in the Nittoseiko GA-211 module (Figure 9). Plumb the system as shown in Figure 10:

1. Nittoseiko AQF-2100H system injection valve on GA-211 unit

   – Disconnect the sample transfer line from Port 2, connect 91 cm of 0.010 in. (0.25 mm) i.d. PEEK tubing with a 10-32 to 10-32 union, and install this on the Dionex Integrion system injection valve Port S.

   – Disconnect the sample waste line from Port 3, connect 81 cm of 0.010 in. (0.25 mm) i.d. PEEK tubing with a 10-32 to 10-32 union, and install this on the Dionex Integrion system injection valve Port W.

2. Dionex Integrion system injection valve

   – Connect the tubing from the IC pump to Port P.

   – Connect the sample loop to Ports L.
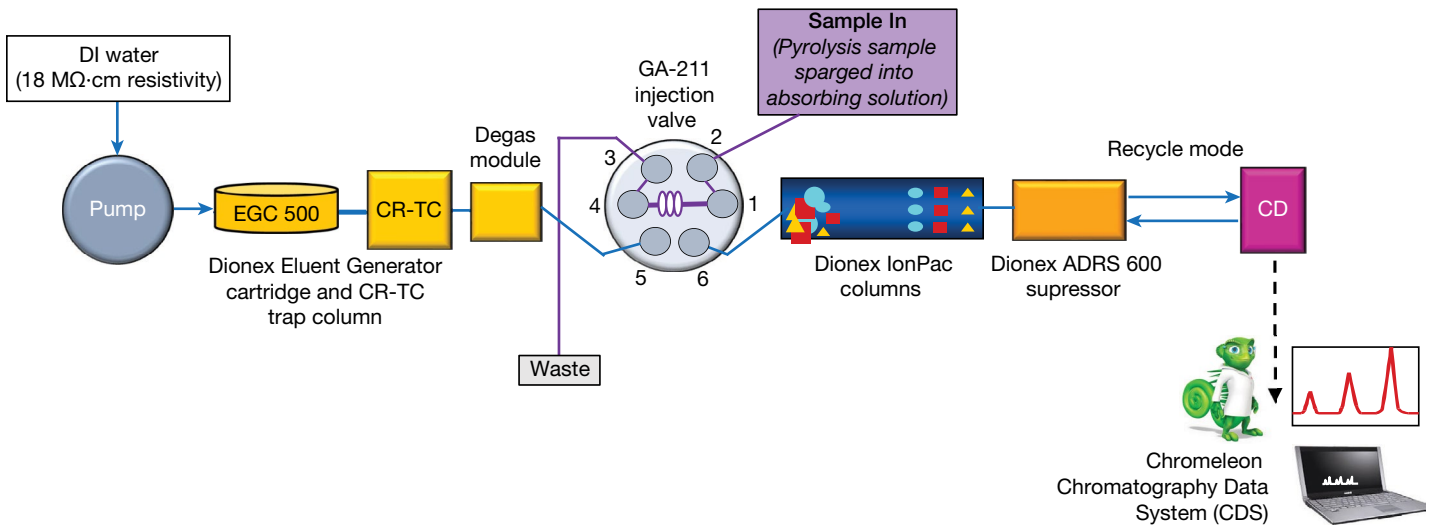
   – Connect the column to Port C.

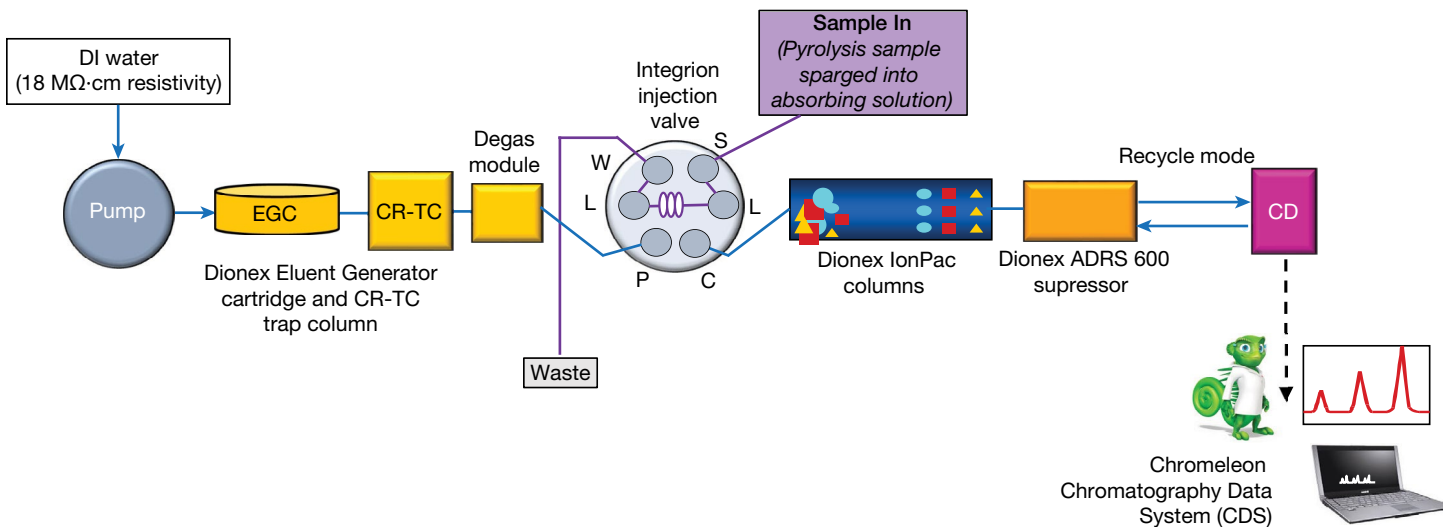Figure 9. Schematic for CIC using the GA-211 injection valve, in Load position

Figure 10. Schematic for CIC using the Integrion injection valve, Load position

Preconcentration

This setup is used when detection of analytes in the low mg/L concentrations is required. In this setup, the sample loop is installed on the Nittoseiko GA-211 injection valve, and a concentrator column is installed on the Dionex Integrion HPIC system injection valve. When the sample is ready for IC analysis, the Nittoseiko GA-211 module loads the sample loop. The GA-211 valve is rotated to the Inject position. The auxiliary pump transfers the sample to the concentrator column and flushes the concentrator column with DI water (3 times the concentrator column volume) before starting the IC analysis. The current recommended anion concentrator column when using the Dionex AXP auxiliary pump is the Thermo Scientific™ Dionex™ IonPac™ UTAC-ULP2 concentrator column (145 µL volume). The sample transfer time is < 0.5 min (3 × 0.145 min) at the Dionex AXP pump 1.0 mL/min. Preconcentrating the sample prior to IC injection is limited by the ionic content of the absorption solution. Plumb the system as shown in Figure 11:

1. Nittoseiko AQF-2100H system injection valve on GA-211 unit

   – Connect the sample loop to Ports 1 and 4.

   – Connect the tubing from the auxiliary pump to Port 6.

   – Connect Port 5 and Dionex Integrion system injection valve Port S with 0.010 in. (0.25 mm) i.d. PEEK tubing.

   – Connect the sample transfer line from the Nittoseiko GA-211 system to Port 2.

   – Connect the sample waste line from the Nittoseiko GA-211 system to Port 3 and route this tubing to a waste container.

2. Dionex Integrion system injection valve

   – Connect the tubing from the IC pump to Port P.

   – Connect the concentrator column to Port L.

   – Connect the column to Port C.

   – Connect the 0.030 in. (0.75 mm) i.d. PEEK tubing waste line to Port W and route this tubing to a waste container.

Tip: Add enough backpressure tubing just after the auxiliary pump and before the injection valve to produce backpressure of at least 1,000 psi at the desired flow rate
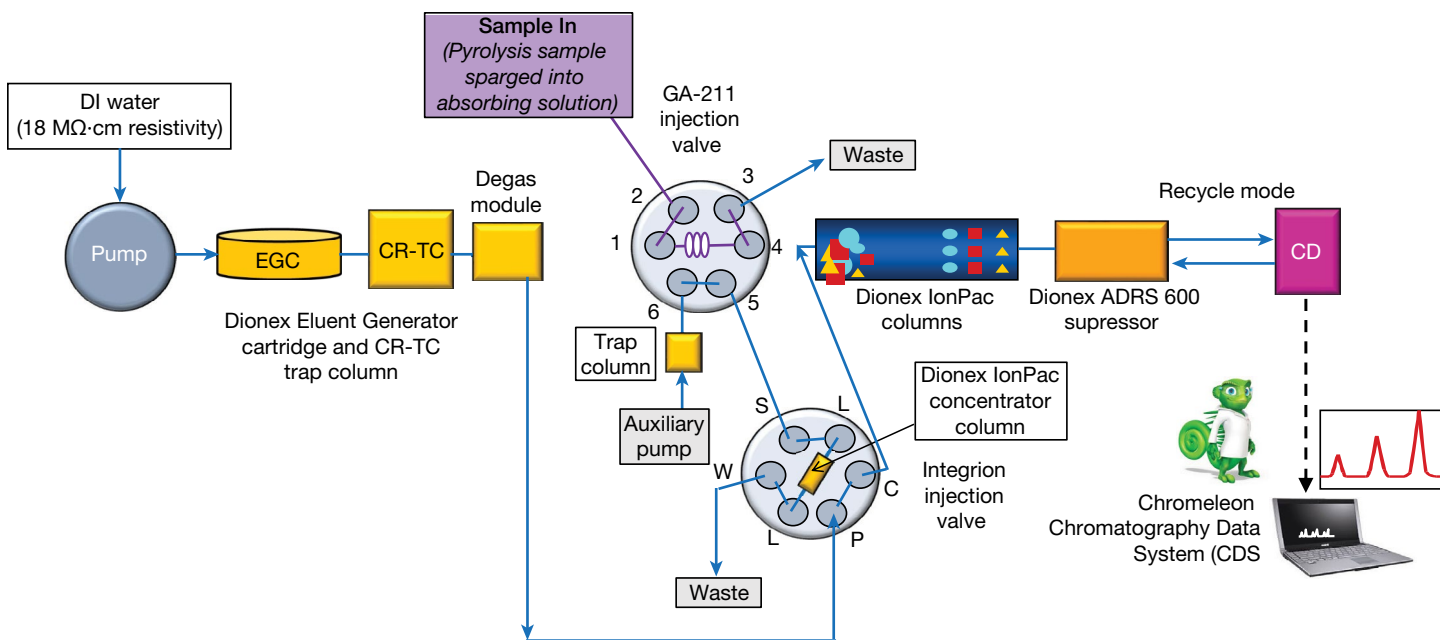

Figure 11. Schematic for CIC using preconcentration, loading sample loop

Programming the Chromeleon instrument program using script commands

Script commands are needed to coordinate the operations and timing of the GA-211 module, GA-211 injection valve, Dionex AXP auxiliary pump, and Dionex Integrion injection valve (Table 1).

Dionex IC PEEK Viper fittings

To achieve the best chromatography, it is important to gently tighten the Thermo Scientific™ Dionex™ IC PEEK Viper™ fittings and to use the fitting assemblies in the following locations.

- Thermo Scientific™ Dionex™ EGC 500 KOH eluent generator cartridge – *Eluent Out* to *Eluent In* on the Thermo Scientific™ Dionex™ CR-ATC 600 continuously regenerated anion trap column

- Dionex CR-ATC 600 continuously regenerated anion trap column – *Eluent Out* to *Eluent In* on the Thermo Scientific™ Dionex™ Degas module

- Between the valve – "Column" port to the guard column when installed on the Integrion HPIC system.

The recommended practice is to tighten the Dionex IC PEEK Viper fittings to finger tight. If leaking is observed, tighten an additional 1/16 to 1/8 turn clockwise.

Caution: Overtightening, or using a wrench to tighten the Dionex IC PEEK Viper fitting, may permanently damage the fitting. Important: Do not remove consumables tracking device tags from the columns or consumable devices. These tags are required for monitoring functionality.

Conditioning electrolytic devices and columns

Install the Dionex EGC 500 KOH eluent generator cartridge and Dionex CR-ATC 600 continuously regenerating anion trap column in the Dionex Integrion HPIC system.

1. Condition the devices according to instructions in the Chromeleon Instrument Panel drop-down menu (Consumables, Install). This information is also available in the product manuals and the Dionex Integrion HPIC System Operator's Manual.[2-6]

2. Condition the columns for 30 min according to the instructions from the Consumables drop-down menu. The general practice is to use the eluent and flow rate conditions listed in the column's QAR report while directing the eluent exiting the column to a waste container.

3. To hydrate the Dionex ADRS 600 dynamically regenerated suppressor, follow the Quick Start Instructions received with the suppressor.

   a) Wait 20 min for the suppressor to fully hydrate before installing the suppressor in the detector compartment.

   b) Install the backpressure loop from the slotted compartment next to the CD detector (exerting an additional ~40 psi) between the CD outlet and the suppressor *Regen In* port.

4. Complete the installation according to the flow diagram in Figure 9, 10, or 11.

Table 1. Script commands for preconcentration using the GA-211 and Integrion injection valves and the Dionex AXP auxiliary pump

Time	Device	Command	Value	Description
-1.1 Equilibration Time				
-1.1	GA-211 module	Wait	AQF2100.GA211.Ready	Wait for GA-211 module to dilute and mix sample. GA-211 pump loads sample into sample loop
-1.0	Integrion Injection Valve	Pump_ECD.InjectValve.LoadPosition		Integrion Injection valve places concentrator column in Load position
	AXP Pump	Pump.state	On	AXP Auxiliary pump is on
	AXP Pump	Pump.flow	1.0	AXP Auxiliary pump is transferring sample from loop to concentrator and washing 3x volume with DI water
	GA-211 Injection Valve	AQF2100.GA211.Inject		GA211 Injection Valve to Inject position. Injects sample to concentrator column
0.0 Inject Preparation				
	Integrion Injection Valve	Pump_ECD.InjectValve.InjectPosition		Integrion valve injects concentrated sample to IC
0.0 Inject				
	Integrion Injection Valve	Pump_ECD.InjectValve.InjectPosition	Duration =15	Integrion valve injects concentrated sample to IC
	AXP Pump	Pump.state	OFF	Auxiliary pump turns off.

Starting the Dionex Integrion HPIC system

To start the system:

1. Turn on the pump.

2. Immediately turn on the Dionex EGC 500 eluent generator cartridge.

3. Turn on the Dionex CR-ATC 600 anion trap column and Dionex DRS 600 anion suppressor when liquid flows through the devices. Note the system pressure. If the system pressure is below 2,000 psi, install additional yellow 0.003 in. (0.076 mm) i.d. PEEK backpressure tubing between the Dionex High-Pressure Degasser and the injection port (Pump port position).

4. Set the eluent concentration, column oven, compartment oven, and cell temperatures, and allow the system to equilibrate for 30 min.

Creating a Combustion IC instrument method and sequence with custom variables

1. Open Chromeleon CDS.

2. Select Data view in the Chromeleon Console.

3. Right-click on Chromeleon Local, select *Custom Variables Editor,* and select *Add* (Figure 12).
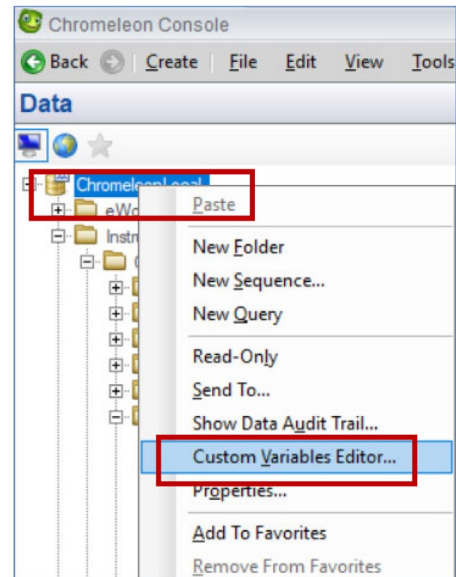
Figure 12. Accessing the custom variables editor

4. Create the custom variables using the data in Table 2. Select OK to close the window. Figure 13 shows the Custom Variable for Measurement_Type.

5. Create a new sequence using the Chromeleon Sequence Wizard. Advance through the tabs without entries and save the sequence.

Table 2. Creating custom variable tables

Name	Context	Type	Description	Items	Default
Pause	Injection	List	Pause	0:Disable 1:Enable	0:Disable
Injector	Injection	List	Injector	0:Manual_mode 1:Solid 2:Liquid 3:Liquefied_gas 4:Gas	0:Manual_mode
Measurement_Type	Injection	List	Measurement_Type	00:Abs_Inj 01:Ext_Inj 30:Boat_prebake 34:Heater_off 35:Gas_off 40:GA_EndProcessing 41:Sensor_Check 42:GA_AllWash	00:Abs_Inj
Constant_Volume	Injection	List	Constant_Volume	0:Disable 1:Enable	0:Disable

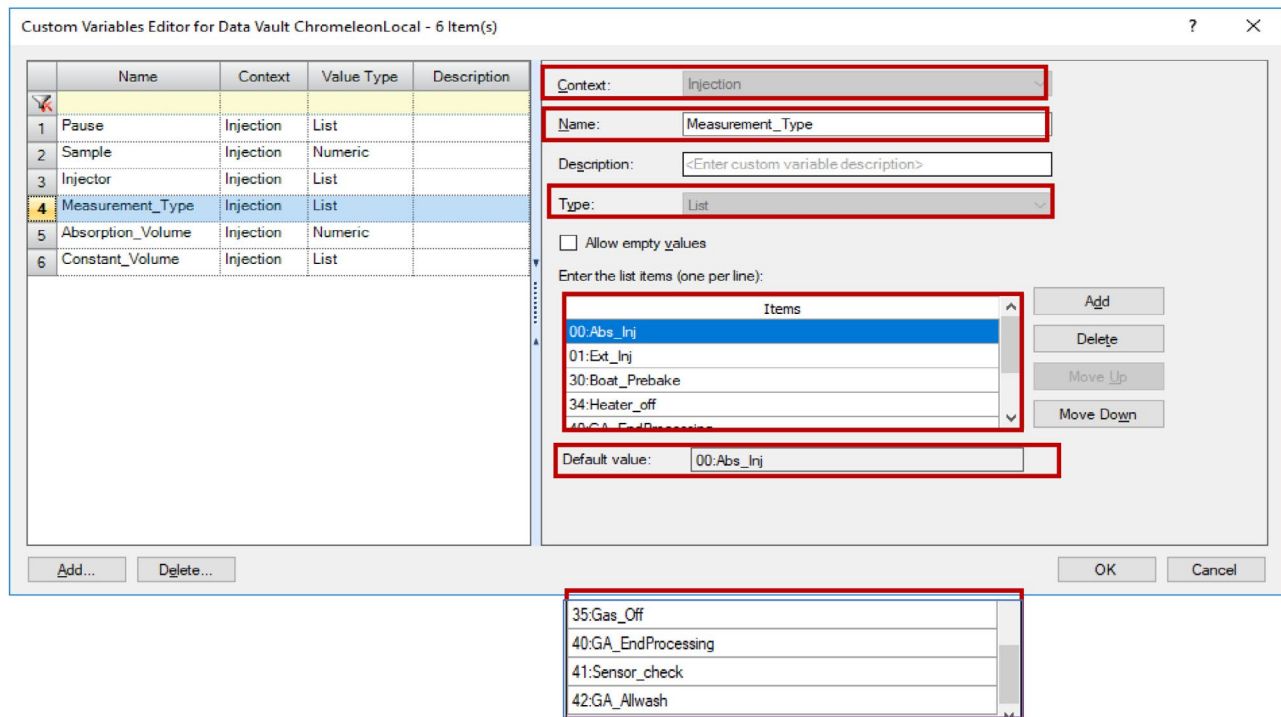| Name	Context	Type	Description	Default	Minimum	Maximum	Unit	Precision
Absorption_Volume	Injection	Numeric	Absorption_Volume	3	0	20	mL	0
Sample	Injection	Numeric	Sample	10	0	250	mg	1
IC_Time	Injection	Numeric	Enter IC run time in seconds +120 s	0	0	6,000	s	0

Figure 13. Creating custom variables: Measurement_Type

6. To import the custom variables into the sequence, select the f_x *Custom Columns* button (Figure 14) in the command lines. Select *Insert Custom Variables,* and *Import Custom Injection Variable from data vault* (Figure 14).

7. Highlight the custom variables table. Select *Finish.* (Figure 14) All custom variables should be visible. Verify the "Value Type" to check for errors.
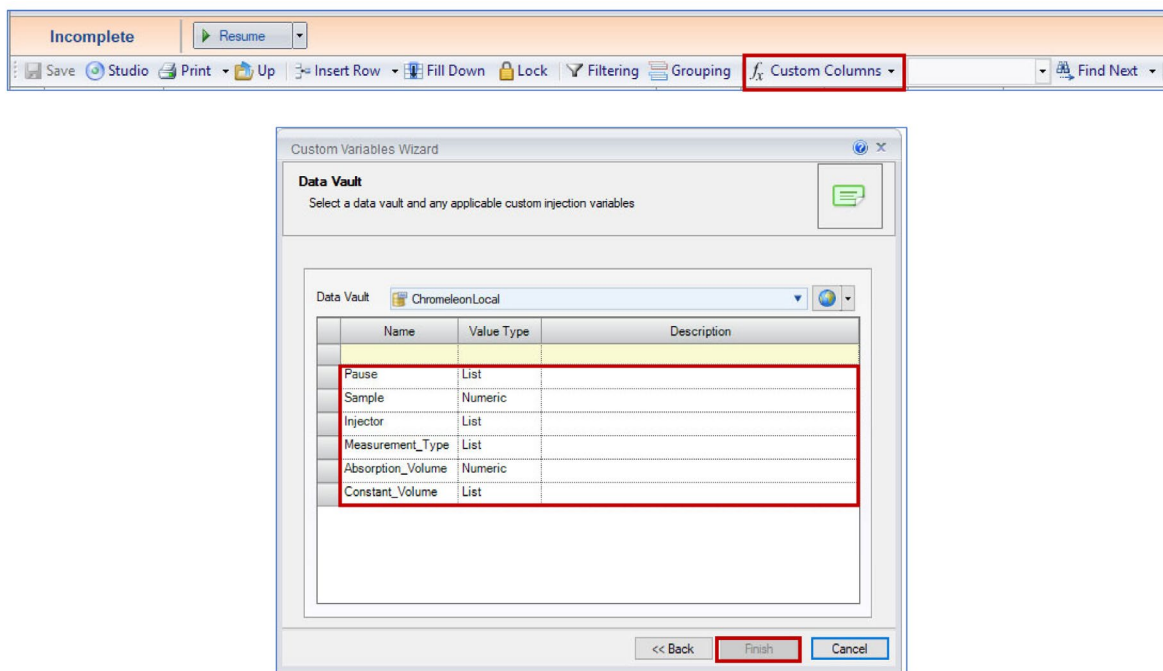
8. Arrange the new custom columns. Select a column, right-click, and select *Table Columns.* Using the Move Up or Move Down buttons, position columns into the desired positions (Figure 15).

Figure 14. Importing Custom Variables Editor
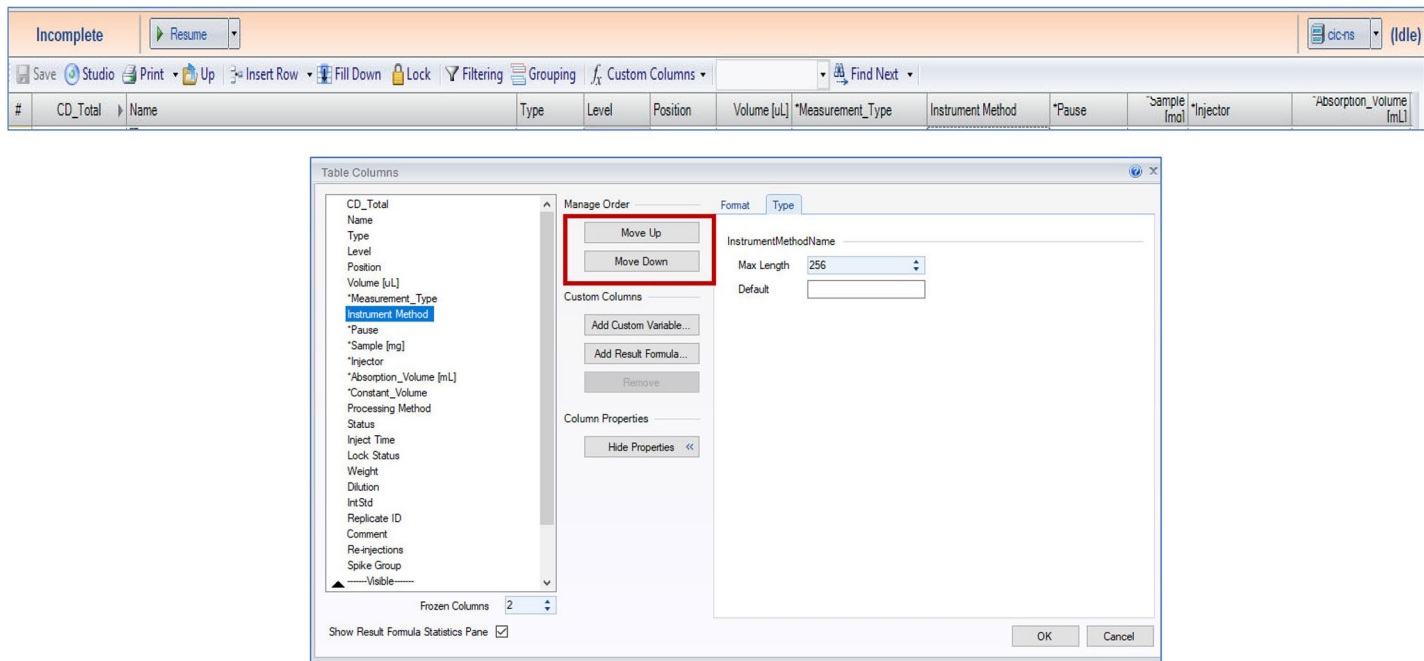
Figure 15. Organizing custom columns

9. Create an Instrument Method using the Chromeleon Method Wizard. Select all diagnostic channels (Figure 16).

10. Enter the Gas Absorption conditions: Water injection time: 12; Drain time: 17; Gas Line Washing Times: 3; Sample Absorption Time: 10 (Figure 17).
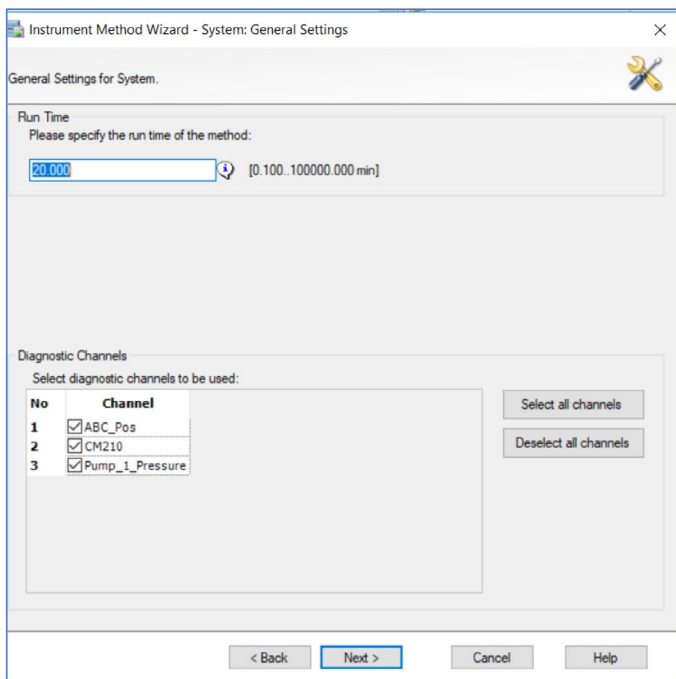
Figure 16. Creating the Instrument Method: Selecting all channels
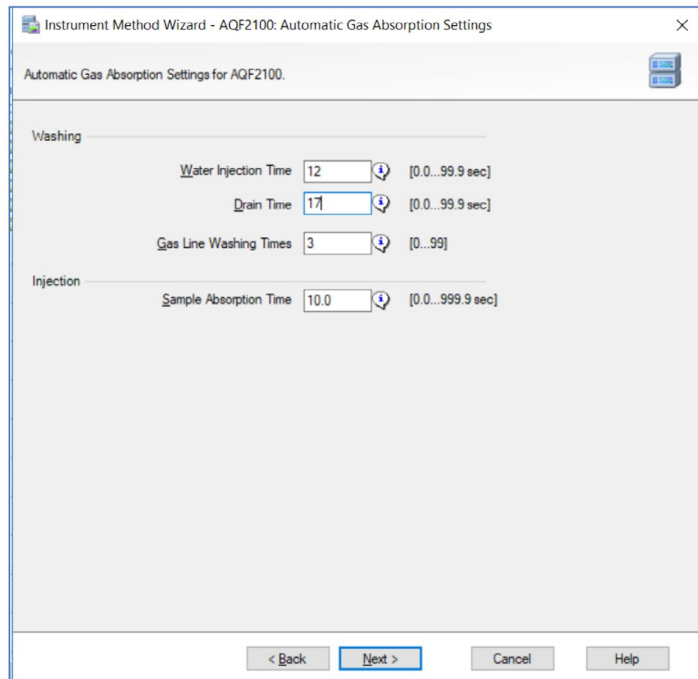
Figure 17. Creating the Instrument Method: Automatic gas absorption settings

11. Use default settings for Furnace Combustion Conditions (Figure 18).
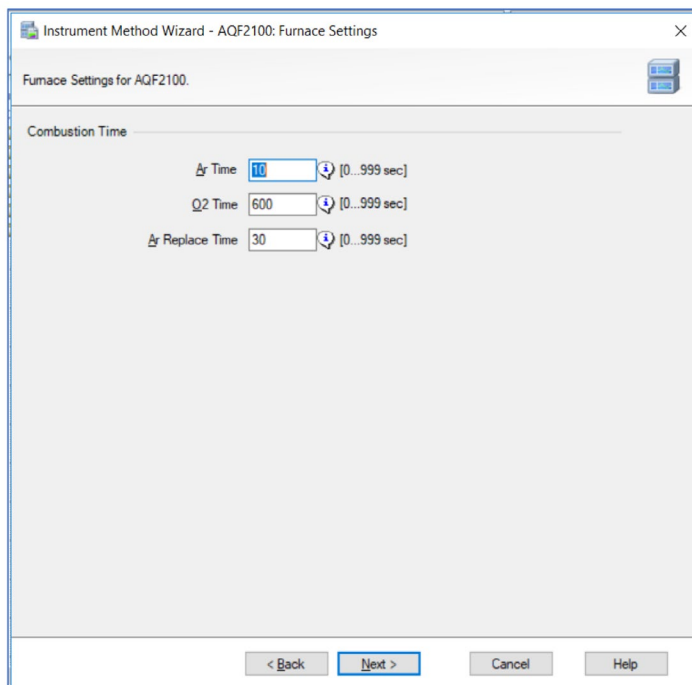
Figure 18. Creating the Instrument Method: Default furnace settings

12. For the Nittoseiko ASC-270LS Liquid and Solid Sample Changer module, use the default settings for solids and liquids (Figures 19 and 20).
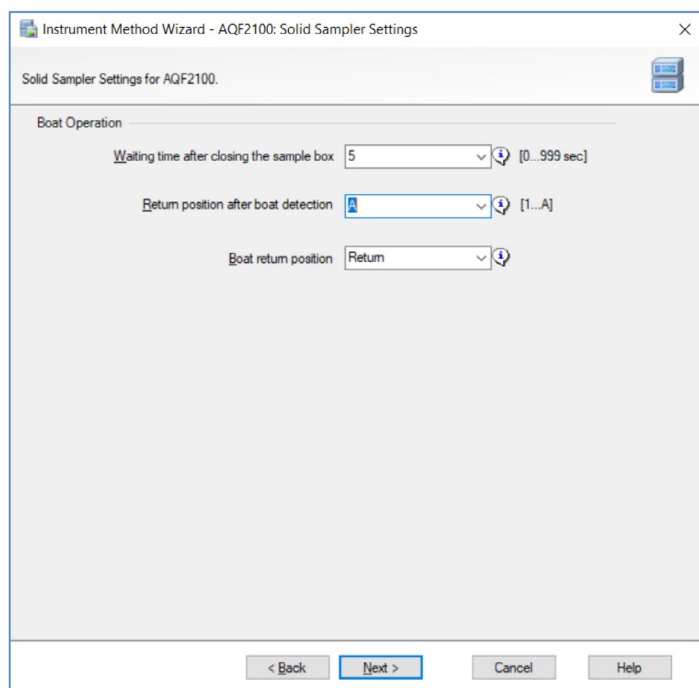
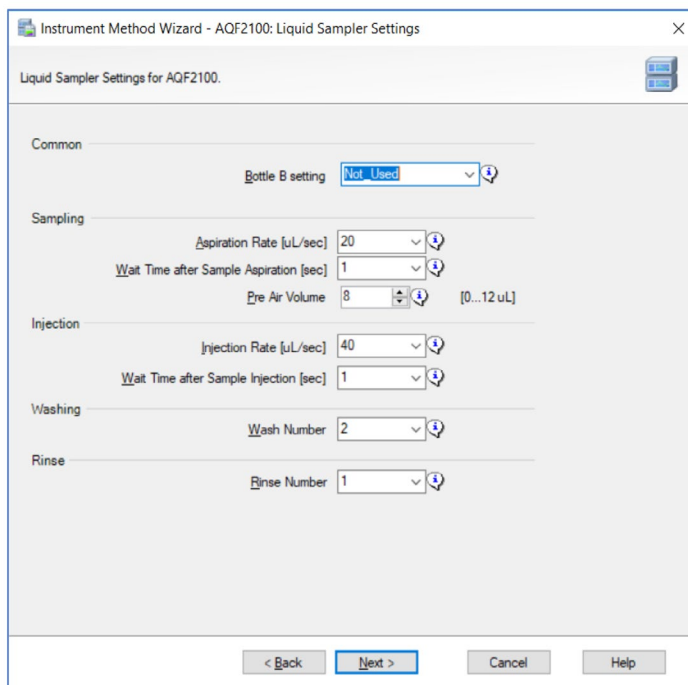Figure 19. Creating the Instrument Method: Default ASC-270LS Solid Sampler settings

Figure 20. Creating the Instrument Method: Default ASC-270LS Liquid Sampler settings

13. Enter Automatic Boat Controller settings from the Conditions section (Figure 21).
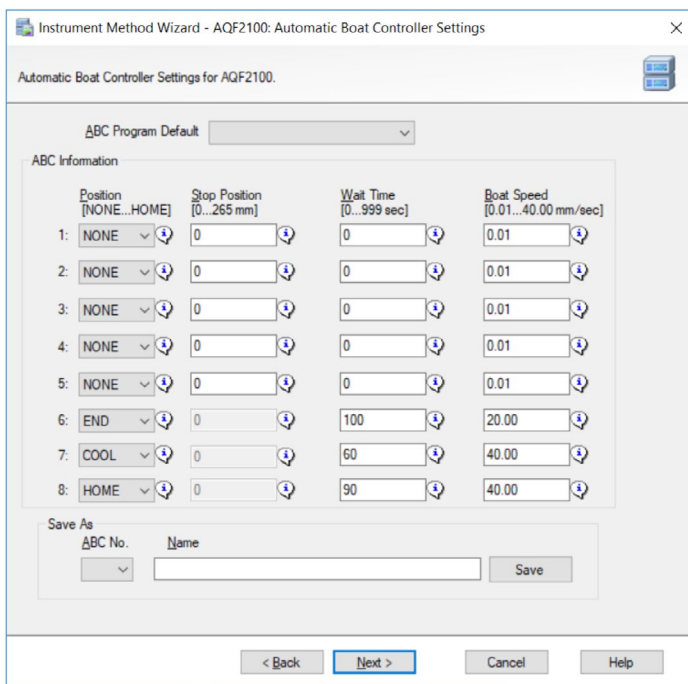
Figure 21. Creating the Instrument Method: Enter in Automatic Boat Controller settings from the Conditions section

14. Enter the IC conditions on the next tabs. On the Injection Type Selection settings tab, enter 0.1 min to load the sample and 15 s to inject the sample (Figure 22).

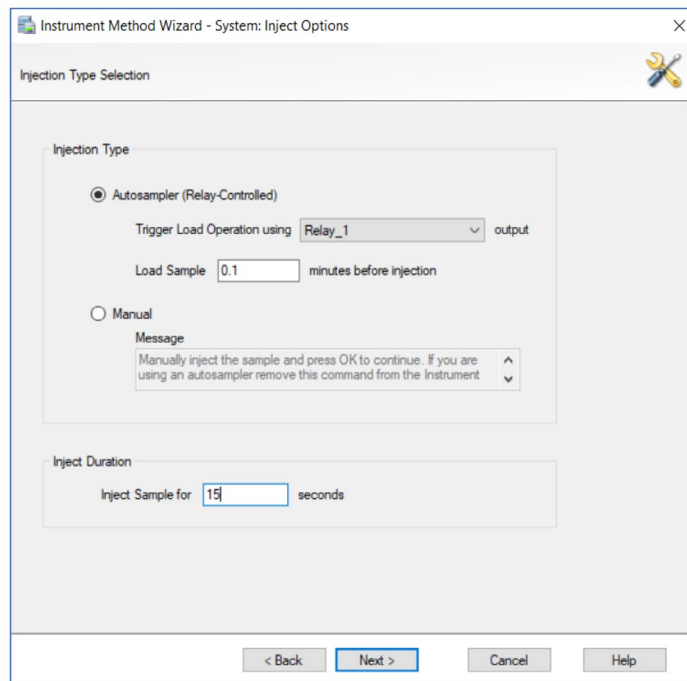15. Continue to enter the IC conditions. Save and check the Instrument Method.

Figure 22. Creating the Instrument Method: Injection Type Selection settings

Chromeleon CDS 7.3 AQF-2100 ePanel

The new Chromeleon CDS 7.3 AQF-2100 ePanel displays the combustion, absorption, and injection functions during the operation (Figure 23). The module lights reflect the status. Green indicates the module is inactive, yellow is active, and red indicates an error.

1. The first horizontal bar of the AQF-2100 ePanel has a Connect slider button to connect and disconnect the combustion system and Status message field.

2. The second horizontal bar displays the drop-down settings table under the Setting button, HF210 furnace status and temperatures, the argon and oxygen gas flows, ABC module, boat position, and sample changer arm movement (Arm Evacuation) field. The furnace and gas flow setting are set using the Settings button.

3. The third horizontal bar displays GA211 module activity, water supply for combustion, External Position for the ES-211 External Sampler sample position, and oxygen reading from the Combustion Monitor CM-210 module. The GA-211 pump light is yellow when the sample loading pump is loading the GA-211 sample loop. The GA-211 injection valve position is displayed as Inject when idle, Load and Inject during sample loading and injecting.

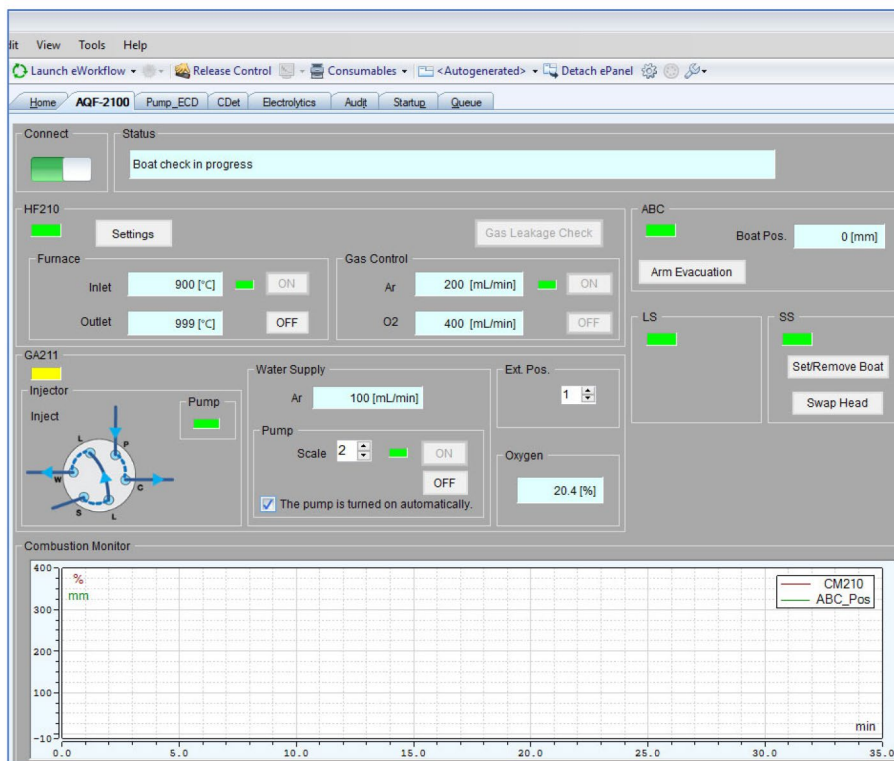4. The boat positions with the oxygen concentration (%) are plotted on the Combustion Monitor graph.

Figure 23. Chromeleon CDS 7.3 AQF-2100 ePanel

Using the custom variables

The combustion conditions are implemented through the custom variables (Figure 24), which are standard (despite their name) built-in configurable objects in Chromeleon CDS.

- Measurement_Type

  - The Measurement_Type defines the analysis type and controls the HF-210 furnace and GA-211 gas absorption modules.

  - Setting 00:Abs_Inj is a combustion treatment of the sample followed by absorption and injection.

  - Setting 01:Ext_Inj is an injection using the External Sampler ES-211 to bypass the combustion, absorption, and dilution steps and directly inject standards to the IC.

  - Setting 34:Heater_off turns off the HF-210 furnace. The furnace temperature starts to cool down to room temperature.

  - Setting 40:GA_AllWash programs the GA-211 to wash the GA-211 liquid lines. This step is recommended at the end of each sequence.

  - Setting 42:GA_EndProcess programs the GA-211 to wash and release the GA-211 module.

A typical process includes samples, liquid or solid, using 00:Abs_Inj or 01:Ext_Inj., followed by 40:GA_AllWash, 42:GA_EndProcess, 2 lines of 34:Heater_off, and 35:Gas_off.

- Pause

  Pause parameters are 0:Disable or 1:Enable. Automated sample introduction uses 0:Disable, where samples are combusted during the IC run and automatically injected when the programed IC_Time is completed. The pause enable function, 1:Enable, stops after each sample IC run and requires a manual start on the AQF-2100 ePanel. This function is useful when the analyst is preparing samples between injections.

- Injector

  The injector custom variable defines the sample injector: 0:solid, 1:liquid, 2:liquefied gas, 3:gas. The ES-211 External Sampler uses 1:liquid; the ASC-270LS liquid solid changer uses either 0:solid or 1:liquid; the Gas Injector GI-260 uses 2:liquefied gas or 3:gas.

- Sample

  The sample weight (mg) or sample volume (µL) is entered into the Sample custom variable.

- Absorption_Volume

  This is the volume of absorption solution in the absorption tube. The recommended volume is 3.5 mL for a 10 mL absorption tube.

- Constant_Volume

  The options for Constant_Volume are 0:Disable or 1:Enable. Constant_Volume is used for quantitation so it should always be enabled, 1:Enable.

- IC_Time

  The IC_Time (in seconds) parameter is used to overlap the combustion of the next sample in the sequence with the current sample being analyzed in the IC. However, the first sample in each sequence in the sequence queue must have an IC_time reflecting the combustion and the IC run. Two additional minutes are added to account for the sample changer operations. Three additional minutes are needed when using preconcentration. Considerations for run times are needed if IC equilibration times is required.

Example: IC run = 10 min (600 s), 5 min (300 s) combustion

$$IC\_Time(first\ sample) = 600 + 300 + 120 = 1{,}020\ s$$

$$IC\_Time(second\ sample) = 600 + 0 + 120 = 720\ s$$

Figure 24. Completed sequence displaying the custom variables

Starting the CIC system

To start the CIC system, first, start the combustion system. Open the Chromeleon Console and select Instrument view. Select the AQF-2100 ePanel and select the Settings button. The Settings window will open. Enter the argon (200 mL/min) and oxygen (400 mL/min) gas flows. Enter the furnace temperatures from the application, for example, 900 °C inlet and 1,000 °C outlet temperatures. Close the Settings window. On the AQF-2100 ePanel, Select the On buttons for the gas and furnace. The gas flows should immediately rise to the set conditions, and the furnace will start to heat up. The water supply argon flow rate is 100 mL/min.

To start the Dionex Integrion system, use the conditions listed in the application. Fill the DI water bottles with fresh ASTM DI water. Prime the pump by loosening the priming knob and pressing *Prime* on the Integrion Pump_ECD panel. When liquid is freely flowing from the pump, turn off the Prime and close the priming knob. Turn on the pump by entering the application flow rate. Immediately start the eluent flow by entering the eluent concentration. Turn on the Dionex CR-ATC 600 trap column and oven and detector temperatures. Turn on the ADRS 600 suppressor by setting constant voltage (2.1 V) or select constant current and enter the current (mA) from the application conditions.

The CIC system is ready when the furnace is at temperature and the total conductivity is <1 µS/cm.

Constant volume determination

Constant volume determination is needed because the combusted sample and directly injected standards experience different dilution effects. The sample is combusted under argon and then hydrated oxygen. During the combustion process, the gaseous sample is aerated into 3.5 mL of absorption solution. After combustion, the sample is diluted to approximately 10 mL. The constant volume determination is a critical parameter. The method calculates the exact dilution factor of the sample as compared to the standard injected directly to the IC. Both the injections use the same valve and the same injection loop.

To determine the constant volume factor, compare the peak area average of triplicate injections of 5.0 mg/L (ppm) phosphate standard directly using the ES-211 External Sampler and through the combustion system using empty, clean boats and 50 mg/L (ppm) phosphate standard as the absorption solution. To ensure the best results, prime the tubing for the ES-211 channel using the absorption solution with 5.0 mg/L (ppm) phosphate standard. The constant volume factor should be ~10x.

Consumables device monitoring

A new feature of the Dionex Integrion HPIC system is consumables device monitoring and tracking. This feature, **if activated,** automatically detects the electrolytic devices and the columns. Review and approval of the devices is required to start the first sequence on the Dionex Integrion HPIC system when new consumable devices are installed. To access this approval, select *Consumables* and then *Inventory.* The device monitoring shows the device history, tracking the part number, size, serial numbers, manufacture lot, installed location (On Device), and Best if Used by Date. Additionally, the device monitoring provides warnings if there is incompatibility with the devices installed.

- To start the sequence, correct any errors, review the inventory, Approve, and Close the page.

- Select the Instrument Queue tab and conduct a Ready Check on the sequence.

- Select Start.

Running the CIC system

To run the CIC system, complete the sequence by entering the sample information and the combustion conditions into the custom variables. Check that the DI water bottles are full, that there is sufficient gas in the tanks, the waste lines are directed to the waste container, and the Dionex Integrion HPIC and Nittoseiko AQF-2100H systems are ready. Add the sequence to queue (Instrument console, Queue tab). Verify that the sequence is correct by pressing *Sequence Ready Check.* Correct any errors, and press *Start.*

Conclusion

This technical note describes the installation and setup of a combustion IC system consisting of a Dionex Integrion HPIC system and a Nittoseiko AQF-2100H system for determination of total sulfur and total halogens in complex matrices. Both systems are operated as a single system under the control and data management of Chromeleon CDS 7 software.

Combustion IC applications can be found in the Thermo Scientific AppsLab Library of Analytical Applications.[7]

For further in-depth treatment of this topic, please consult with the treatise "Handbook of Ion Chromatography, 3–Volume Set, 4th Ed. (2016) ISBN 3527329285" by Joachim Weiss. This handbook can also be ordered from Thermo Fisher Scientific as P/N 1R120400-0050.

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