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 TN72211¹). 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).
- 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

 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.

Dionex Integrion HPIC System				X
Dionex integriori in re bystem				~
Thermal Controls TTL Inputs General Pump Device Name: Electrolytics	Options Detectors	Low E	Pressure Valves Sta lectrolytics Injer	ate Devices
Name	Connector	Auto	Description	CC/CV
Suppressor	A	Yes	Suppressor	CC/CV
CR_TC	В	Yes	CR_TC	
EluentGenerator	С	Yes	Eluent Generator	
<				>
To remove an unused device fror Double-click or press F2 to edit t	n the instrumer he current sele	nt, clearth ction.	e corresponding check t	XX.
		Ok	Cancel	Help

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).

Devices	×
Device <u>N</u> ame: Pump_1	
l✓ Pressure Signal(s)	Keep pumping even if communication fails
< <u>B</u> ack	Next > Cancel Help

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

 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).

AQF-2100 Configuration	×
General Gas Absorption Sample Injector	
Connection Enable Simulation Mode COM No: Prolific USB+to-Serial Comm Port (COM4)	
Search	
Device Configuration	
System Name: AQF2100	
Furnace	
HF-210	
Device Name: HF210	
OK Can	cel

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).
 - \times AQF-2100 Configuration General Gas Absorption Sample Injector Connection Enable Simulation Mode COM No: Prolific USB-to-Serial Comm Port (COM4) -Search Device Configuration System Name: AQF2100 Furnace HF-210 • Device Name: HF210 ОK Cancel
 - Figure 6. General tab. Select the furnace type.

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

👌 AQF-2100 Configuration	х
General Gas Absorption Sample Injector	
Select the following items for GA-210/GA-211. Device Device Name: GA211 Absorption Tube Type (mL) ES(External Solution Selection Valve)	
ES-211	
Combustion Monitor CM-210 Device Name: CM210	
OK Cancel	

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).

AQF-2100 Configuration	Х
General Gas Absorption Sample Injector	
Select a Sample Injector	
Device Name	
Automatic Boat Controller : ABC	
Liquid Sampler: LS	
Solid Sampler: SS	
Gas Injector:	
Liquid Sampler Settings	
Tray Type: 4mL	
Syringe Size (uL): 50 🔽	
OK	el 🛛

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 \times 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.

- 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.

Time	Device	Command	Value	Description
-1.1 Equilibr	ation 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
	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
-1.0	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 Pr	reparation			
	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.

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

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.

Name	Context	Туре	Description	Items			Default	
Davias	Inication	Liet	Deuee	0:Disa	able		0.Diachle	
Pause	Injection	LIST	Pause	1:Ena	ble		U:DISable	5
				0:Mar	nual_mode			
				1:Soli	d			
Injector	Injection	List	Injector	2:Liqu	lid		0:Manua	l_mode
				3:Liqu	uefied_gas			
				4:Gas	;			
				00:Ab	os_Inj			
				01:Ex	t_lnj			
				30:Bc	oat_prebake			
Measurement Type	Injection	Liet	Measurement Type	34:He	eater_off		00.Abs	Ini
Weddarennent_Type	njootion	LIOT	Weddarennent_type	35:Ga	as_off		00.7 00_	
				40:GA	A_EndProce	ssing		
				41:Se	nsor_Check			
				42:GA	A_AllWash			
Constant Volume	Injection	List	Constant Volume	0:Disa	able		0.Disable	2
oonotant_volanio	ngoodon	Liot	conclant_volamo	1:Ena	ble		0.0100.010	
Name	Context	Туре	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	0	0	6,000	S	0

Table 2. Creating custom variable tables

seconds +120 s

Name Context Value Type Description I Pause Injection List 1 Pause Injection Numeric 2 Sample Injection List 3 Injector Injection List 4 Measurement_Type Injection List 5 Absorption, Volume Injection List 5 Absorption, Volume Injection List 7 Allow empty galues Enter the list tems (one per line): 6 Otabs_inj Items Add 00.4bs_inj 01-Ext_inj 30-Boat_Prebake Add 34 Heater_off Move Lip Move Lip Move Down Defet Move Down Move Down Default value: 00-Abs_inj Move Down Move Down Add Dejete OK Cancel	ust	tom Variables Editor fo	or Data Vault (ChromeleonLoca	al - 6 Item(s)					?	×	
Add Defete Add Defete		Name	Context	Value Type	Description	Context:	Injection	,	4			
1 Pause Injection List Name: Measurement_Type 2 Sample Injection Numeric Degorption: CEnter custom variable description> 3 Injector Injection List Degorption: CEnter custom variable description> 4 Measurement_Type Injection List Image: Measurement_Type 5 Absorption_Volume Injection List Image: Center custom variable description> 5 Constant_Volume Injection List Image: Center custom variable description> 6 Absorption_Volume Injection List Image: Center custom variable description> 6 Constant_Volume Injection List Image: Add 00:Abs_hij OitExt_hij Items Add 00:Abs_hij OitExt_hij Move Down Move Down Add Dejete OK Cancel 35:Gas_Off 40:GA_EndProcessing 41:Sensor_check 42:GA_Alwash	¥											
2 Sample Injection Numeric 3 Injection List Imjection 4 Measurement_Type Injection List Type: 5 Absorption_Volume Injection List Imjection 5 Absorption_Volume Injection List Imjection 6 Constant_Volume Injection List Imjection 7 Secondard Type: List Add 6 Constant_Volume Injection List Add 9 Constant_Volume Injection List Move Up 10 Cot_Inji Otext_Inji Move Down Move Down 10 Defete Otext_Inji Move Down Move Down 10 Cot_Inji Otext_Inji Move Down Move Down 10 Cot_Inji Imjection	1	Pause	Injection	List		Name:	Measurement_Type					
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Absorption_Volume Injection Numeric Absorption_Volume Injection List Items Add OtAbs_Inj Delete Move Up Move Up Move Dogn Default value: OtAbs_Inj OtAbs_Inj OtAbs_Inj Move Dogn Add Delete OK Cancel Add Delete	4	Measurement_Type	Injection	List		Type:	List	3	v.			
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35:Gas_Off 40:GA_EndProcessing 41:Sensor_check 42:GA_Allwash		Add Delete.							ОК	Can	cel	
35:Gas_Off 40:GA_EndProcessing 41:Sensor_check 42:GA_Allwash						_		_				
40:GA_EndProcessing 41:Sensor_check 42:GA_Allwash						35:Gas_Off						
41:Sensor_check 42:GA_Allwash						40:GA_EndP	rocessing	_				
42-GA Allwash						41:Sensor_ch	neck					
14-547 S (1017 S (117						42:GA Allwa	sh					

Figure 13. Creating custom variables: Measurement_Type

- 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).
- Highlight the custom variables table. Select *Finish.* (Figure 14) All custom variables should be visible. Verify the "Value Type" to check for errors.
- 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).

Incomplete Resume	•				
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	Name	Value Type	Description		
	Pause	List			
	Sample	Numeric			
	Injector	List			
	Measurement_Type	List			
	Absorption_Volume	Numeric			
	Constant_Volume	List			
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Figure 14. Importing Custom Variables Editor

Incomplete	Resume									ic-ns (Idle)
🔚 Save 🧿 Studio 🛃 I	Print 🔹 💼 Up 📴 Insert Row 🔹 🎛 Fill Down 🔒 Lock 🍸 Fil	tering 冒 Grouping	f_{χ} Custor	n Columns 🔹		🔹 🏨 Find Next 🔹				
# CD_Total > Na	ame	Туре	Level	Position	Volume [uL]	*Measurement_Type	Instrument Method	*Pause	"Sample Imol *Injector	"Absorption_Volume [mL]

Figure 15. Organizing custom columns

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

Instrum	nent Method Wizard	System: General Settings	×
eneral Se	ttings for System.		×
Run Time Please 20.000	specify the run time of th	ne method:	
Diagnosti Select	c Channels diagnostic channels to b	be used:	
No 1			Select all channels
2	CM210		Deselect all channels
3	Pump_1_Pressure		
		(Bala News	

Figure 16. Creating the Instrument Method: Selecting all channels

10. Enter the Gas Absorption conditions: Water injection time:12; Drain time: 17; Gas Line Washing Times: 3; Sample Absorption Time: 10 (Figure 17).

📑 Instrument N	lethod Wizard - AQF2100: Au	tomatic Gas Abso	orption Settings	×
Automatic Gas Ab	sorption Settings for AQF2100.			
Washing				
	Water Injection Time	12	[0.099.9 sec]	
	<u>D</u> rain Time	17	[0.099.9 sec]	
	Gas Line Washing Times	3	[099]	
Injection				
	Sample Absorption Time	10.0	[0.0999.9 sec]	
	< Bac	k Next >	Cancel	Help

Figure 17. Creating the Instrument Method: Automatic gas absorption settings

11. Use default settings for Furnace Combustion Conditions (Figure 18).

mace Settings for AQF2100.		
ombustion Time		
E	y Time 👖 🕄 [0999 sec]	
<u>0</u>	2 Time 600 (0999 sec)	
<u>A</u> r Replac	e Time 30 (0999 sec)	

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).

📸 Instrument Method Wizard - AQF2100: Solid Sam	npler Settings		×
Solid Sampler Settings for AQF2100.			
Boat Operation			
$\underline{W} aiting time after closing the sample box$	5	✓ (i) [0999 sec]	
Return position after boat detection	A	√ 🎲 [1A]	
Boat return position	Return	~ 4	
< <u>B</u> ack	<u>N</u> ext >	Cancel	Help

Figure 19. Creating the Instrument Method: Default ASC-270LS Solid Sampler settings

uid Sampler	Settings for AQF2100.			
ommon				
	Bottle B setting	Not_Used	$\sim \odot$	
Securities				
amping	Aspiration Rate [u] /sec]	20 3		
	Wait Time after Sample Appiration [eac]			
	Trac fine and sample represent [see]		10 12	
niection			[012 02]	
njection	Injection Rate [uL/sec]	40 ~ (1)		
	Wait Time after Sample Injection [eac]			
	War nine arter Sample Injection [sec]	· · · · · · ·		
Vashing	Wash Number	2		
	<u>11</u> 0011101100	4		
linse	Rinse Number	1 ~3		
	_	4		
	< <u>B</u> ack	<u>N</u> ext >	Cancel	Help

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

13. Enter Automatic Boat Controller settings from the Conditions section (Figure 21).

ABC Program [Default		~	/			
Position [NONEHOME	Stop Posit	tion m]	Wait Time	í.	Boat Spee	ed 00 mm/sec]	
1: NONE V	0	•	0	•	0.01	•	
2: NONE ~	0	•	0	•	0.01	•	
3: NONE ~	0	\$	0	•	0.01	•	
4: NONE ~	0	•	0	•	0.01	•	
5: NONE ~	0	•	0	•	0.01	•	
6: END 🗸	0	٩	100	•	20.00	•	
7: COOL 🗸	0	٩	60	\$	40.00	•	
8: HOME 🗸	0	•	90	\$	40.00	•	
Save As	Name						
	Manie				Save	A	

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

- 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.

Injection Type				
(Au	tosampler (Relay-Contro	alled)		
0.0	Trigger Load Operatio	on using Relay 1	✓ output	
	Load Sample 01	minutes before inject	ion	
- ···		minutes before inject		
⊖ Ma	Message			
	Manually inject the sa using an autosampler	ample and press OK to continue r remove this command from the	If you are A	
Inject Duration	Sample for 15	seconds		
nijec	aniple for 15	seconds		

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

	Run Finished														di	c-ns 🔹 (Id
	Save 🗿 Studio (🛃 Print 🕞 💼 Up 🗌 📴 Inse	rt Row 🔹 📳 Fi	II Down 🔒 Lo	ock Y	Filtering 📇	Grouping f_x (Sustom	Columns -		- 4	Find Next 🔹				
#	CD_Total +	Name	Туре	Level	Posi tion	Volume [uL]	Instrument Method	Proce	Status	"Sample	*Injector	"Measurement_I	Absorption Volume Im	TL_1 me [sec]	"Constant_Vol ume	*Pause
1	None	unintended error: forg	Unknown		12	100.0	Extended run	7-A_	Interrupted	40	2:Liquid	00:Abs_Inj	4	5500	1:Enable	0:Disable
2	None	unintended error: forg_	Unknown		12	100.0	Extended run	7-A_	Interrupted	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	1:Enable
3	None	unintended error: forg	Unknown		12	100.0	Extended run	7-A	Interrupted	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	1:Enable
4	*	manual start from pan	Unknown		7	100.0	Extended run	7-A	Finished	40	2:Liquid	00:Abs_Inj	4	5500	1:Enable	1:Enable
5	4	manual start from pan	Unknown		7	100.0	Extended run	7-A_	Finished	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	1:Enable
6	None	combustioncc, isocra	Unknown		7	100.0	Extended run	7-A	Interrupted	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	1:Enable
7	None	combustioncc, isocra	Unknown		12	100.0	Extended run	7-A	Interrupted	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	0:Disable
8	None	combustioncc, isocra	Unknown		7	100.0	extended run	7-A_	Interrupted	40	2:Liquid	42:GA_AllWash	4	3900	1:Enable	0:Disable
9	*	manual start from pan	Unknown		7	100.0	extended run	7-A_	Finished	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	0:Disable
10	*	manual start from pan	Unknown		12	100.0	extended run	7-A	Finished	40	2:Liqu	00:Abs_Inj	4	3900	1:Enable	0:Disable
11	*	combustioncc, isocra	Unknown		7	100.0	extended run	7-A.	Finished	40	2:Liquid	00:Abs_Inj	4	3900	1:Enable	0:Disable
12	man	🛛 wash	Unknown	0101.6000000000000000000000000000000000	7	100.0	extended run	7-A_	Finished	40	2:Liquid	42:GA_AllWash	4	3900	1:Enable	0:Disable
13		Shutdown	Unknown		7	100.0	extended run	7-A	Finished	40	2:Liquid	40:GA_EndPr_	4	3900	1:Enable	0:Disable
14		Shutdown	Unknown		7	100.0	extended run	7-A	Finished	40	2:Liquid	34:Heater_off	4	3900	1:Enable	0:Disable
15	1	Shutdown	Unknown	l.	7	100.0	extended run	7-A	Interrupted	40	2:Liquid	35:Gas_off	4	3900	1:Enable	0:Disable

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 $<1 \ \mu$ S/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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