Combustion-IC

Configuring a combustion-ion chromatography system using a complete workflow

Authors
Terri Christison and Neil Rumachik

Keywords
C-IC, combustion-IC, PFAS, EPA 1621, pyrolysis, Cindion

Introduction

Analyzing samples with complex matrices can be challenging due to the incompatibility of the sample's physical properties with the analytical technique. For example, ionic halide content can only be measured by ion chromatography (IC) when the sample is fully dissolved in a liquid, preferably water, extracted, or washed off the surface of a solid material. Combustion-ion chromatography (C-IC) has the advantage of eliminating complex sample matrices to directly measure the total halogens and total sulfur.[1-5] C-IC has been demonstrated for halide determinations in other challenging liquid matrices, such as tea and wastewater.[6,7] In addition, adsorbable organic fluorine (AOF), in conjunction with C-IC, has been demonstrated and incorporated into U.S. EPA Method 1621 to screen for per- and polyfluoroalkyl substances (PFAS) in water samples.[8-14]

A new C-IC total workflow system has been introduced: the Thermo Scientific™ Cindion™ Combustion Ion Chromatography System. It includes a standalone sample preparation module, the Thermo Scientific™ Cindion™ Combustion/Absorption Module, and an autosampler that can be configured for solid or liquid samples. A standalone Thermo Scientific™ Cindion™ LPG/gas Module is also an option.

In this technical note, we describe the configuration and operation of the Cindion C-IC system with the following configurations: Cindion combustion/absorption Module with Cindion autosampler and each of numbers 1-4 below.

1. Solids sampler and with the offline Cindion adsorption module needed for U.S. EPA Method 1621[15]
2. Solids sampler
3. Liquids sampler
4. Eluent monitor with any configuration

thermo scientific

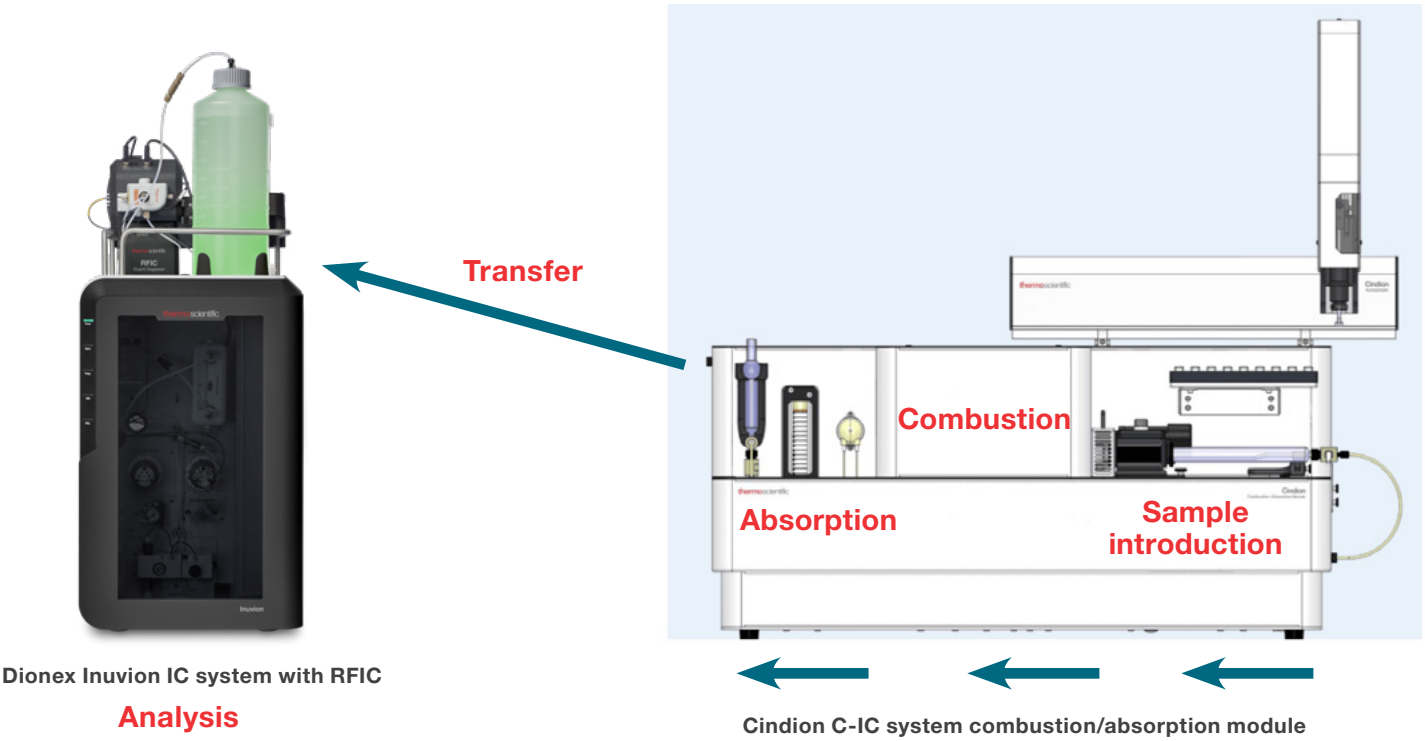


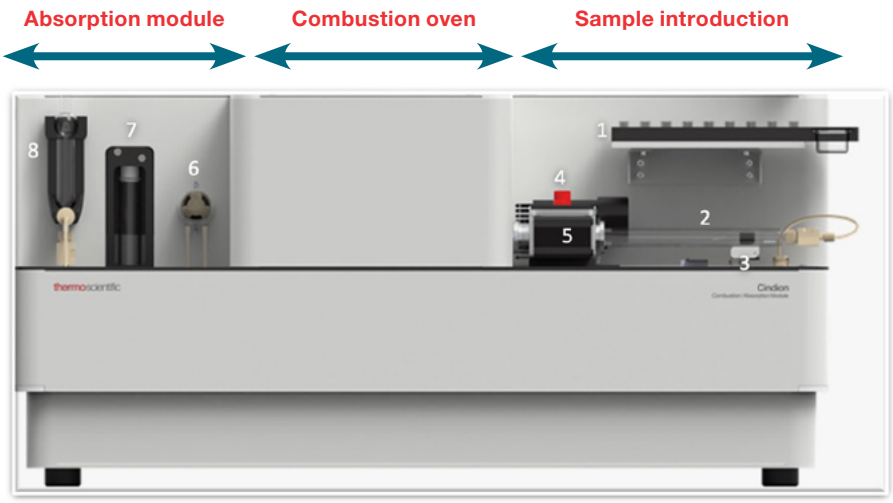
Figure 1. Sample flow through the Cindion C-IC system

Instruments

Cindion C-IC system:

- Cindion combustion/absorption module (P/N B51006425) with Thermo Scientific™ Cindion™ C-IC Solids Kit (P/N B51006427)
- Thermo Scientific™ Cindion™ Solids/Liquids Autosampler (P/N B51006429)
- Thermo Scientific™ Cindion™ Adsorption Module (P/N B51006430) that includes 6 column holders
- Thermo Scientific™ Dionex™ Inuvion™ with Reagent-free Ion Chromatography (RFIC™) System (P/N 22185-60108)
- Optional Thermo Scientific™ Dionex™ Inuvion™ Eluent Monitor, 4 L (P/N 22185-62708)
- Thermo Scientific™ Muffle Furnace, 2 L small bench top, to clean combustion cups, (Thermo Scientific FB1415M, Fisher Scientific 10-552) – recommended for U.S. EPA Method 1621

Figure 2 shows the components of the Cindion C-IC combustion/absorption system.



1. Solids tray
2. Boat guiding tube
3. Boat drive
4. Boat injection port
5. Boat station
6. Peristaltic pump for hydration
7. Sample syringe
8. Absorption tube

Figure 2. Components of the Cindion combustion/absorption module

Software

Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS), version 7.3.2 MUd with Cindion C-IC and eluent monitor drivers.

Consumables

IC Consumables

- Dionex Inuvion eluent monitor, optional, 4 L (P/N 22185-62708)
- Dionex Inuvion eluent monitor, optional, 2 L (P/N 22185-62707)
- Thermo Scientific™ Dionex™ IonPac™ Columns, 2 mm i.d. analytical guard columns, defined by your application
- Thermo Scientific™ Dionex™ EGC 500 KOH Eluent Generator Cartridge: (P/N 075778)
- Thermo Scientific™ Dionex™ CR-ATC 600 Electrolytic Trap Column: (P/N 88662)
- Thermo Scientific™ Dionex™ Inuvion™ RFIC Eluent Degasser Module: (P/N 106-60001)
- Thermo Scientific™ Dionex™ ADRS 600 Anion Suppressor (2 mm): (P/N 088667)
- Thermo Scientific™ Dionex™ E/O Eluent Organizer with two 2 L HDPE eluent bottles (P/N 072057), plus one 4 L HDPE eluent bottle (P/N 063292)
- Thermo Scientific™ Dionex™ IC PEEK Viper™ Fitting Tubing Assembly Kit (P/N B51000232)
- PEEK Dionex sample loop: 100 µL, P/N 042951

General

- Fisherbrand™ Elite™ adjustable, 200 µL pipette and pipette tips: Fisher Scientific (P/N FBE00200) and (P/N 02-707-409)
- Fisherbrand™ Nitrile Class 10 Cleanroom Non-Sterile Gloves (ISO 4), (P/N 12892004 (A-F X-Small to 2X-Large))
- Polypropylene vials, 1 mL, recommended Fisher Scientific (P/N 03-452-358) and polyethylene caps, PFAS-free vials and caps to store open PFAS standards Fisher Scientific (P/N 03-452-581)

Liquids sampler

- Thermo Scientific Cindion 2 mL vial with septa, 100/pack (P/N B51006450)
- Quartz wool used in combustion tube and used to absorb liquid injections (P/N B51006451)

Solids sampler

- Polypropylene vials, 1 mL, recommended Fisher Scientific (P/N 03-452-358) and polyethylene caps, PFAS-free vials and caps to store open PFAS standards Fisher Scientific (P/N 03-452-581)
- Fisherbrand™ Crucible tongs, Fisher Scientific (P/N 13-820-023)
- Fisherbrand™ Nitrile Cleanroom gloves, Class 10, ISO 4, Fisher Scientific (P/N 12892004 (A-F X-Small to 2X-Large))
- Fisherbrand™ Elite™ pipette, adjustable, 200 µL pipette and Fisherbrand™ SureOne™ pipette tips: Fisher Scientific (P/N FBE00200) and (P/N 02-707-409)
- Fisherbrand™ Stainless steel tweezers, Fisher Scientific (P/N 12-000-157)
- Newtex ZetexPlus heat resistant gloves, 200 series, Fisher Scientific (P/N 11-392-53)
- Thermo Scientific™ Cindion™ C-IC Solids sampler kit (solid boat, solids tray, solids gripper), reserve supplies, Thermo Scientific (P/N B51006427)
- Thermo Scientific™ Cindion™ C-IC Solids quartz cups, Thermo Scientific (P/N B51006446)
- Thermo Scientific™ Nalgene™ HDPE, wide mouth, 125 mL bottles, Fisher Scientific (P/N 02-893-5C)
- Thermo Scientific™ Nalgene™ vacuum polypropylene desiccator to store clean combustion cups, Fisher Scientific (P/N 08-642-5)

Figure 3 shows the Cindion C-IC glassware.

Adsorption module

- Thermo Scientific™ Cindion™ C-IC Granular Activated Carbon (GAC) Columns (P/N B51006452, package of 100) and Column Holders (P/N B1006453, each) used to adsorb samples using Cindion C-IC adsorption module
- Hamilton 700 Microsyringe, 100 µL, metal plunger: Fisher Scientific (P/N 14-813-091)

#	Item
1	Absorption tube
2	Solids boat
3	Liquids boat
4	Z-fold combustion tube
5	Solids gripper, sample handler
6	Liquids handler syringe
7	Quartz combustion cup

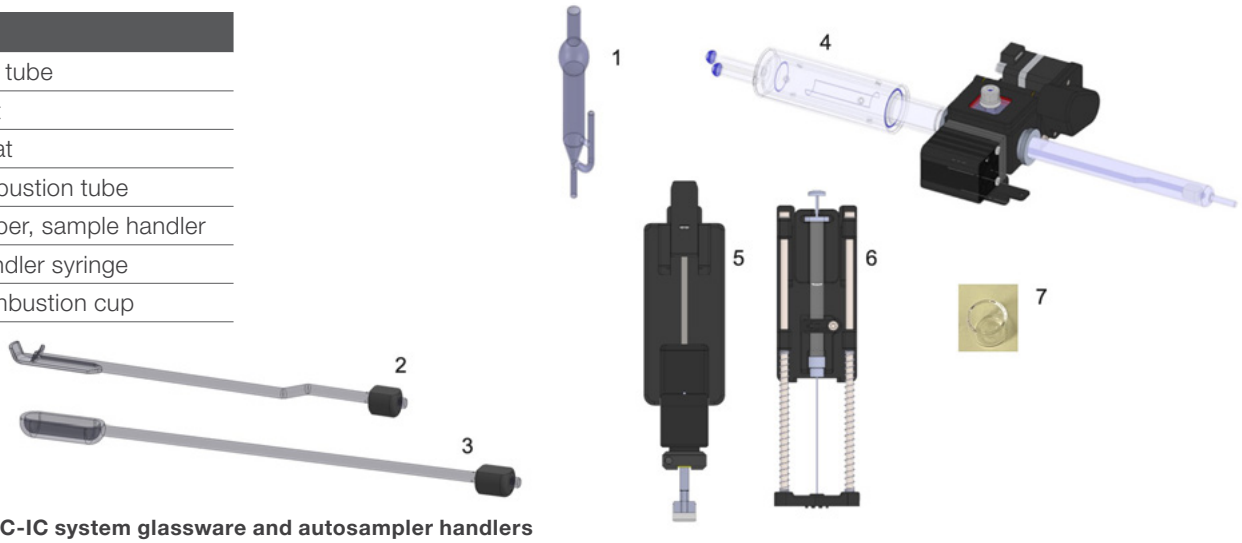


Figure 3. Cindion C-IC system glassware and autosampler handlers

Reagents

- DI water (18 MΩ cm resistivity, TOC < 50 ng/mL)

Reagents for cleaning containers and Cindion C-IC adsorption unit for U.S. EPA Method 1621

- Ammonium hydroxide, 28-30 w/w%, ACS certified, Fisher Scientific (**P/N A669S-500**) (CAS 1336-21-6)
- Methanol, UHPLC-MS grade, Fisher Scientific (**P/N A458-1**) (CAS 67-56-1) lowest PFAS-containing methanol
- Methanol, Optima grade for cleaning, Fisher Scientific (**P/N A454-4**) (CAS 67-56-1)

Reagents for U.S. EPA Method 1621

- Sodium nitrate, ACS certified, Fisher Scientific (P/N S343-3) (CAS 7631-99-4)
- For PFAS methods using the Cindion C-IC adsorption unit: 50 µg/mL (in methanol) L-PFHxS sodium perfluoro-1-hexanesulfonate standard (Wellington Laboratories L-PFHxS), used for spike-recovery experiments (F equivalence = 64.6%; 29.26 µg/mL F)
- Drierite, indicating, 500g, 8 mesh for storing clean combustion cups, Thermo Scientific (**P/N 21909-5000**)
- Thermo Scientific™ Dionex™ chloride standard, 1000 mg/L (**P/N 037159**)
- Thermo Scientific™ Dionex™ fluoride standard, 1000 mg/L (**P/N 037158**)
- 1000 mg/L Fluoride standard, second source, SPEX CertiPrep, Fisher Scientific (**P/N AS-F9-5Y**)

Optional reagents

- Certified reference materials
- Halogen reagents: chlorobenzene, fluorobenzene, bromobenzene

Instrument configuration

The C-IC system is controlled and managed by Chromeleon CDS with ePanels automatically generated for the Dionex Inuvion IC system and Cindion C-IC system in the Instrument Console. The main ePanel has most of the controls and displays needed to run the instrument. The Cindion C-IC system has additional sub-ePanels for less frequently used controls.

Initially, during the power-up, the Cindion C-IC system LED will light up yellow. Wait until the Cindion C-IC system homing process is completed: the absorber syringe moves up and down, and the boat door opens and closes. The boat tube also moves but is typically not noticeable. After the homing process is finished, the LED will turn green.

1. **Creating a C-IC Chromeleon Instrument Configuration**
   - To create a Chromeleon configuration file, Start Chromeleon Services Manager, select configure instrument and then create an instrument name, such as Cindion_CIC
   - Add the Dionex Inuvion IC system module from the list under Thermo Scientific, IC: Dionex Integrated Modules
   - Add the DDK Cindion module from the module list under Thermo Scientific (Figure 4)
   - Select OK

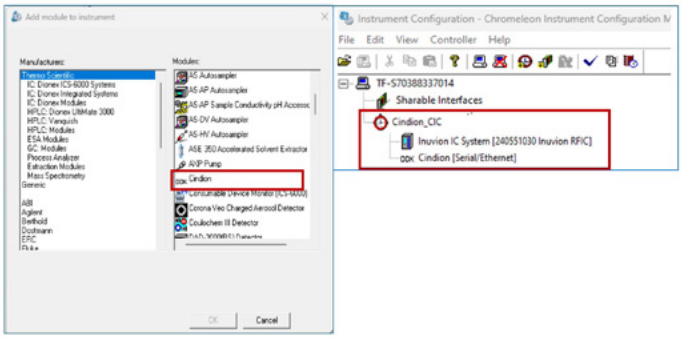


Figure 4. Chromeleon instrument configuration: Add DDK Cindion module

- Right-click on the DDK Cindion icon. The communication channels will be displayed (Figure 5). By default, the Cindion C-IC autosampler, oven, and absorber communicate through the IP addresses. Record your IP addresses for historical reference. In the case where IP addresses can't be used, the configuration can be set to communicate through com ports. The Chromeleon Instrument Configuration is the same for the solids tray and the liquids tray.
- Select OK. Important: Wait while the Cindion C-IC configuration checks are complete with the ending messages of 'Finished Loop' and 'Cindion Connection established successfully.' Save and close the configuration.

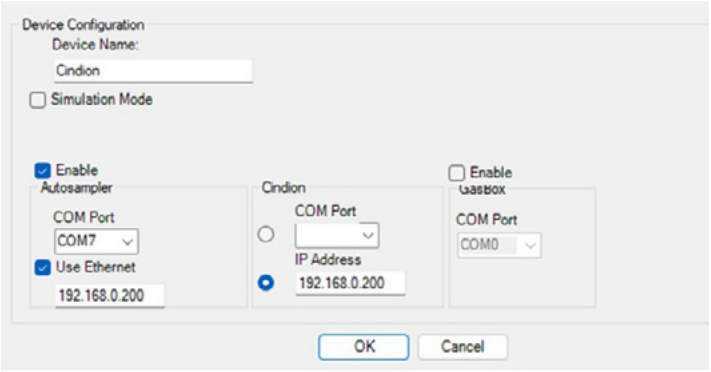


Figure 5. IP and COM addresses for Cindion C-IC system configuration

2. **Configuring and installing the Dionex eluent monitor**

   The Dionex eluent monitor, offered in 4 L and 2 L bottle sizes, glows green when the water container is full and glows red when the container is either empty or below your previously set "empty" setting. We recommend using the Dionex eluent monitor 4 L bottle size for the Cindion C-IC system absorber wash solution. It is also advantageous to use a 2 L eluent monitor for the 2 L Dionex Inuvion eluent bottle.

   - To install the eluent monitor modules, refer to the Dionex Inuvion IC System operating manual[16]
   - Add the eluent monitor to the Chromeleon CDS instrument configuration, under the Thermo Scientific drop-down menu
   - Set the low-level set point and the intensity for the green and red glow.

3. **Configuring the Cindion C-IC adsorption module**

   The Cindion C-IC adsorption module is an offline, standalone unit with a touchscreen control system. No other configuration is needed.

Instrument setup

1. **Installing the Dionex Inuvion IC system with RFIC**

   Install and set up the Dionex Inuvion IC system with RFIC as illustrated in Figure 6 and the instrument and consumable manuals.[16-22] Alternatively, an autosampler can be installed temporarily or permanently into the Chromeleon configuration.[22]

2. **Conditioning and hydrating IC consumables**

   Condition and install the IC consumables according to the Figure 6 flow diagram and the instructions under the Consumables drop-down menu (Figure 7), except for the suppressor. During conditioning, direct all flow output to waste. First, install and condition the Dionex EGC 500 KOH cartridge, and then install and condition the CR-ATC 600 trap column with the cartridge. Next, install and condition the guard column for at least 30 minutes using the QAR conditions from the storage box of the analytical column. Ensure that flow is directed to waste after the guard. Following this, remove the guard column and install the analytical column. Repeat the 30-minute conditioning for the analytical column using the same QAR conditions with flow directed to waste. Re-install both the guard and the analytical columns.

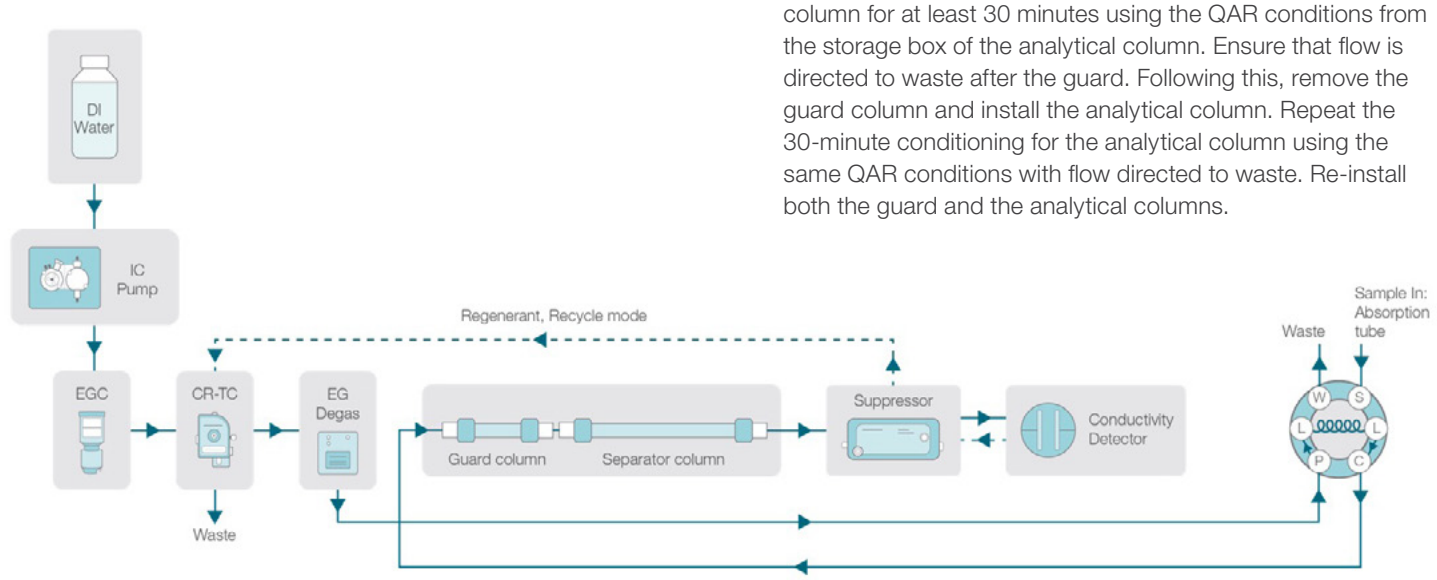


Figure 6. Dionex Inuvion IC system with RFIC flow diagram

It is recommended for best results, to hydrate the suppressor for 10 min using DI water at 0.2 mL/min and 0.5 mL/min 20 min. The suppressor can be installed after this process. Install the suppressor into the flow path according to Figure 6. Additional information can be found in the product manuals.[16-22]

To condition the Dionex Inuvion IC system temporarily install the sample loop listed in the QAR report and condition the Inuvion IC system overnight using the conditions listed in the column's QAR report. Prepare the QAR report standard. Temporarily install a luer lock connection on the manual injection port. Create a sequence using the QAR conditions. Run n = 7 injections using a manual start and a 1 mL syringe to dispense the standard. Verify the total conductivity baseline has <1 µS/min drift and verify the results against the QAR report. Remove the luer lock connection, reinstate the connection to the Cindion C-IC combustion system, and re-install the 100 µL sample loop.

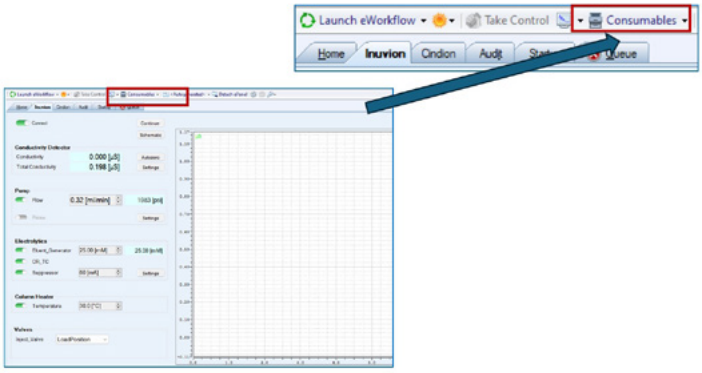


Figure 7: Use the Consumables drop-down menu to condition the IC consumables.

3. **Installing the Cindion C-IC combustion/absorption module**

   The Cindion C-IC system installation is managed by the service organization. The autosampler calibration data are saved in the service sub-ePanels. It is advisable to record the parameters for historical records and as part of good lab practices.

   If it is necessary to switch assemblies from solids to liquids, follow the instructions on the Sample Type button on the main ePanel (Figure 8). It is important that the boat tube, which is the glass tube enclosing the boat and boat arm, is properly sealed into the docking station. Videos on how to switch assemblies are available.[23-24] For U.S. EPA Method 1621, only oxygen gas is needed for the combustion process. The oxygen line is split into two lines entering Cindion as the hydration carrier and combustion gas.

   For the liquid sampler, provide sample vials to the service representative to ensure the best alignment.

4. **Installing the Cindion C-IC system transfer line to the Dionex Inuvion IC system injection valve**

   Refer to the Dionex Inuvion IC system operating manual. Green PEEK tubing (0.02 in i.d., 0.51 mm i.d.) should be used in the transfer line from the manual injection port connector to the Dionex Inuvion IC system injection valve to minimize back pressure to the absorber syringe in Cindion C-IC system.

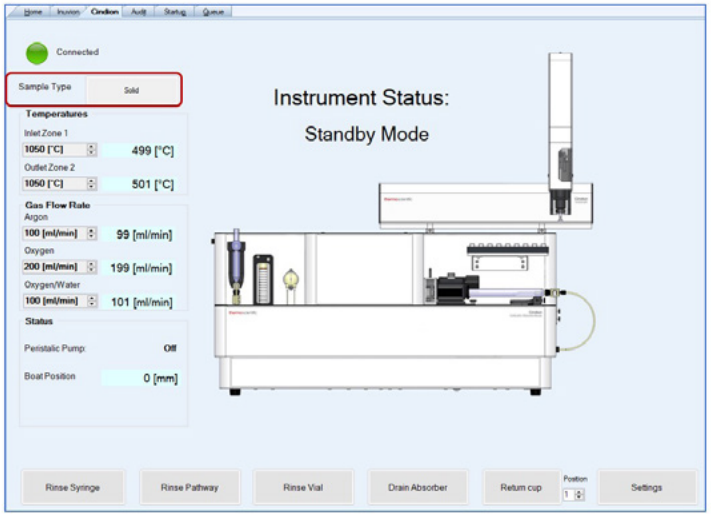


Figure 8. Use the Sample Type button to switch to different sample handling

Preparing and cleaning the instruments prior to analysis

To condition and clean the Cindion C-IC system for trace-level analyses, it is critical to clean and condition all instruments to their best operating conditions.

1. **Conditioning the Dionex Inuvion IC system**

   The best results are obtained when the Dionex Inuvion IC system has total conductivity readings < 1 µS-cm. To accelerate the conditioning, run an eluent gradient from low to high concentration, for example, start at 5 mM KOH and increase to 75 mM KOH over 10 min. Repeat the gradient eluent profile three times. If the total conductivity remains high, condition the Inuvion at 80 mM KOH for one hour,[5-10] mM above the intended gradient, and then re-equilibrate at the starting eluent conditions.

2. **Starting the Cindion C-IC combustion/absorption system**

   Rinse one 4 L, one 2 L, and one 125 mL HDPE bottle with methanol followed by 6 rinses with DI water. Install the 100 mL bottle at the hydration connection, the 2 L bottle for the absorber solution, and 4 L for the absorber wash solution. DI water is the absorber solution when determining only halides, whereas 100 to 1000 mg/L hydrogen peroxide solution, dependent on the application, is recommended as the absorber solution to convert sulfur species to sulfate.

   To start the furnace and gas flows, enter the temperatures and gas flows on the main Cindion C-IC ePanel (Figure 8). Enter the recommended combustion conditions in Table 1 to heat up the Cindion C-IC system.

3. **Cleaning the Cindion C-IC absorption module**

   Cleaning the C-IC system is essential when analyzing concentrations at µg/L (ppb) levels, such as is needed for U.S. EPA Method 1621. The recommended cleaning order of the Cindion C-IC system is to clean the Cindion C-IC absorber module first, followed by the combustion oven, sample boat (liquid sampler), and finally, cups (solids) and vials (liquid).

Table 1. Initial combustion conditions for method development

Recommended conditions	U.S. EPA Method 1621*	Organic or volatile samples	Inorganic samples
Furnace: Heater 1 (ºC)	1050	1050	1050
Furnace: Heater 2 (ºC)	1050	1050	1050
Argon carrier (mL/min)	Oxygen: 100	Argon: 150	Argon: 150
Oxygen primary (mL/min)	Oxygen: 200	Oxygen: 250	Oxygen: 250
Oxygen/water (turbo) (mL/min)	Oxygen: 100	Oxygen: 100	Oxygen: 100
Boat programs	3 mm/s to 75 mm, hold 60 s	1 mm/s to 75, hold longer	3 mm/s to 150 mm, hold 180-300s
	3 mm/s to 150 mm, hold 300 s	1 mm/s to 100 mm, hold longer	
		1 mm/s to 150 mm, hold longer	

*For U.S. EPA Method 1621, all three gas channels are oxygen

The absorber routines are located on the main Cindion C-IC ePanel. Run these routines 3 to 5 times, in this order: Rinse Syringe, Rinse Pathway, Rinse Vial, and Drain Absorber (Figure 8).

4. **Cleaning the Cindion C-IC combustion oven**

   To clean the combustion oven when using the solid tray and solid boat, create a sequence with positions where cups are absent (“no cup”). When the oven reaches temperature, run the sequence with the Cindion C-IC and Dionex Inuvion IC systems connected (see Creating Chromeleon Instrument sequences section). For U.S. EPA Method 1621, the expectations are that the baseline contamination of fluoride should be < 0.06 µS-min area counts after six “no cup” injections. Run additional “no cup” injections if the fluoride concentration is higher than the suggested limit.

5. **Cleaning solid sampler cups**

   For U.S. EPA Method 1621, the recommended cleaning process is soaking and scrubbing the sample cups with methanol followed by rinsing with DI water per U.S. EPA Method 1621 Section 4.2.2. To further minimize PFAS contamination, use UHPLC-MS grade methanol. To minimize potential devitrification and extend their usable life, allow the cups to air dry before placing them in the muffle furnace. Bake in the muffle furnace for 1 hour at 700 °C. After the cups are cool enough to remove, store the clean cups in a desiccator. Alternatively, two cycles through the Cindion C-IC system typically removes fluoride contamination below the U.S. EPA Method 1621 baseline contamination level. However, combustion cups that previously contained fluoride standard solutions should be cleaned in the muffle furnace to extend their life.

6. **Cleaning the Cindion C-IC liquid sampler**

   The Cindion C-IC liquid sampler can be impacted by additional sources of contamination than the solid sampler: solvent, solvent containers, quartz wool, and sample vials and caps. For analysis of trace PFAS, always use UHPLC-MS grade solvents.

   Quartz wool is required in the liquids boat. The quartz wool readily adsorbs PFAS contamination; therefore, the initial furnace cleaning process with the liquids boat will require more time through the furnace. Set up a sequence according to the “Creating Chromeleon Instrument sequences” section to run overnight with the liquids handler, liquids tray, and liquids boat. Run the sequence without vials in the tray.

   Sample vials and vial caps can be a contamination source and potentially cross-contaminate samples, but likely only impact analysis when analyzing low concentrations, such as µg/L and lower concentrations. For PFAS-related samples, use caps without septa and vials without potential fluorocarbon contamination. HDPE vials and caps are recommended for storing PFAS standards.

7. **Cleaning the offline Cindion C-IC adsorption module**

   The adsorption module is used to concentrate AOF or adsorbable organic halides onto GAC columns. The resultant carbon is analyzed by C-IC. To achieve baseline contamination levels needed for AOF applications such as U.S. EPA Method 1621 and other low µg/L concentration applications, it is critical to clean the flow path in the adsorption module before use. For mg/L concentration AOX methods, it is less critical, but advantageous, to clean the adsorption module.

   Prepare 1% ammonium hydroxide (v/v) in methanol according to U.S. EPA Method 1621 Section 7.1.2. Wash the sample containers and rinse vials at least 3 times with the 1% ammonium hydroxide in methanol followed by at least three rinses with DI water. Prepare 2 M sodium nitrate and 10 mM sodium nitrate according to U.S. EPA Method 1621 Section 7.1.4. Prepare 10% methanol in DI water necessary for the solvent rinse step by the Cindion C-IC adsorption system.

   Program the conditions in Table 2 into the adsorption module. For example, to program the Sample, press the Sample button (Figure 9) and Reset Defaults (Figure 10) buttons. Select the up and down arrows to display the desired conditions. Press Save and press the return symbol by selecting blue arrow at the lower right side of the screen (Figure 10). Program the other adsorption steps and clean steps in a similar way. Record conditions for each step as a reference. Table 2 shows representative depth and position conditions for each stage.

Table 2. U.S. EPA Method 1621 adsorption instrument conditions

	Reagent	Volume (mL)	Depth*
Adsorption steps at 2.9 mL/min			
Sample	Aqueous	100-120, Set volume to 140	130
Sodium nitrate rinse	10 mM sodium nitrate	25	120
DI water rinse	DI water	20	120
Clean steps at 10 mL/min			
DI water	DI water	40	120
Solvent	10% methanol in DI water	40	120
DI water	DI water	40	120

*Arbitrary units

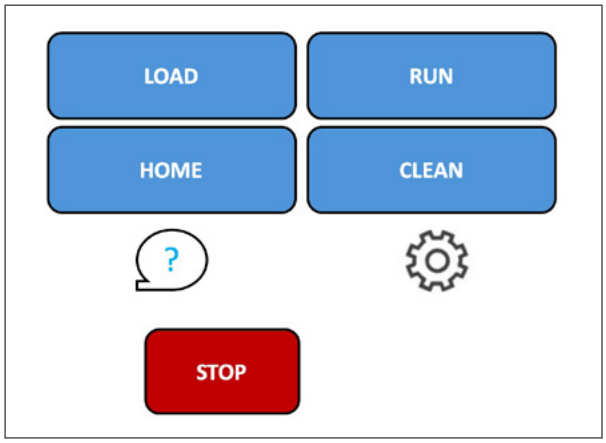


Figure 9A. Cindion C-IC adsorption module: Touch screen displaying instrument status

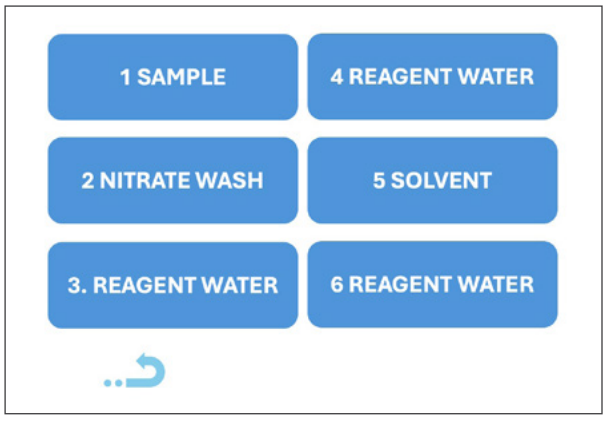


Figure 9B. Cindion C-IC adsorption module: Touch screen displaying program buttons

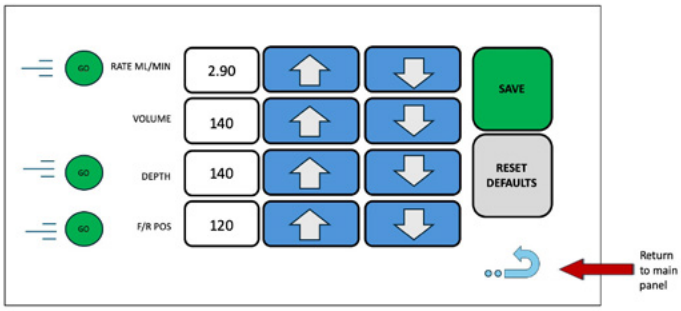


Figure 10. Programming the conditions

To clean the new adsorption module and the GAC holder cartridges, install empty GAC holder cartridges in the six channels and fill the 50 mL DI water and solvent rinse vials. Run the previously programmed “CLEAN” routine three times from the touch screen.

To assess the baseline contamination of the Cindion C-IC adsorption module, wait until cleanliness is established on the C-IC system. The baseline cleanliness is determined by establishing the Method Blanks on each channel. Clean any tools and sample bottles with methanol followed by DI water. Add 100 mL of DI water and 0.5 mL 2 M sodium nitrate to the sample bottle. Install two GAC columns in the GAC carriers and install the GAC carriers to the adsorption module. Pump the sample onto the GAC columns, rinse with 25 mL 10 mM sodium nitrate, and 20 mL DI water. Analyze the GAC columns by C-IC. The results should be 3 times smaller than your lowest calibration standard to be considered clean. Repeat the cleaning process after each sample set or when needed.

Instrument control

1. **Controlling the Cindion combustion/absorption module**

   The main ePanel has controls for temperature, gas flow, manual cleaning routines for the absorber module, and a manual command to return the cup to the solid sampler tray. Additionally, peristaltic pump activity and boat position are displayed; the peristaltic pump is “on” during combustion to pump hydration water. The boat position is displayed as zero when at the home position and the distance in mm from the boat station during combustion.

   However, it is important to note that the temperature and gas flows are critical for complete combustion. For the best results, it is advisable to periodically and gently verify that the seal of the boat guiding tube to the boat station is tight. Gently try to tighten the boat station knob. In addition, periodically verify that the solid sampler gripper is operating within alignment. The solid gripper should pick up and place the cup correctly without movement or out of position and should deliver the combustion cup to the boat exactly in the center of the boat “paddle”. Periodically verify the exact movements of the solid gripper.

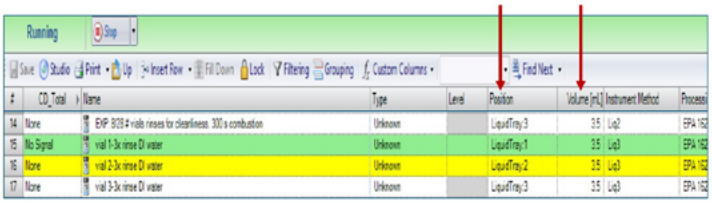


Figure 11. Chromeleon sequence using Cindion C-IC system with the liquid sampler

2. **Controlling the Cindion C-IC adsorption module**

   The adsorption module has these functions that are controlled by the touchpad: HOME, LOAD TRAY, RUN, and CLEAN. The HOME command brings the tray to the back of the instrument. The LOAD TRAY command brings the tray to the front so that samples and vial solutions can be loaded. The RUN command pumps the sample onto the GAC column holders containing the GAC columns, followed by a sodium nitrate rinse, DI water rinse, and 5 mL purge as a drying step. The CLEAN command brings the tray closer to the front, to rinse with the first DI water rinse, then rinse with solvent and lastly, the second DI water rinse. It is recommended to install empty column holders so that they are included in the cleaning steps. Two full cleaning cycles are recommended to achieve cleanliness needed for µg/L analysis levels. For detailed instructions, please see the Cindion C-IC system user manual.

3. **Creating Chromeleon CDS Instrument sequences**

   Use the Chromeleon Wizard to create a new sequence. The C-IC sequence is the same as other IC sequences, with minor exceptions: the Position column (Figure 11, first arrow) reflects the presence of the liquid or solid sample tray indicating the vial position; the Volume column (Figure 11, second arrow) is the absorption volume in mL. For example, in a solid sampler sequence, the Position is “SolidTray#, where # is the cup tray position number. The injection sample loop volume is not indicated in the sequence.

4. **Creating Chromeleon CDS instrument methods**

   Use the Chromeleon Wizard to create an instrument method by selecting new method from the instrument method drop-down menu in the sequence. Enter the combustion, absorber, and boat program parameters for the solid or liquid sampler into the instrument method (Table 1 and Table 3). For both liquid and solid samplers, the valve must remain in the inject position for 360 seconds after injection to allow the Cindion C-IC absorber syringe to wash the flow path.

Table 3. Absorber conditions for U.S. EPA Method 1621

Absorber settings	
Backflush volume (mL)	3
Rinse syringe volume (mL)	10
Rinse syringe cycles	1
Rinse pathway volume (mL)	5.0
Rinse pathway cycles	2
Rinse vial volume (mL)	7
Rinse vial cycles	2
Rinse transfer time	10

Table 4. Additional liquid sampler conditions

Settings	
Rinse cycles	70
Rinse volume (µL)	250
Decompress	Mark box
Wash cycles	1
Wash volume (µL)	200
Pump cycles	4
Sample volume (µL)	200
Pump volume (µL)	250
Sample fill dwell time (s)	3
Inject volume (µL)	20

If the liquid sampler is installed, enter additional parameters into the instrument method as shown in Table 4. The Decompress function pressurizes the vial before withdrawing the sample to minimize a void volume or a volume variation due to the vacuum created when withdrawing large volumes of sample. Enter the IC conditions for your application. Check and save the method and apply the method to the Chromeleon sequence. A couple of terms for the liquid sampler are different than IC instruments. The “Inject Volume” is the flush volume or what the IC autosamplers call the “cut volume”. The “Sample Volume” is the volume injected into the Cindion C-IC liquids boat.

5. **Creating Chromeleon processing methods**

   The Chromeleon processing method for C-IC is the same as for other IC methods. However, the EPA has implemented the use of relative standard error (RSE) for determining and selecting which calibration curve has the best fit for your data. For more information, refer to previous applications and technical notes.[12,13,25,26]

Conclusion

This technical note provides comprehensive guidance on configuring and operating the full Cindion C-IC system workflow. The system offers a complete workflow, starting from offline sample preparation for AOX analysis, progressing through combustion and absorption, and concluding with IC analysis. It includes an autosampler for solids and liquids, along with an optional module for compressed and liquefied gases, ensuring versatility in sample handling. The Z-fold combustion tube is designed to introduce oxygen at multiple points and create an extended flow path that enhances combustion efficiency while minimizing length. To minimize devitrification, the system introduces water only during combustion. Additionally, Chromeleon CDS seamlessly controls Cindion C-IC system operation and data processing.

References

1. Huang, B. and Rohrer, J. Application Note 72573 Determination of halogens in polymers and electronics using a combustion ion chromatography system. 2017. Thermo Fisher Scientific, Sunnyvale, CA, USA.
2. Aggrawal, M. and Rohrer, J. Application Note 72349 Determination of chlorine, bromine, and sulfur in polyethylene materials using combustion ion chromatography. 2017. Thermo Fisher Scientific, Sunnyvale, CA, USA.
3. Aggrawal, M. and Rohrer, J. Application Update 72588 Determination of chlorine, bromine, and sulfur in polyethylene materials using combustion IC with a carbonate/bicarbonate eluent. 2018. Thermo Fisher Scientific, Sunnyvale, CA, USA.
4. Hu, J. and Rohrer, J. Application Note 73280 Determination of fluorine and chlorine in iron ore using combustion ion chromatography. 2019. Thermo Fisher Scientific, Sunnyvale, CA, USA.
5. Christison, T. and Rohrer, J. Application Note 73865 Pyrohydrolytic combustion ion chromatography: Determination of total chlorine and sulfur in cleanroom gloves, 2020. Thermo Fisher Scientific, Sunnyvale, CA, USA.
6. Yang, H., Khor, D., and Rohrer, J. Application Note 72268 Determination of fluoride in tea using a combustion ion chromatography system. 2017. Thermo Fisher Scientific, Sunnyvale, CA, USA.
7. Hu, J. and Rohrer, J. Application Note 72333 Determination of Adsorbable Organic Halogen in Wastewater using a Combustion Ion Chromatography System. 2017. Thermo Fisher Scientific, Sunnyvale, CA, USA.
8. Neist, U., Klocke, I., Georgii, S. PhD., Brunn, H., Prof. PhD., and Jensen, D. Customer Application Note CAN 73481 AOF by combustion IC – non-targeted complemental determination of PFAS in aqueous samples. 2020. Hessian State Laboratory, Wiesbaden, Germany and Thermo Fisher Scientific, GmbH, Dreieich, Germany.
9. Jones, J.L., Burket, S.R., Hanley, A., Shoemaker, J.A. Development of a standardized adsorbable organofluorine screening method for wastewaters with detection by combustion ion chromatography. Analyt. Methods, 2022, 36.
10. U.S. EPA Environmental Protection Agency, Office of Water. Report on the Single-laboratory Validation of Clean Water Act Method 1621 for Adsorbable Organic Fluoride (AOF). EPA 820-R-22-003. April 2022.
11. U.S. Environmental Protection Agency (EPA). Multi-laboratory Validation Study Report for Method 1621: Determination of Adsorbable Organic Fluorine (AOF) in Aqueous Matrices by Combustion Ion Chromatography (C-IC), EPA 821-R-24-003. U.S. Environmental Protection Agency, Office of Water (4303T), Office of Science and Technology Engineering and Analysis Division, Washington, DC, USA, January 2024.
12. U.S. Environmental Protection Agency (EPA). Method 1621 Determination of Adsorbable Organic Fluorine (AOF) in Aqueous Matrices by Combustion Ion Chromatography (C-IC), EPA 821-R-24-002. U.S. Environmental Protection Agency, Office of Water (4303T), Office of Science and Technology Engineering and Analysis Division, Washington, DC, USA, January 2024.
13. Christison, T. and Rumachik, N. Application Note AN007428: Results from EPA draft Method 1621 Collaboration Study: Screening of PFAS compounds in wastewater using adsorbable organic fluorine with combustion ion chromatography (C-IC). Thermo Fisher Scientific, Sunnyvale, CA, USA, 2024.
14. Christison, T. and Rumachik, N. Technical Note TN003056: Techniques for successful implementation of EPA Method 1621: Screening of PFAS compounds using adsorbable organic fluorine with combustion IC. Thermo Fisher Scientific, Sunnyvale, CA, USA, 2024.
15. Christison, T. and Rumachik, N. Application Note AN003774: Enhanced screening of PFAS compounds in wastewater: Implementing U.S. EPA Method 1621 with improved combustion ion chromatography. Thermo Fisher Scientific, Sunnyvale, CA, USA, 2025.
16. Thermo Fisher Scientific: Dionex Inuvion Ion Chromatography System Operating Manual, 2025
17. Thermo Fisher Scientific: Suppressor manual
18. Thermo Fisher Scientific: Dionex IonPac AS24 column product manual.
19. Thermo Fisher Scientific: Eluent generator cartridge manual.
20. Thermo Fisher Scientific: Cindion Combustion System Operating Manual, 2025.
21. Thermo Fisher Scientific: Cindion Adsorption system operating manual, 2025.
22. Hu, Jingli and Rumachik, N. Technical Note TN003853: Configuring the Thermo Scientific™ Cindion™ C-IC system for a 2-in-1 operation: seamless switching between Combustion-IC and Standalone IC with an AS-AP Autosampler, Thermo Fisher Scientific, Sunnyvale, CA, USA, 2025.
23. Thermo Fisher Scientific: Thermo Scientific Cindion Combustion Ion Chromatography (C-IC) System: Solids Autosampler Configuration Setup.
24. Thermo Fisher Scientific: Thermo Scientific Cindion Combustion Ion Chromatography (C-IC) System: Liquids Autosampler Configuration Setup.
25. Jensen, D. Technical Note TN003551 Enhanced calibration precision: Leveraging RSE and WLS for optimal function optimization. Thermo Fisher Scientific GmbH, Dreieich, Germany, 2025.
26. Jensen, D. Technical Note TN003643: A short guide towards enhanced calibration precision, Thermo Fisher Scientific GmbH, Dreieich, Germany, 2025.

Learn more at **thermofisher.com/cindion**