IC-MS

Installation Guide for customers using IC-MS, IC-MS/MS, or IC-HRAM MS

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Installation Guide

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Legal Manufacturer Address

Thermo Fisher Scientific
527 Lakeside Drive
Sunnyvale, California 90485
United States

Contents

Contacting Us 4

1 Using this Manual 1

1.1 About this Manual 2

1.2 Conventions 3

1.2.1 Safety Messages 3

1.2.2 Special Notices and Informational Notes 3

1.2.3 Typographical Conventions 4

1.3 Reference Documentation 5

2 Safety 7

2.1 Safety Symbols and Signal Words 8

2.1.1 Safety Symbols and Signal Words in This Manual 8

2.1.2 Observing this Manual 9

2.1.3 Safety Symbols on the Detector 9

2.2 Intended Use 11

2.3 Safety Precautions 12

2.3.1 General Safety Information 12

2.3.2 Qualification of the Personnel 12

2.3.3 Personal Protective Equipment 13

2.3.4 Electrical Safety Precautions 14

2.3.5 General Residual Hazards 14

2.4 Solvent and Additive Information 18

2.5 Compliance Information 19

3.0 Instrument Overview 20

3.1 Mass Spectrometer Operating Principle 21

3.2 Overview of an IC/MS Analysis 23

3.3 Software Operation 25

3.3.1 Thermo Scientific ISQ EC single quadrupole mass spectrometer using Chromeleon CDS software 25

3.3.2 Thermo Scientific TSQ Triple Quadrupole mass spectrometers using Chromeleon CDS software 25

3.3.3 Thermo Scientific TSQ Triple quadrupole mass spectrometer using SII for Xcalibur software 26
3.3.4 Thermo Scientific Q Exactive Hybrid Orbitrap-Quadrupole mass spectrometers using SII for Xcalibur software 26
3.4 Two software (Chromeleon + Xcalibur) Installation 27
3.4.1 Case 1: Anions + Cations + HPLC with a single MS and single computer 27
3.4.2 Case 2: IC and LC with MS and independently using two computers 27
3.4.3 Case 3, Automatic switching from anions to cations for IC-MS 28
4.0 Installation 29
4.1. Positioning the IC system to the MS system 29
4.2 Components of the IC-MS Installation Kit (P/N 22153-62049) 29
4.3 Installing Contact Closure (TTL) cables from Dionex IC AS-AP Autosampler to MS system using SII for Xcalibur 29
5.0 Critical Items for Using Ion Chromatography with MS detection or Electrochemical-MS Detection 32
5.1 Critical Items for Using Suppressed Conductivity-Mass Spectrometry 32
5.2 Critical Items for Using Electrochemical Detection (ED) with Mass Spectrometry Detection 34
6.0 Electrolytic Devices 37
6.1 Backpressure limits for electrolytic devices 37
6.2 IC PEEK Tubing 38
6.3 Selecting Electrolytic Suppressor or Electrolytic Regenerated Desalter Devices 40
6.4 Selecting Eluent Generation Consumables 41
7.0 Conditioning Consumables 42
7.1 Conditioning Eluent Generator Cartridges and CR-TC trap columns 42
7.2 Conditioning Columns 42
7.3 Conditioning Suppressors and ERD desalting devices. 42
8.0 AXP and AXP-MS Auxiliary Pumps 43
8.1 Selecting AXP and AXP-MS pumps 43
8.2 AXP pump for Suppressor Regenerant 44
8.3 AXP-MS Pump: Recommended for Make-up Flow, and Carrier for ISQ EC/EM Calibrant flow 45

9.0 Electronic configuration 47
9.1 IC-MS Software 47
9.2 Finding the COM port for the AXP and AXP-MS pumps 47
9.3 Chromeleon: IC-MS system using a Dionex Integrion HPIC system coupled to Thermo Scientific ISQ EC mass spectrometer 48
9.4 Chromeleon: IC-MS system with Dionex Integrion coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer 51
9.5 Chromeleon: IC-MS system with Dionex ICS-5000+ or ICS-6000 coupled with Thermo Scientific ISQ EC mass spectrometer 51
9.6 Chromeleon: IC-MS system with Dionex ICS-5000+ or ICS-6000 HPIC System coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer 53
9.7 SII for Xcalibur: IC-MS system with Dionex Integrion coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer or Thermo Scientific Hybrid Orbitrap-Quadrupole High Resolution Mass Spectrometer 53
9.8 SII for Xcalibur: IC-MS system with Dionex ICS-5000+ or ICS-6000 HPIC System coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer or Thermo Scientific Hybrid Orbitrap-Quadrupole High Resolution Mass Spectrometer 55
9.9 Two-Software (Chromeleon + Xcalibur) Installation 56
9.9.1 Case 1: Anion + Cation + HPLC with a single MS and single computer 56
9.9.2 Case 2: IC and LC with MS and independently using two computers 56
9.9.3 Case 3: Automatic Switching to determine anions and cations by IC-MS 57
10. Using the Diverter Valve 59
10.1 Programming the Diverter valve to Direct IC Flow to MS 59
10.2 Programming the Diverter valve to Direct IC Flow to Waste or Recycle 62
11. Triggers, Emergency Methods and TTL connections 64
11.1 TTL Settings 64
11.2 Using the AXP Low Pressure Error Trigger 64
11.3 Using A High Conductivity Trigger 65
11.4 Creating a Virtual Channel to monitor suppressors 70
12. Start Up and Shutdown Practices 73
12.1 Start and condition IC system independently from the MS system 74
12.2 Good start-up practices for IC-MS systems 75
12.3 Good shutdown practices for IC-MS systems 76
13. Triggering Contact Closure 77

13.1 Using Chromeleon software to trigger contact closure 78
13.2 Using SII for Xcalibur software to trigger contact closure 79
13.3 Activating the Contact Closure Trigger in SII for Xcalibur 80
13.4 Two software (Chromeleon + Xcalibur) Installation 82

Appendix I: General Decision Chart for System Safety Operation 83

RUN mode (Flow to the MS) 86
IC measurements with MS 86
IC only measurement with suppressor and/or CR-TC, but no recycle mode possible 86
IC only measurement without suppressor and/or CR-TC, no recycle mode 86
IC only measurement with Recycle Mode 87
IDLE mode (Flow diverted away from MS) 87
IDLE system without Recycle Mode, at least one electrolytical device needed 88
IDLE system without Recycle Mode, no Electrolytical devices needed 88

Appendix II. Configuration Schematics 89

IC-MS with EGC and CD: 89
IC-MS with EGC and CD, with external regeneration of suppressor by default 92
IC-MS with Carbohydrates: 94
MS-detection only (no PAD), Manually prepared Eluents (ICS-5000, ICS-6000 only) 95
IC-MS for Carbohydrates: MS-detection with PAD, Manually prepared Eluents (ICS-5000, ICS-6000 only) 96
IC-MS for Carbohydrates: MS-detection with PAD, RFIC-generated Eluents 98

1 Using this Manual

This chapter provides information about this manual, the conventions used throughout the manual, and the reference documentation that is available in addition to this manual.

1.1 About this Manual

This manual provides information about customers using IC-MS on Thermo Scientific ion chromatography (IC) systems coupled to Thermo Scientific mass spectrometry (MS) systems.

The layout of this manual is designed to provide quick reference to the sections of interest to the user. To obtain a full understanding of your detector, read this manual thoroughly.

This manual also contains safety messages, precautionary statements, and special notices that can prevent personal injury, damage to the detector, or loss of data when followed properly.

Note the following:

- The detector configuration may vary; therefore, not all descriptions necessarily apply to your particular detector.
- If some detail applies to only one model or variant, the model or variant is identified by name.
- Illustrations in this manual are provided for basic understanding. They can vary from the actual model of the detector or component. However, this does not influence the descriptions. No claims can be derived from the illustrations in this manual.

1.2 Conventions

This section describes the conventions that are used throughout this manual.

1.2.1 Safety Messages

The safety messages and precautionary statements in this manual appear as follows:

- Safety messages or precautionary statements that apply to the entire manual and all procedures in this manual are grouped in the Safety chapter.
- Safety messages or precautionary statements that apply to an entire section or to multiple procedures in a section appear at the beginning of the section to which they apply.
- Safety messages that apply to only a particular section or procedure appear in the section or procedure to which they apply. They appear different from the main flow of text.

Safety messages are often preceded by an alert symbol and/or alert word. The alert word appears in uppercase letters and in bold type.

Make sure that you understand and follow all safety messages presented in this manual.

1.2.2 Special Notices and Informational Notes

Special notices and informational notes in this manual appear different from the main flow of text. They appear in boxes and a note label identifies them. The label text appears in uppercase letters and in bold type.

WARNING Indicates that failure to take note of the accompanying information may result in personal injury.

NOTICE Indicates that failure to take note of the accompanying information could cause wrong results or may result in damage to the device.

TIP Highlights information of general interest or helpful information that can make a task easier or optimize the performance of the detector.

1.2.3 Typographical Conventions

These typographical conventions apply to the descriptions in this manual:

Data Input and Output

- The following appears in **bold** type:
  - Input that you enter by the keyboard or that you select with the mouse
  - Buttons that you click on the screen
  - Commands that you enter by the keyboard
  - Names of, for example, dialog boxes, properties, and parameters
- For brevity, long expressions and paths appear in the condensed form, for example: Click **Start > All Programs > Thermo Chromeleon 7 > Services Manager > Start Instrument Controller**.

References and Messages

- References to additional documentation appear *italicized*.
- Messages that appear on the screen are identified by quotation marks.

Viewpoint

If not otherwise stated, the expressions *left* and *right* in this manual always refer to the viewpoint of a person that is facing the detector from the front.

Particularly Important Words

Particularly important words in the main flow of text appear *italicized*.

Electronic Manual Version (PDF)

The electronic version (PDF) of the manual contains numerous links that you can click to go to other locations within the manual. These include:

- Table of contents entries
- Index entries
- Cross-references (in blue text), for example, to sections and figures

1.3 Reference Documentation

In addition to this operating manual, other documentation is available for reference.

Hardware Documentation

Additional hardware documentation includes the following:

Operating manuals for the other modules of the IC system

- *Instrument Installation Qualification Operating Instructions*

TIP Electronic versions of these manuals are available as PDF (Portable Document Format) files. To open and read the PDF files, Adobe™ Reader™ or Adobe™ Acrobat™ is required.

Software Documentation

Additional software documentation includes the following:

- *Chromeleon™ 7 Help and documents*
  The *Chromeleon 7 Help* provides extensive information and comprehensive reference material for all aspects of the software. For basic information about device installation and configuration, refer to the *Installation Guide*; for specific information about a certain device, refer to the *Instrument Configuration Manager Help.* In Chromeleon 7, devices are called modules.

  For information about the main elements of the user interface and step-by-step guidance through the most important workflows, refer to the *Quick Start Guide*.

  For a concise overview of the most important workflows, refer to the *Reference Card*.

- *Chromeleon 7 Help*
  The *Chromeleon 7 Help* provides extensive information for all aspects of the software, including device installation and configuration.

TIP The *Chromeleon* Help and documents are included in the software shipment.

Third-Party Documentation

Refer also to the user documentation provided by the manufacturers of third-party components and materials, for example, Safety Data Sheets (SDSs).

Third-party documentation includes the user documentation for the gas supply, as applicable:

- User documentation for the nitrogen generator
- User documentation for the Dionex AXP and AXP-MS Auxiliary pumps are provided by Thermo Scientific Dionex brands.
- User documentation for the mechanical or roughing pump.

2 Safety

This chapter provides general and specific safety information and informs about the intended use of the detector.

2.1 Safety Symbols and Signal Words

2.1.1 Safety Symbols and Signal Words in This Manual

This manual contains safety messages to prevent injury of the persons using the detector. The safety symbols and signal words in this manual include the following:

Always be aware of the safety information. Do not proceed until you have fully understood the information and consider the consequences of what you are doing.

CAUTION Indicates a hazardous situation that, if not avoided, could result in minor or moderate injury.

WARNING Indicates a hazardous situation that, if not avoided, could result in serious injury.

2.1.2 Observing this Manual

Observe the following:

- Before installing or operating the detector, read this manual and the detector manual carefully to be familiar with the detector and this manual. The manual contains important information with regard to user safety as well as use and care of the detector.
- Always keep the manual near the detector for quick reference.
- Save this manual and pass it on to any subsequent user.

Read, understand, and comply with all safety messages and precautionary statements presented in this manual.

2.1.3 Safety Symbols on the Detector

The table lists the safety symbols that appear on the detector or on labels affixed to the detector. Follow the safety notices in this manual to prevent the risk of operator injury or damage to the detector.

Symbol	Description
	Indicates a potential hazard. Refer to this manual to avoid the risk of personal injury and/or to prevent damage to the device.
I O	Power supply is on Power supply is off
~	Indicates alternating current.
	Indicates a terminal for protective grounding.
	Indicates that an electrical shock *could* or *might* occur.
	Alerts you to the presence of a hot surface that *could* or *might* cause burn injuries.
	Indicates that eye damage *could* or *might* occur.

Symbol	Description
	Alerts you to carefully read your instrument's documentation to ensure your safety and the instrument's operation ability. Failing to carefully read the documentation *could* or *might* put you at risk for a physical injury.
	Indicates that physical injury *could* or *might* occur if two or more people do not lift an object.

2.2 Intended Use

The instrument is intended for use in ion chromatography (IC) mass spectrometry (MS) applications. The system is for use by qualified personnel and in laboratory environments only.

The instrument is intended for laboratory research use only. It is not intended for use in diagnostic procedures.

Laboratory Practice

Thermo Fisher Scientific recommends that the laboratory in which the MS system is used follow best practices for IC-MS analyses. This includes among others:

- Using appropriate standards
- Regularly running calibration
- Establishing shelf life limits and following them for all consumables used with the system
- Running the system according to the laboratory's verified and validated 'lab developed test' protocol

2.3 Safety Precautions

2.3.1 General Safety Information

All users must observe the general safety information presented in this section and all specific safety messages and precautionary statements elsewhere in this manual during all phases of installation, operation, troubleshooting, maintenance, shutdown, and transport of the detector.

If the instrument is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the detector could be impaired. Observe the following:

- Operate the instrument only within its technical specifications.
- Use only the replacement parts and additional components, options, and peripherals specifically authorized and qualified for the detector by Thermo Fisher Scientific.
- Perform only the procedures that are described in this operating manual and in supporting documents for the detector.
  Follow all instructions step-by-step and use the tools recommended for the procedure.
- Open the enclosure of the detector and other components only if specifically instructed to do so in this manual.
- Thermo Fisher Scientific cannot be held liable for any damage, material or otherwise, resulting from inappropriate or improper use of the detector. If there is any question regarding appropriate usage, contact Thermo Fisher Scientific before proceeding.

Safety Standard

The Mass Spectrometers are a Safety Class I instrument (provided with terminal for protective grounding). The Thermo Scientific IC and MS systems have been manufactured and tested according to international safety standards.

2.3.2 Qualification of the Personnel

Observe the information below on the proper qualification of the personnel installing and/or operating the detector.

Installation

Only skilled personnel are permitted to install the detector and to establish the electrical connections according to the appropriate regulations. Thermo Fisher Scientific recommends always having service personnel certified by Thermo Fisher Scientific perform the installation (for brevity, referred to as Thermo Fisher Scientific service engineer).

If a person other than a Thermo Fisher Scientific service engineer installs and sets up the module, the installer is responsible for ensuring the safety of the module and system and the warranty may be voided.

General Operation

The detector is designed to be operated only by trained and qualified personnel in a laboratory environment.

All users must know the hazards presented by the detector and the substances they are using. All users should observe the related Safety Data Sheets (SDSs).

2.3.3 Personal Protective Equipment

Wear personal protective equipment and follow good laboratory practice to protect you from hazardous substances. The appropriate equipment depends on the hazard. For advice on the hazards and the equipment required for the substances you are using, refer to the material handling and safety data sheet provided by the vendor.

An eyewash facility and a sink should be available nearby. If any substance contacts your skin or eyes, wash the affected area and seek medical attention.

Protective Clothing

To protect you from chemical splashes, harmful liquids, or other contamination, when performing any work on or near the IC-MS system put on appropriate protective clothing, such as a lab coat. For information about the proper handling of a particular substance and for advice on specific hazards, refer to the safety data sheet (SDS) for the substance you are using.

Protective Eyewear

To prevent liquids from striking your eyes, put on appropriate protective eyewear, such as safety glasses with side shields. If there is a risk of splashing liquids, put on goggles or use a face shield or work in the exhaust hood.

Gloves

To protect you from harmful liquids and avoid personal injury during maintenance or service, put on appropriate protective gloves. Consult a chemical compatibility chart to select the appropriate glove for the chemical.

2.3.4 Electrical Safety Precautions

WARNING— Electric Shock or Damage to the Device

High voltages are present inside the device that could cause an electric shock or damage to the device.

- Do not make any changes to the electrical or grounding connections.
- If you suspect any kind of electrical damage, disconnect the power cord and contact Thermo Fisher Scientific Technical Support for assistance.
- Do not open the housing or remove protective panels unless specifically instructed to do so in this manual.
- Do not place liquid reservoirs directly upon the device. Liquid might leak into the device and get into contact with electronic components causing a short circuit. Instead, place liquid reservoirs in the eluent/solvent rack that is available for the IC system.

2.3.5 General Residual Hazards

Pay attention to the following general residual hazards when working with the instrument:

WARNING—Hazardous Substances

Many organic solvents, eluents (mobile phases), and samples are harmful to health. Be sure that you know the toxic and infectious properties of all substances that you are using. You may not know the toxic or infectious properties of many substances that you are using. If you have any doubt about a substance, treat it as if it contains a potentially harmful substance. For advice on the proper handling of a particular substance, refer to the Safety Data Sheet (SDS) of the manufacturer. Observe the guidelines of Good Laboratory Practice (GLP).

- Be sure that you know the properties of all substances that you are using. Avoid exposure to harmful substances. If you have any doubt about a substance, handle the substance as if it is potentially harmful.
- Wear personal protective equipment as required by the hazard and follow good laboratory practice.
- Reduce the volume of substances to the minimum volume required for sample analysis.
- Do not operate the detector in a potentially flammable environment.
- Avoid accumulation of harmful substances. Make sure that the installation site is well ventilated.
- Dispose of hazardous waste in an environmentally safe manner that is consistent with local regulations. Follow a regulated, approved waste disposal program.

WARNING—Biohazard

Biohazardous material, for example microorganisms, cell cultures, tissues, body fluids, and other biological agents can transmit infectious diseases. To avoid infections with these agents:

- Assume that all biological substances are at least potentially infectious.
- Wear personal protective equipment as required by the hazard and follow good laboratory practice.
- Dispose of biohazardous waste in an environmentally safe manner that is consistent with local regulations. Follow a regulated, approved waste disposal program.

WARNING—Self-Ignition of Solvents

Solvents with a self-ignition temperature below 150°C might ignite when in contact with a hot surface (for example, due to leakage in the chromatography system).

WARNING—Hazardous Vapors

Install the IC-MS system in a well-ventilated laboratory. If the eluent or sample includes volatile or flammable solvents, do not allow them to enter the workspace. If the mobile phase or sample includes volatile or flammable solvents, avoid open flames and sparks.

- The exhaust gas may contain hazardous fumes. To avoid an accumulation of the exhaust gasses, ensure that the exhaust gas is absorbed by a fume hood or other ventilating device. Maintain a well-ventilated laboratory. Do not vent directly into the laboratory.
- Avoid open flames and sparks.

CAUTION—Escape of Hazardous Substance from PEEK Capillaries

In the IC system, capillaries made of PEEK are used.

- Certain chemicals, for example, trichloromethane ($CHCl_3$), dimethyl sulfoxide (DMSO), or tetrahydrofuran (THF) may be incompatible with IC consumables, such as columns, eluent generator cartridges, and suppressors.
- For more information, refer to the product manuals of the application columns, suppressors, and eluent generator cartridges to confirm chemical compatibility

CAUTION—Sparking due to Electrostatic Discharge

Always use the grounding coupler with the MS, located on the MS source. Liquid flowing through capillaries can generate static electricity. This effect is particularly present with insulating capillaries and non-conductive solvents (for example, pure acetonitrile). Discharge of electrostatic energy might lead to sparking, which could constitute a fire hazard. Prevent the generation of static electricity near the chromatography system.

2.4 Solvent and Additive Information

To protect optimal functionality of the instrument, observe these recommendations on the use of solvents and additives:

- Use only solvents and additives that are compatible with all parts in the flow path.
- Follow any specific recommendations presented in other sections of this manual. Refer also to the *operating manuals for all modules in the IC system*. They may provide additional guidelines and information.

2.5 Compliance Information

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations. When the device is delivered to you, it meets all pertinent electromagnetic compatibility (EMC) and safety standards as described in this manual.

Changes that you make to the device may void compliance with one or more of these EMC and safety standards. Changes to the device include replacing a part or adding components, options, or peripherals not specifically authorized and qualified for the product by Thermo Fisher Scientific. To ensure continued compliance with EMC and safety standards, replacement parts and additional components, options, and peripherals must be ordered from Thermo Fisher Scientific or one of its authorized representatives.

The device has been shipped from the manufacturing site in a safe condition.

3.0 Instrument Overview

This chapter introduces you to the mass spectrometer and the main components.

3.1 Mass Spectrometer Operating Principle

IC-MS technologies can provide sensitive and selective detection of ionic species. Interfacing Ion Chromatography (IC) and MS provides the separation scientist with one of the most powerful analytical tools available. Both IC and MS have developed to a point whereby they represent two of the most important techniques in characterizing and detecting ionic species.

Mass spectrometry is a very powerful analytical technique used in a number of fields, including:

- Identification of unknown compounds
- Quantitation of known compounds
- Determination of chemical structure

The basic function of an MS detector is to measure the mass-to-charge ratio of ions. The unit of mass used is the Dalton (Da). One Dalton is equal to 1/12 of the mass of a single atom of carbon-12. This follows the accepted convention that an atom of carbon-12 has exactly 12 atomic mass units (amu). The MS detector does not directly measure molecular mass, but rather the mass-to-charge ratio of the ions. Electrical charge is a quantized property and so can exist only as an integer; that is, 1, 2, 3, and so on. The unit of charge used here (z) is that which is on an electron (negative) or a proton (positive). Therefore, the mass-to-charge ratio measured can be denoted by *m/z*. Most ions encountered in mass detection have just one charge after ion chromatography. In this case, the mass-to-charge ratio is often referred to as the "mass" of the ion.

Mass spectrometers currently used for IC-MS are single quadrupole (Thermo Scientific™ ISQ™ EC), tandem (Thermo Scientific™ TSQ™ Altis triple quadrupole MS/MS or Thermo Scientific™ TSQ™ Quantis triple quadrupole MS/MS, and the Thermo Scientific™ TSQ™ Fortis triple quadrupole MS/MS) or hybrid quadrupole-Orbitrap (Thermo Scientific™ Q Exactive™ Series). The degree of insight about the sample identity from the MS data is typically proportional to the **mass accuracy** and the **resolving power** of the mass spectrometer used, which usually are proportional to the price of that instrument.

Peak Confirmation and Identification

Unit-resolution mass spectrometers such as **single quadrupole (SQ)** mass spectrometers provide nominal mass which can provide peak confirmation (as opposed to peak identification). That is, combined with chromatography and expected retention times, the m/z peak is confirmed or refuted as the expected peak identity.

> For example, perchlorate (ClO_4^-) has two isotopologues (*m/z* 99, *m/z* 101) based on the presence of ^{16}oxygen and ^{18}oxygen. If the SQ detects both *m/z* 99 and *m/z* 101 eluting at the same retention time as perchlorate, the SQ confirms that it is perchlorate. If the unknown peak is detected as *m/z* 99 but *m/z* 101 is not detected, then it is not perchlorate.

High-resolution accurate mass (HRAM) mass spectrometers such as Orbitrap, have resolving power >60,000 resulting in a mass accuracy determination to ppm level and thus establish a compound's molecular formula, elemental, and isotopic composition. The exact mass parameter alone does not provide much insight into the molecular structure.

In addition to the unit mass or accurate mass, most tandem and hybrid mass spectrometers can also provide **MS/MS** or **MS^n data**, where a precursor ion is isolated, excited, collided

and/or reacted with a reagent or collision gas, and fragmented. The fragmentation reactions under defined conditions are highly reproducible and are often characteristic of the structure. The resulting fragments (product ions) can be isolated and fragmented again, to produce the MS^2 or MS^3 spectrum, etc. The collection of MS^n data from many or all ions of the compound formed in the ion source, called **MS^n tree**, can serve for very precise structural identification.

Quantitation

The benefit of **quantitation** by mass spectrometry links to the **selectivity at the molecular level**. When used for quantitation, the mass spectrometer as an analytical instrument acts as a **comparator**: it compares the signal of a sample with the signal of a chemical standard to report the absolute mass or concentration of the substance. This is due to differences in ionization efficiencies per compound.

The currently accepted "gold standard" of **targeted quantitation** is by a triple quadrupole MS (often abbreviated as QqQ) coupled to a front-end chromatograph. The «QqQ» denotes two analyzing quadrupoles (denoted by the upper-case Qs) and either a quadrupole (lower-case q) or some other multipole (e.g. hexapole or octapole) collision cell. MS/MS experiments have an increase in selectivity where the first quadrupole analyzer (Q1) selects the precursor ion(s), which collide with inert gas, fragment in the collision cell (q), and the resulting product ions are analyzed in the last quadrupole (Q3). The analyst then selects and monitors the most characteristic and interference-free pair of ions: the precursor ion and the product ion, together called a «**transition**». This transition represents the gas-phase chemical reaction that occurs during the flight of the precursor ion through the mass spectrometer, sometimes also referred to as «**selected reaction monitoring**» (**SRM**) or «**multiple reaction monitoring**» (**MRM**). The terms SRM, MRM, and MS/MS refer to exactly the same process that is responsible for increased selectivity of the triple quadrupole MS at the molecular level.

Quantitation in IC–MS using a SQ is always performed on the *selected* (by the analyst) *m/z* signal(s) or on a linear combination (called **selected ion monitoring** or **SIM) of ~~both~~ isotopologues.** For example, perchlorate has two *m/z* based on monoisotopic and ^{37}Cl isotopes in perchlorate, respectively. The relative abundance of ^{18}O in perchlorate is 4*0.2%=0.8% vs. ~33% ^{37}Cl.

For **untargeted quantitation**, or analysis of unknown compounds (unknowns), we need **high-resolution accurate mass (HRAM)** mass spectrometers that can operate at sufficiently high resolving power (typically 60,000 or above) to identify unknowns, and with sufficiently high speed (typically defined by the width of the chromatographic peaks) in order to perform quantitation with enough data points across the chromatographic peak. It is important to note that HRAM is fully capable of targeted quantitation as well.

3.2 Overview of an IC/MS Analysis

In a typical IC-MS analysis, the ion chromatograph (IC) portion of the system separates the analytes of interest from the sample matrix using ion-exchange chromatography. As most IC-MS applications use Reagent-Free IC™ (RFIC™) to generate the eluent, the IC pump dispenses deionized water (ASTM Type 1 deionized water) to the eluent generator cartridge where the eluent is electrolytically generated inline by the cartridge and Chromeleon instrument method conditions. An isocratic or gradient elution can be created by entering the desired eluent concentration at the set time during the analysis run. The program adjusts the mA of current proportional to the eluent concentration and inversely proportional to the flow rate. The eluent is pumped through the IC column (containing the ion-exchange stationary phase) under high pressure. An autosampler introduces a measured quantity of sample into this eluent stream.

As the eluent stream passes through the IC column, the analytes of interest in their ionic form are separated from the sample matrix. The rate at which the components of the sample elute from the column depends on their relative affinities to the eluent (mobile phase) and to the solid particles with the ion exchange chemistry specific to each column. As the separated ionic analytes exit the IC column, they pass through the IC electrolytic suppressor where the eluent is converted to water and the charged ions are converted to the free-acid form (anion-exchange) or free-base form (cation-exchange). In doing so, provides a higher conductance response by the Conductivity Detector and compatibility plus lower chemical noise for MS detection. The analytes leaving the IC are in the ionized state, mostly as singly charged, and enter the MS using electrospray ionization (ESI) soft ionization to create an ionized gas stream. As the mass spectrometer analyzes the ionized species and determines each mass-to-charge ratio (*m/z*) and ion's intensity as a function of its concentration ~~(counts per min in Chromeleon and counts per second in SII for Xcalibur and Xcalibur~~), it sends a data stream to the data system computer. When the system setup includes a syringe pump and divert/inject valve, there are three additional ways to introduce a sample into the mass spectrometer, as described in Table 1.

Method	Description
Direct infusion	Connect a syringe pump directly to the ESI source of the mass spectrometer.
High-flow infusion	Use a union tee to combine the flow from a syringe pump with the flow from an IC pump.
Manual loop injection	Connect a sample loop, a disposable plastic syringe with green PEEK (0.03 in i.d., 0.76 mm i.d.) tubing to connect in sampler position, and an IC pump to an external divert/inject valve. After you fill the sample loop with sample, switch the position of the divert/inject valve, which places the contents of the sample loop in the path of the eluent flow produced by the IC pump.

Table 1: Methods of sample introduction into the mass spectrometer

See Figure 1 for an overview of an IC-MS system

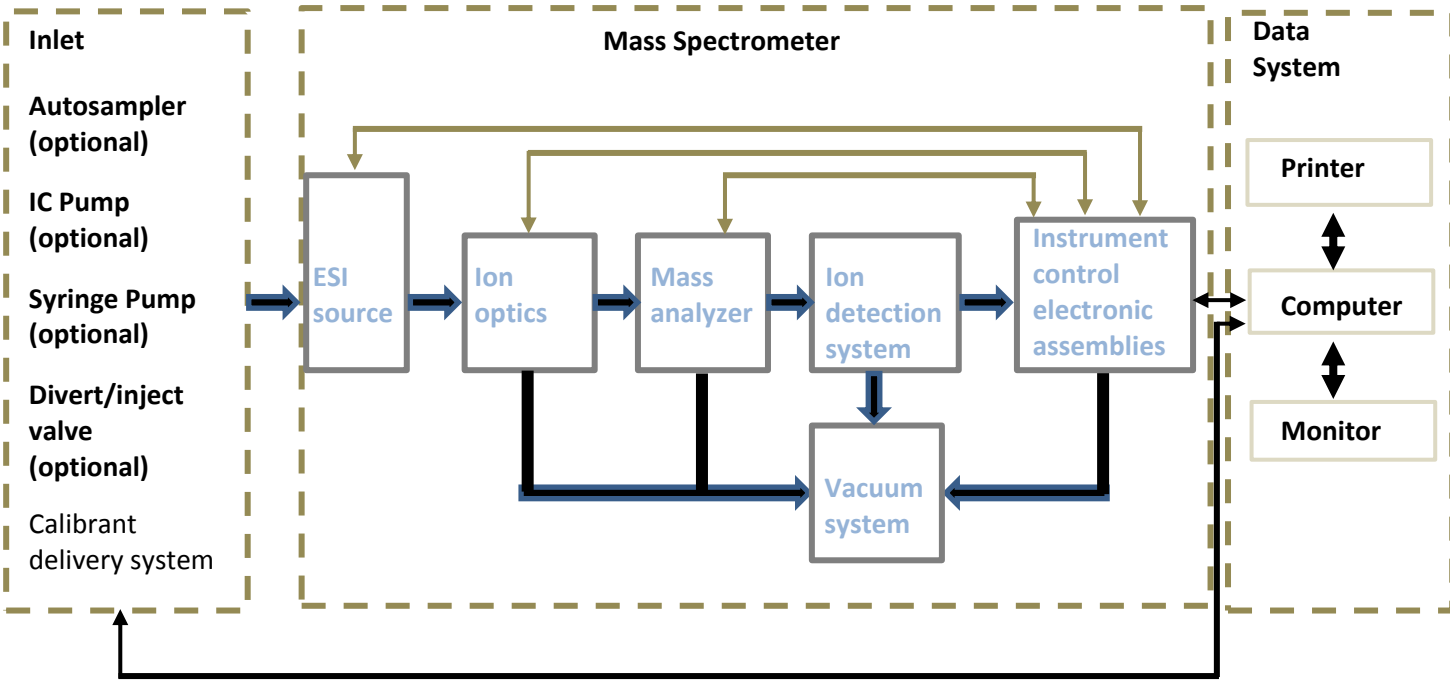


Figure 1: Diagram of an IC/MS system.

In an IC-MS system, the eluent stream is vaporized to ionized gas stream (with or without make-up solvent) and enters the ion source already ionized. The ion optics extracts the ions from the neutrals (e.g., solvents) and transfers them to the quadrupole which filters the ions (*m/z*) of interest. The resulting mass-selected ions are then transferred to the ion detection.

3.3 Software Operation

The IC-MS systems are designed to be operated from a computer configured with the Thermo Scientific™ software: 1) Thermo Scientific™ Chromeleon Chromatography Data System (CDS) with Thermo Scientific™ software or 2) Thermo Scientific™ SII for Xcalibur™ software with Xcalibur and Foundation software. CDS is a full version of Chromeleon, whereas SII for Xcalibur is a version of Chromeleon software embedded in Xcalibur software. The Chromeleon CDS software provides complete instrument control, data acquisition, and data management, whereas the SII for Xcalibur is an integrated part of Xcalibur software to manage the IC control, configuration and the IC data acquisition. The IC channels are incorporated as analog channels in SII for Xcalibur (with Xcalibur and Foundation). Everything else is managed by Xcalibur: sequence, MS scan, instrument methods, tune files, data acquisition, and the post-acquisition processing.

The data system serves as the user interface to the mass spectrometer, the IC autosampler, IC system, and syringe pump. Refer to the mass spectrometer manuals and Chromeleon Help or Xcalibur manuals for more information. Each sequence of loading a mass analyzer with ions followed by mass analysis of the ions is called a scan.

3.3.1 Thermo Scientific ISQ EC single quadrupole mass spectrometer using Chromeleon CDS software

For the Thermo Scientific ISQ EC single quadrupole mass spectrometer utilizes both a full scan type and/or selected ion monitoring (SIM) scan type to create, fragment, and detect ions. The ability to vary not only the ionization and ion polarity modes, but the scan mode and scan type, provides greater flexibility in the instrumentation for solving complex analytical problems.

The Method Editor in Chromeleon allows the user to set up scans in a variety of ways, combining both full scans and SIM scans. The user can also call for scans of either or both polarities. Simplified ion source controls and a simplified method editor make setting up an instrument method easier than ever, while the ability to switch to manual controls means that users who wish to control each device independently do not lose that freedom. For more information on the Method Editor, please see the Chromeleon Help file. Users can set up full scans to get a picture of the entire mass spectrum as well as set up SIM scans to monitor for the presence of specific ions. The ability to apply source collision induced dissociation (sCID) gives the user the capability to reduce the presence of adducts, or to create confirmation ions via fragmentation in the ion optics of the system.

3.3.2 Thermo Scientific TSQ Triple Quadrupole mass spectrometers using Chromeleon CDS software

The Method Editor in Chromeleon allows the user to set up scans similarly to the ISQ EC: full scan, selected reaction monitoring SRM scans, and product ions scans.

3.3.3 Thermo Scientific TSQ Triple quadrupole mass spectrometer using SII for Xcalibur software

The Instrument Method in SII for Xcalibur with Xcalibur allows the user to set up the same scans: full scan, selected reaction monitoring SRM scans, and product ions scans.

3.3.4 Thermo Scientific Q Exactive Hybrid Orbitrap-Quadrupole mass spectrometers using SII for Xcalibur software

Although Chromeleon CDS can run an IC-MS system with the Q Exactive MS family and collect data, the data processing of the high-resolution accurate mass data can be challenging. SII for Xcalibur with Xcalibur is recommended for this configuration. If untargeted analysis (e.g., unknowns) is desired, then export the data to FreeStyle directly from Chromeleon software.

The Instrument Method in SII for Xcalibur with Xcalibur allows the user to set up: full scan with or without data dependent MS/MS (dd-MS/MS), SIM scans, parallel-reaction monitoring (PRM) scans, and data independent acquisition scans.

- Full scan - with or without data dependent MS/MS (dd-MS/MS) with typically top 5 fragmented into MS/MS spectra.
- SIM - selected ion monitoring provides simple method setup with maximum selectivity and sensitivity. It is ideal for quantifying small numbers of compound in samples of moderate complexity.
- PRM - parallel-reaction monitoring provides high selectivity and high sensitivity with confident targeted confirmation. Quantitation is carried out post-acquisition for greater throughput. High-resolution, accurate-mass Orbitrap spectra ensure confident confirmation through library matching.
- DIA - data independent acquisition enables large-scale targeted proteomics studies with qualitative confirmation. This simple and universal acquisition set-up does not require detailed sample knowledge prior to data acquisition. DIA increases reproducibility and comprehensiveness with a complete record of quantitative data. It facilitates retrospective analysis of additional analytes.

3.4 Two software (Chromeleon + Xcalibur) Installation

For IC-MS installations it is common that the analyst may plan to switch from anions to cations or from IC-MS to HPLC-MS (Figure 2). As the MS can only accept one channel at a time, an effective solution would be to use a two-software installation: Chromeleon and SII for Xcalibur or Chromeleon and Xcalibur.

The software configuration can use SII for Xcalibur with Xcalibur by having two separate instrument configurations. However, the IC eluent generator system and the suppressor will undesirably turn-off when the configuration is switched to HPLC-MS. It is often more convenient to use Chromeleon independently for the IC-MS applications from Xcalibur for the HPLC-MS applications.

3.4.1 Case 1: Anions + Cations + HPLC with a single MS and single computer

Two software installation is typical when you need switch automatically between 2 or more lines connected to a single MS (Figure 2). See Section 9.9.2 for more details

Figure 2: Configuration with two IC channels, one HPLC channel to one MS using one computer.

3.4.2 Case 2: IC and LC with MS and independently using two computers

In this example, the customer wants to operate with IC or LC with the MS but also stand alone. In this case, the MS is connected to one computer and chromatographic devices (IC and LC) are standalone systems controlled by a second computer.

All quantification and post-acquisition steps are managed by MS software. Chromeleon CDS is used only to control chromatographic hardware.

Chromeleon CDS is installed on chromatographic computers without MS control (Figure 3).

Figure 3: Configuration with two chromatography channels controlled by one computer and the MS using a second computer

On Xcalibur instrument setup (MS PC), only the MS is included in the configuration. The IC or LC instruments are not in the configuration. See Section 9.9.2.

3.4.3 Case 3, Automatic switching from anions to cations for IC-MS.

It is very common to run both anion and cation applications using the capabilities of the Dionex ICS-6000 HPIC modular dual channel IC system. To accommodate the single MS channel, the IC is configured with Chromeleon CDS on one or two timebases with the MS configured in Xcalibur. See Section 9.9.3 Case 3, Automatic switching from anions to cations. This cannot be performed with the ISQ EC MS.

4.0 Installation

This chapter provides information for installing IC-MS systems.

The mass spectrometer is installed and set up by a Thermo Fisher Scientific service engineer or representative. The service engineer checks that the installation is correct, and that the detector operates as specified. The engineer also demonstrates the basic operation and main features.

Pay attention to the safety guidelines and observe all site requirements.

4.1. Positioning the IC system to the MS system

As with all detectors, it is ideal to have the separation (IC) as close as possible to the detection (MS). Additionally, reducing the distance between the IC and MS reduces the backpressure and stress on the IC electrolytic suppressor. See Minimizing backpressure on the suppressor, Section 6.

1) Position the IC as close to the MS as possible (side-by-side provides the minimum distance) to minimize tubing length. If the MS will be shared by more than one IC or other chromatography system, it may be more practical to install the IC on a mobile bench.

2) Position the IC so that the tubing leaving the IC is as close as practical to the MS.

3) For applications using an Integrion system, position the Integrion to the right of the Q Exactive mass spectrometer so that the diverter valve is closest the MS ion source.

4) For the modular systems, stack the autosampler on top of the DC Detector-Column Oven module. Position the IC with the IC diverter valve as close to the MS ion source as possible. Select the CD detector for the configuration that is closest to the Automation Manager valve.

4.2 Components of the IC-MS Installation Kit (P/N 22153-62049)

The following are components of the IC-MS Installation Kit (P/N 22153-62049)

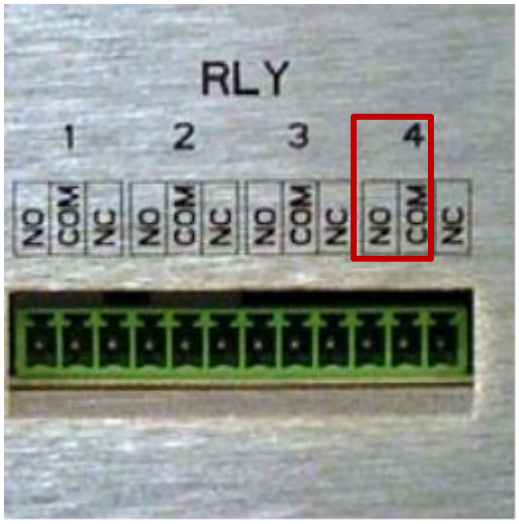
- Tubing, fittings
- IC PEEK Viper fittings
- 2-pin connector for MS Contact Closure cable
- 4 L bottle for water regenerant
- 1 L glass bottle for solvent

4.3 Installing Contact Closure (TTL) cables from Dionex IC AS-AP Autosampler to MS system using SII for Xcalibur

4.3.1 Contact Closure is not needed when using Chromeleon CDS software to control the IC-MS system and data collection. Therefore, Contact Closure cables are not needed.

4.3.2 Conditions:

- When using SII for Xcalibur with Xcalibur and Foundation software to control the IC-MS system and data collection. The start initiation is through Contact Closure.
- Contact Closure starts the sequence by loading the instrument methods and preparing the instruments to collect data. Both instruments go to Ready state.
- Contact Closure using Relay 4, initiates the Dionex AS-AP autosampler to start and inject the sample. The sample injection initiates data acquisition by both the IC and MS systems.
- Install a Contact Closure (TTL) cable (for example, Red Black twisted pair) between the MS and the Dionex AS-AP autosampler. To assemble the two-wire contact closure cable, use the contact closure mating connector (P/N 00004-21512) supplied in the MS Setup Kit and the contact closure cable and provided with the IC or HPLC



For the Dionex AS-AP autosampler, connect the red wire to Relay 4 NO and black wire to COM. (Figure 4). See the Dionex AS-AP autosampler operating manual for more information.

Figure 4. Relay connections on back of the Dionex AS-AP autosampler for TTL cable. Red wire to Relay 4 NO and black wire to COM

For the Q Exactive MS family, connect the other end of the cable to the MS, Start In, Pins 1 and 2 (Figure 5 below). See the Q Exactive Operating "User I/O Connections" section.

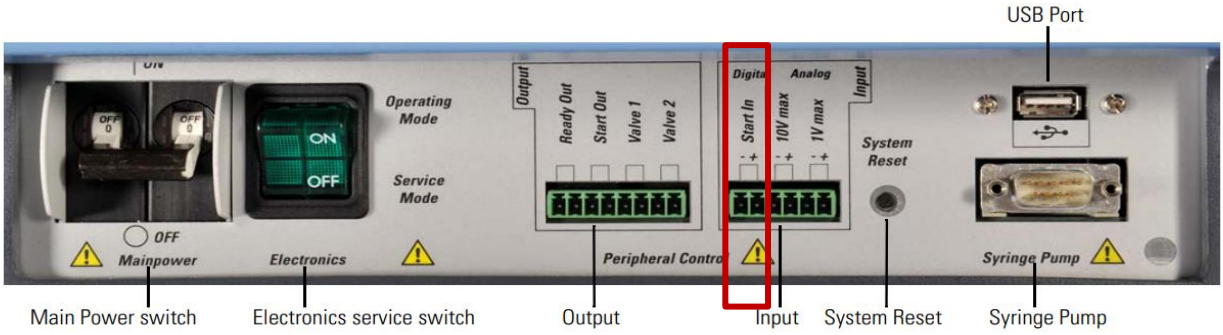


Figure 5. Relay connections on Q Exactive mass spectrometer for Contact Closure (TTL) cable. Use Start In, Pins 1 and 2.

For the TSQ Altis, Quantis, and Fortis MS family, connect the other end of the cable to the MS, Start In, Pins 1 (ground) and 4 (Figure 6). See the *TSQ Series II TSQ Altis, TSQ Quantis, and TSQ Fortis Getting Connected Guide* for more information.

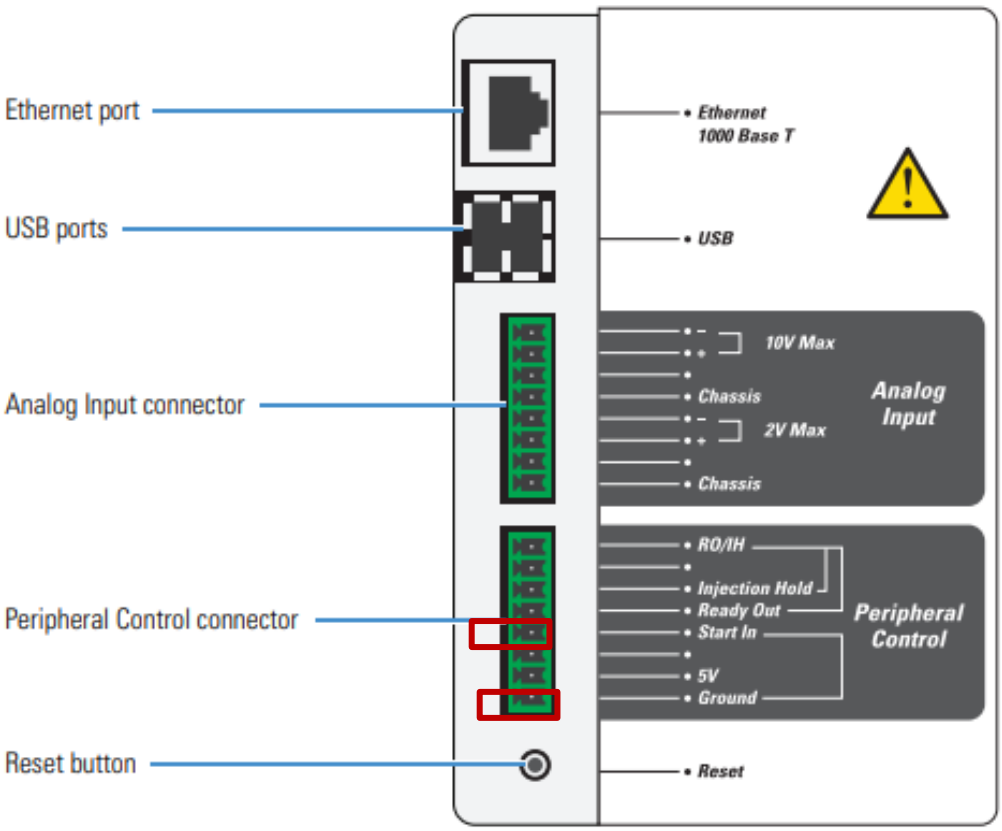


Figure 6. Relay connections on TSQ (Fortis, Altis, Quantis) mass spectrometers for Contact Closure (TTL) cable. Use Start In, Pins 1 and 4.

Activate Relay 4 in Instrument Configuration for the Dionex ASAP autosampler. See Sections 9.7-9.9, Electronic configuration.

5.0 Critical Items for Using Ion Chromatography with MS detection or Electrochemical-MS Detection

5.1 Critical Items for Using Suppressed Conductivity-Mass Spectrometry

5.1.1 Reagents

- Always use clean DI water: ASTM Type 1 deionized water for manually prepared eluent or eluent generation, and regenerant water for the suppressor.
- Always use Optima grade solvent for applications requiring desolvation solvent (make-up flow). Using a new unopened bottle ensures that the solvent has not been contaminated
- Desolvation solvent:
    - Desolvation solvent is not needed for many applications using the HESI II MS probe such as the ISQ EC mass spectrometer, TSQ MS and Q Exactive MS systems.
    - However, some of the older applications include desolvation solvent.
    - Some of the carbohydrate applications use 0.5 mM LiCl to create lithium adducts detectable in positive mode.

5.1.2 Physical Location

- Position the IC system as close as possible to the MS system to minimize the tubing length and subsequent backpressure to the suppressor and diffusion of previously separated chromatography peaks.
- It is advisable that the IC be placed on a cart or mobile bench when the customer plans to use both HPLC and IC with the MS.

5.1.3 Electrolytic Suppression

A properly operating electrolytic suppressor (DRS 600, ERD 500, ERS 500e, SRS) is critical to IC-MS applications. Failure of the suppressor may lead to severe damage to the MS.

It is CRITICAL that the suppressor has:

- Sufficient and continuous regenerant flow, 2x eluent flow, while under operation.

    - Sufficient programmed current (constant current mode) or programmed voltage (constant voltage mode). Refer to application document conditions.
    - Backpressure < 50-100 psi so that the suppressor is not stressed (tubing length, mixing tee, diverter valve, MS probe).
- Hydration: Follow the new suppressor hydration process that is recommended for the DRS 600 suppressor family, included in the suppressor box. See section on Conditioning Consumables.
- Monitor: Always use a CD detector inline after the suppressor or ERD 500 to monitor ion-suppression (desalting).
- Regenerant: ALWAYS use an AXP-Pump or the extra pump on the ICS-6000 system for external suppressor (and CR-TC, if applicable) regeneration.
    - A peristaltic pump is not recommended as pressure can't be monitored and emergency measures can't be applied. Failure of the pump dispensing regenerant can cause severe damage to the MS.
    - The AXP auxiliary pump can lose prime from an air bubble in the line. Prime and remove > 20 mL of water during priming.
    - Verify that the liquid is flowing into the suppressor regenerant port.
    - Regenerant flow rate should be 2x of eluent flow rate.

- Measure: Always measure the pressure before and after the suppressor during hydration and during installation. Limit backpressure to the suppressor to be < 50 psi.
    - Remove any backpressure coils added after CD Out on the CD detector. Install these backpressure coils on the waste position in the IC diverter valve.
    - Recommend IC PEEK Viper fittings (0.007 in i.d., 0.178 mm i.d.) for the IC-MS interface including CD Out to diverter valve to capillary on MS.
    - As an alternative where peak diffusion is not an issue, black PEEK (0.010 in i.d., 0.254 mm i.d.) for the IC-MS interface. Additionally, replace the Red PEEK tubing at the MS Source with Black PEEK tubing.
    - For experienced IC-MS users and where isobaric compounds have similar elution times, recommend Red PEEK tubing (0.005 in i.d., 0.127 mm i.d). for the IC-MS interface and MS probe. Provides less diffusion of chromatographically separated peaks but requires monitoring the backpressure to the suppressor as it can exceed safe limits.
- Total system pressure must be > 2000 psi while running or in Standby mode. This is necessary for eluent generator devices.
- Preventative Action:
    - Always program the diverter valve to direct the eluent flow away from the MS at the end of each instrument program.

- Always run a sequence with emergency programs in case the suppression fails during the sequence. See High Conductivity Trigger and low-pressure error for the auxiliary pumps.
- Always use a standby method at the end of the sequence. Required: The Standby method must have a flow rate sufficient to maintain 2000 psi system pressure.

5.1.4 Diverter Valves and Sequence control

Diverter valves are used to direct eluent flow from the IC system that may not any have analytes of interest, such as before the void volume or before the first analyte of interest or just prior to the end of a sample run. Diverter valves can also be programmed to direct the eluent flow away from the MS to minimize ion suppression from the sample matrix.

- Always use a Diverter Valve, preferably an IC PEEK valve.
- Always program the diverter valve in the Instrument Method to direct flow away from MS near the end of each injection.
- Always program the diverter valve in the Instrument Method to direct flow away from MS to minimize high ionic content from the sample matrix. For example: U.S. EPA Method 557, U.S. EPA 332.0

5.2 Critical Items for Using Electrochemical Detection (ED) with Mass Spectrometry Detection

5.2.1 Reagents

Same as Section 5.1.1

5.2.2 Physical Location

Same as Section 5.1.2

5.2.3 Electrolytic Suppression or Desalting

- Flow Splitter: Split the stream after the column, 40-50% to ED cell and 50-60% to the ERD 500 device to CD Conductivity Detector to MS.
  - Option 1: Split the flow after column to the ED and to the suppressor-CD-MS by using a tee, and backpressure tubing before the ED cell.
  - Option 2: Split the flow after column to the ED and to the suppressor-CD-MS by using a flow splitter.
- Select the suppressor or the Dionex ERD 500 based on the eluent concentration and the split ratio. Use a suppressor for low eluent concentrations. For eluent concentrations > 100 mM (typically sodium acetate plus sodium hydroxide), select the ERD 500 device. See sections on Electrolytic suppressors.
- As of this revision, the Dionex ERD 500 device requires an interface cable (P/N 22181-98150) to operate properly with the Dionex ICS-6000 HPIC system. P/N 22181-98150 is a non-customer orderable part. Contact your sales or service representative for this part.

A properly operating electrolytic suppressor or desalter (DRS 600, ERS 500e, SRS 300, ERD 500) is critical to IC-MS applications. Failure to suppress or desalt may lead to severe damage to the MS.

It is CRITICAL that the suppressor or desalter has:

- Sufficient and continuous regenerant flow, 1x eluent flow, while under operation.
- Sufficient programmed current (constant current mode) or programmed voltage (constant voltage mode). Refer to application document conditions.
- Backpressure < 50-100 psi so that the suppressor is not stressed (tubing length, mixing tee, diverter valve, MS probe).

- Hydration: Follow the new suppressor hydration process that is recommended for the DRS 600 suppressor family in the suppressor box. See section on Conditioning Consumables.
- Monitor: Always use a CD detector inline after the suppressor or ERD 500 to ensure enough ion-suppression.
- Regenerant: ALWAYS use an AXP-Pump or the extra pump on the ICS-6000 system for external suppressor (and CR-TC, if applicable) regeneration.
  - Caution: A peristaltic pump is not recommended as pressure can't be monitored and emergency measures can't be applied. Failure of the pump dispensing regenerant can cause severe damage to the MS.
  - The AXP auxiliary pump can lose prime from an air bubble in the line. Prime and removed > 10 mL of water during priming.
  - Verify that the liquid is flowing into the suppressor regenerant port.
  - Regenerant flow rate should be 1x of eluent flow rate.
  - Backpressure < 50-100 psi so that the suppressor or ERD is not stressed (tubing length, mixing tee, diverter valve, MS probe).
- Note: As of this revision, the Dionex ERD 500 device requires an interface cable (P/N 22181-98150) to operate properly with the Dionex ICS-6000 HPIC system. P/N 22181-98150 is a non-customer orderable part. Contact your sales or service representative for this part.

- Measure: Always measure the pressure before and after the suppressor during hydration and during installation. Backpressure to suppressor or the ERD 500 device should be < 100 psi.
  - Remove any backpressure coils added after CD Out on the CD detector. Install these backpressure coils on the waste position in the IC diverter valve to minimize baseline disturbances in the chromatography.
  - For experienced IC-MS users, recommend Red PEEK tubing (0.005 in i.d., 0.127 mm i.d). for the IC-MS interface and MS probe. Provides less diffusion of chromatographically separated peaks but requires maintaining low backpressure to suppressor.

    - For less experienced users, recommend IC PEEK Viper fittings (0.007 in i.d., 0.178 mm i.d.) or black PEEK (0.010 in i.d., 0.254 mm i.d.) for the IC-MS interface.
    - Additionally, replace the Red PEEK tubing at the MS Source with Black PEEK tubing.
- Monitor: Always use a CD detector inline after the suppressor or ERD 500 to ensure sufficient ion-suppression (desalting).
- Always use a StandBy method (with reduced IC flow, reduced suppressor current, and diverter positioned away from the MS) as last injection of a sequence. Required: The StandBy method must have a flow rate sufficient to maintain 2000 psi system pressure.
- Preventative Action: Always use an emergency programs in case the suppression fails during the sequence. See High Conductivity Trigger and low-pressure error for the auxiliary pumps.

5.2.4 Diverter Valves and Sequence control

- Same as 5.1.4

6.0 Electrolytic Devices

6.1 Backpressure limits for electrolytic devices

The Thermo Scientific Dionex electrolytic devices are membrane devices that can be damaged or broken if the back pressure on the device exceeds the devices' limitations and if DI water has ceased flowing through the regenerant channels. Backpressure is defined as pressure exerted after the device.

Always use an emergency shutdown program (high conductivity trigger), and a low-pressure program when using the AXP pump to deliver the regenerant water to the electrolytic suppressor, electrolytic removal device, or electrolytic trap column.

For IC-MS applications, the backpressure from the MS and IC-MS interface can irreversibly damage the suppressor or desalter if the pressure exceeds 150 psi.

Please note the maximum backpressure limitations for the following electrolytic devices in Table 2:

Device	Model	System pressure (psi)	Maximum Backpressure specification (psi)	Recommended Operating backpressure limit (psi)
Eluent Generator cartridge	EGC 500	5000	5000	< 5000
	EGC II	3000		
Electrolytic Trap Column	CR-TC 500 CR-TC 600	5000	5000	< 5000
Electrolytic suppressor	DRS-600 ERS 500e	5000	Eluent side: 150	< 50-100
			Regenerant side: 20	< 20
	SRS 300	3000	Eluent side: 150	< 50-100
			Regenerant side: 20	< 20
Electrolytic Desalter	ERD-500	5000	Eluent side: 150 Regenerant side: 20	< 50-100 < 20
Carbonate Removal Device*	CRD 200 CRD 300	Not suitable for IC-MS	100	< 5000
EG Degas module	EG Degas module	5000	5000	< 5000
Inline Electrolytic Water Purifier (EWP)	EWP-500	5000	5000	< 5000

Table 2: System and Consumable Pressure Tolerances

- Exception: When used as a sample preparation device on the low-pressure side of the IC, such as the between the injection valve and the IC pump.

6.2 IC PEEK Tubing

IMPORTANT— The colors are custom blended. Use only Dionex IC tubing. Check inside diameter (i.d.) to verify size**.**

Note:

- Remove any backpressure coils added after CD Out on the CD detector. Install these backpressure coils on the waste position in the IC diverter valve to minimize baseline disturbances from pressure changes during the diverter valve rotation.

- For most users, recommend Dionex IC PEEK Viper fittings (0.007 in i.d., 0.178 mm i.d.) for the IC-MS interface. Additionally, replace the Red PEEK at the MS Source to IC PEEK Viper fittings.
- As an alternative where peak diffusion is not a problem, use black PEEK (0.010 in i.d., 0.254 mm i.d.) for the IC-MS interface. Additionally, replace the Red PEEK at the MS Source to Black PEEK tubing

- For experienced IC-MS users when isobaric compounds have similar elution times and peak diffusion must be minimized, recommend Red PEEK tubing (0.005 in i.d., 0.127 mm i.d.) for the IC-MS interface. Red PEEK tubing provides less diffusion of chromatographically separated peaks. Caution: requires minimizing and monitoring the backpressure to suppressor as Red PEEK tubing can cause excessive backpressure to the suppressor.

Color	I.D.	Location 1
IC PEEK Viper (Do not use other Viper fittings)	0.007 in 0.18 mm	Preferred. 1. Between EGC cartridge & CR-TC column 2. Between Degas module to Injection Valve 3. Between column connections 4. Between column and suppressor 5. Between suppressor CD detector 6. Recommended for most cases between the IC diverter valve and MS
Gray Blue	0.0025 in 0.062 mm	Capillary IC (0.4 mm i.d. columns) only
Yellow backpressure	0.003 in. 0.075 mm	1. ICS-6000+ DP2 (or AXP pump) for dispensing solvent to provide pump backpressure. 2. AXP pump to dispense continuous water at low flow rates to the suppressor regenerant channel 3. Backpressure tubing for AXP pump to load calibrant during ISQ EC/EM MS calibration
Red	0.005 in. 0.125 mm	1. From Injection Valve to remainder of CD detector for 2 mm i.d. columns 2. Optional, when isobaric compounds require separation and have similar elution times. Connects eluent lines from CD out to diverter valve, and MS. May cause excess pressure and irreversible damage to suppressor. Check effect of tubing on suppressor backpressure.
Black Note: different than standard Upchurch size	0.010 in. 0.25 mm	1. From Pumps to EGC to Injection Valve (or IC PEEK Viper fittings) 2. Short length in Waste Port of injection valve to start the waste line 3. Alternative to IC PEEK Viper fittings. Connects eluent lines from CD out to diverter valve and to MS
Blue	0.013 in 0.33 mm	1. Standard tubing for ASAP autosampler transfer line from autosampler to injection valve
Orange	0.020 in. 0.50 mm	1. Sometimes used as sample loops.
Green	0.030 in. 0.75 mm	1. Prudent use to minimize organic contamination: From inside solvent bottle to solvent pump (DP Pump 2 or AXP-MS pump) 2. Waste line

Table 3: IC PEEK Tubing

6.3 Selecting Electrolytic Suppressor or Electrolytic Regenerated Desalter Devices

The Dionex DRS 600 or ERS 500e are used for most IC-MS applications. Some cationic and anionic pesticides require the suppressor without resin, such as the SRS-300 suppressor.

Electrolytic suppressors are preferred in ED-MS (or Pulsed Amperometic Detection -Mass Spectrometry (PAD-MS) applications where the total eluent concentrations are < 200 mM for 4 mm flow rates and < 50 mM for 2 mm flow rates. Use an ERD-500 device when the total eluent concentration is above these limits. See Table 4.

- As of this revision, the Dionex ERD 500 device requires an interface cable (P/N 22181-98150) to operate properly with the Dionex ICS-6000 HPIC system. P/N 22181-98150 is a non-customer orderable part. Contact your sales or service representative for this part.

IC System	Suppressor Mode	Suppressor	ERD-500
ICS-6000	Dynamic or constant voltage	DRS 600	N/A
	Constant current	DRS 600 or ERS 500e SRS 300 type (zwitterions, polar pesticides)	Applications using sodium acetate eluent
Integrion HPIC	Constant current	DRS 600 or ERS 500e SRS 300 type (zwitterions, polar pesticides)	N/A
ICS-5000+ HPIC	Constant current	DRS 600 or ERS 500e SRS 300 type (zwitterions, polar pesticides)	Applications using sodium acetate eluent

Table 4. Electrolytic Suppressors and Electrolytic Regenerant Desalting devices

6.4 Selecting Eluent Generation Consumables

The Dionex DRS 600 or ERS 500e are used for most IC-MS applications. Some cationic and anionic pesticides require the suppressor without resin, such as the SRS-300 suppressor.

Electrolytic suppressors are preferred in ED-MS applications where the total eluent concentrations are < 200 µegy for 4 mm flow rates and < 50 µegy for 2 mm flow rates. Use an ERD-500 device when the total eluent concentration is above these limits. See Table 5 for the recommended electrolytic devices with each IC system.

IC System	Eluent Generator Cartridge	Electrolytic Trap	Degas device
ICS-6000	Dionex EGC 500	CR-TC 600	HP EG Degas
Integrion HPIC	Dionex EGC 500	CR-TC 600	HP EG Degas
ICS-5000+ HPIC	Dionex EGC 500	CR-TC 500	HP EG Degas

Table 5. Recommended Electrolytic Devices with each IC system.

7.0 Conditioning Consumables

7.1 Conditioning Eluent Generator Cartridges and CR-TC trap columns

Condition the eluent generator cartridges and CR-TC trap columns according to product manuals and the installation instructions received with the devices.

TIP—For more information, review the consumables conditioning drop-down instructions on the Chromeleon Console as well as the product user manuals.

7.2 Conditioning Columns

Condition the guard and separation columns 30 min using the Quality Assurance Report (QAR) conditions. Direct the flow to waste. Follow the directions in the Column Product Manual.

7.3 Conditioning Suppressors and ERD desalting devices.

- Conditioning is done without applying voltage or current to the suppressor or the ERD desalting device for 20 min hydration and 20 min resting.
- Temporarily install a black (0.010 in i.d., 0.254 mm i.d.) or green (0.03 in i.d., 0.762 mm i.d.) PEEK tubing from the Eluent Out port to the Regen In port. Direct the Regen Out stream to waste.
- Hydrate the eluent and regenerant sides of the suppressor or ERD by pumping < 10 mM eluent into the Eluent In port of the suppressor at 0.15 mL/min for 10 min.
- Do not allow the pressure to exceed 100 psi during the hydration.
- Caution: Do not run the hydration step longer than 10 min.

TIP—For more information, review the Suppressor Installation Instructions.

- Stop the IC flow and allow to sit for 20 min before use.

8.0 AXP and AXP-MS Auxiliary Pumps

Figure 7. "New" AXP pump models after 2016

8.1 Selecting AXP and AXP-MS pumps

The AXP and AXP-MS auxiliary pumps (Figure 7) are single piston pumps and therefore more susceptible to trapping air in the pump, blocking the pump from dispensing water, solvent, or regenerant solution (losing prime).

Recommended AXP pump	Liquid
AXP pump (P/N 063973) with flow rates up to 10 mL/min.	Suppressor Regenerant: ASTM Type I deionized water
AXP-MS pump (P/N 060684) with flow rates from 0.01 to 1.0 mL/min Or second pump on ICS-5000+ or ICS-6000 DP module	Carrier: ASTM Type I deionized water as a carrier for ISQ EC/EM AutoTune solution. See Thermo Scientific Technical Note TN72611
	Desolvation (or make-up flow): Solvent if needed for the application. Acetonitrile, methanol, or isopropyl alcohol.
	Ionization: Some carbohydrate applications use 0.5 mM lithium chloride (LiCl) at 50 µL/min.

Table 6. Recommending AXP or AXP-MS pump for each function

Caution: The AXP-MS pump has an internal flow restrictor that is needed for Thermo Scientific ISQ EC and ISQ EM MS calibration. This flow restrictor must be removed for regenerant flow above 0.25 mL/min.

It is critical that regenerant (water or chemical) continuously flow through the regenerant channel of the operating suppressor or operating Electrolytically Regenerated Desalter (ERD) device.

- Prime the pump if a new solution or water is added or when air may have been introduced into the tubing lines. Prime the pump so that > 20 mL has been removed.
- Verify that the pump is dispensing liquid at the expected rate to the suppressor or ERD desalter before turning on the device. Manually prime if necessary.
- Use an emergency program in case the pump fails to deliver the regenerant. See low-pressure trigger section.
- Install yellow backpressure tubing for a backpressure above 500 psi. The pump pressure must be above 500 psi for the low-pressure trigger to work, and 1000 psi is recommended.

8.2 AXP pump for Suppressor Regenerant

- For external suppressor regeneration it is strongly recommended to use an AXP pump or the extra pump on a Thermo Scientific ICS-5000+ or ICS-6000 HPIC system.
- The use of a peristaltic pump is not recommended since regenerant flow cannot be monitored. Failure of the suppressor may lead to severe damage of the MS.

Parts List	Thermo Scientific P/N	Description
AXP Pump	063973	Flows 0.1 to 10 mL/min
AXP Pump Ship kit		• Power Cord • 10 Pin Connector, 4 Pin Connector • Adapter RJ to DB9m Modular Data Cable • USB cable/adapter • Eluent Tubing with End-of-Line Eluent Filter and Fittings • Rheodyne 9055-022 (20 µL Loop) • Tubing for Seal Wash • Nut and Ferrule (Rheodyne 9726-015) for Peek Tubings (10-32) • Syringe
Unions (coupler)	042806	1/4-28 Female to 10-32 Female
High pressure PEEK tees	IDEX P/N P-727 Fisher Scientific P/N 10392975	1/16” OD Tubing (not adjustable), 10-32 coned threads, max. 3500 psi (242 bar), ID = 0.020" (0.50 mm, 0.57 µL
4L HDPE bottle	067796	Bottle, 4 L, RFIC, 4L-Bottle without stopper assembly (enhanced H2O-volume available for external regeneration of suppressor, to be used with one of the stopper assemblies already provided with PN 072057)

	063292	Bottle, 4 L, RFIC, 4L-Bottle with stopper assembly (enhanced H2O-volume available for external regeneration of suppressor
Flow splitter	IDEX P/N P-451 Fisher Scientific P/N 05-700-384	Adjustable Flow Splitter for 1/17" OD Tubing, 10-32 coned threads, biocompatible, 1.2 / 2.8 µL (closed/fully open) volume, max. 800 psi (55 bar)
	IDEX P-727 Fisher Scientific P/N 05-700-185	High Pressure PEEK Tee for 1/16" OD Tubing (not adjustable), 10-32 coned threads, max. 3500 psi (242 bar), ID = 0.020" (0.50 mm, 0.57 µL)

Table 7. AXP Pump Parts list

8.3 AXP-MS Pump: Recommended for Make-up Flow, and Carrier for ISQ EC/EM Calibrant flow

The AXP-MS pump provides a stable flow rate at low flow rates and therefore is recommended for make-up flow when recommended in the application and ISQ AutoTune, when provided with an additional pulse dampener and backpressure coil.

Note: Make up flow is not obligatory for ISQ EC/EM MS spectrometer, but recommended for many earlier IC-MS, IC-MS/MS, and IC-HRAM MS applications.

Parts List	Thermo Scientific P/N	Description
AXP-MS Pump	060684	Flows 0.01 to 1.0 mL/min
AXP Pump Ship kit		• Power Cord • 10 Pin Connector, 4 Pin Connector • Adapter RJ to DB9m Modular Data Cable • USB cable/adapter • Eluent Tubing with End-of-Line Eluent Filter and Fittings • Rheodyne 9055-022 (20 µL Loop) • Tubing for Seal Wash • Nut and Ferrule (Rheodyne 9726-015) for Peek Tubings (10-32) • Syringe
Backpressure coil	053765	1000 psi backpressure coil
Twisted pair cable	043598	ASSY, WIRE, TWPR,96IN+BAG (Twisted Black and Red Wires for the TTL connection and 8-pin connector

Unions, reducing (coupler)	042806	1/4-28 Female to 10-32 Female
High pressure PEEK tees	IDEX P-727 Fisher Scientific P/N 05-700-185	1/16” OD Tubing (not adjustable), 10-32 coned threads, max. 3500 psi (242 bar), ID = 0.020" (0.50 mm, 0.57 µL
For applications using desolvation or make-up solvent: 1 L glass bottle	062595	Dionex (THERMO) Bottle 1L, glass, screw cap with openings for eluent tubing
For applications using the ISQ EC/EM mass spectrometer • GM-3 Gradient Mixer	042126	GM-3 Gradient Mixer used for ISQ AutoTune
• Backpressure tubing	053765	Additional 1000 psi backpressure coil (needed for stable flow during AutoTune)

Table 8. AXP Pump Parts list

9.0 Electronic configuration

9.1 IC-MS Software

IC-MS applications can use Thermo Scientific™ Chromeleon™ Chromatography Data Systems (CDS) software or Thermo Scientific™ SII for Xcalibur with Thermo Scientific™ Xcalibur™ or Thermo Scientific™ TraceFinder™ to operate the instruments, collect and analyze the data. See Table 9 below.

	IC-MS System			
Software Program	IC System	MS*	AXP pumps	Section
--	COM Port for AXP & AXP-MS pumps			9.2
Chromeleon	Dionex Integrion HPIC system	ISQ EC Single Quadrupole	AXP-MS	9.3
	Dionex Integrion HPIC system	TSQ Triple Quadrupole	AXP & AXP-MS	9.4
	Dionex ICS-5000+ or ICS-6000 system	ISQ EC Single Quadrupole	AXP-MS	9.5
	Dionex ICS-5000+ or ICS-6000 system	TSQ Triple Quadrupole	AXP & AXP-MS	9.6
Xcalibur, SII for Xcalibur	Dionex Integrion HPIC system	TSQ Triple Quadrupole or Hybrid Orbitrap-Quadrupole	AXP & AXP-MS	9.7
	Dionex ICS-5000+ or ICS-6000 system	TSQ Triple Quadrupole or Hybrid Orbitrap-Quadrupole	AXP & AXP-MS	9.8

Table 9: Configurations for IC-MS

* The ISQ EC and ISQ EM Single Quadrupole Mass spectrometers are designed to run only on Chromeleon CDS software. That is, these single quadrupole mass spectrometers are not compatible with SII for Xcalibur or Xcalibur.

9.2 Finding the COM port for the AXP and AXP-MS pumps

1) Connect power cord. Connect micro-USB cable from the AXP pump to the computer.
2) Download micro-USB driver from PSB or SSI
3) Connect USB serial cable or USB serial adapter to computer.
4) Turn on the AXP pump.
5) Install the drivers using the CD provided.
6) Device Manager: Open Device Manager to identify the USB serial ports being used by AXP pumps. Shown in 8: COM5 and COM6.

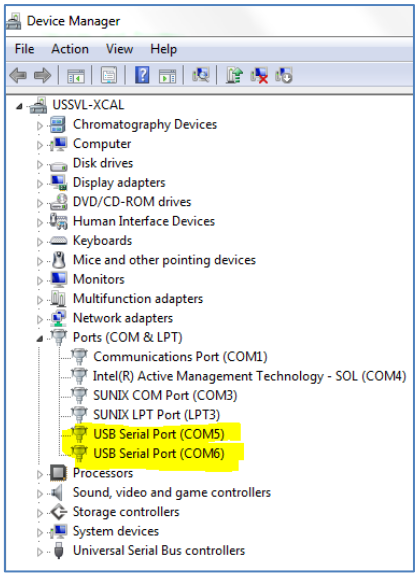


Figure 8: Device Manager displaying COM ports used by AXP Pumps

9.3 Chromeleon: IC-MS system using a Dionex Integrion HPIC system coupled to Thermo Scientific ISQ EC mass spectrometer

9.3.1 Find the COM port the AXP and AXP-MS pumps by following Section 9.2

9.3.2 Opening Chromeleon Configuration Manager program

a) Start Chromeleon Instrument Services Manager program

b) Select the link Configure Instruments to open the Chromeleon Instrument Configuration Manager program.

c) Select *Controller*, Connect to *Instrument Controller* on the drop-down list. Select *My Computer*, *Chromeleon Instrument Controller*, and OK button.

9.3.3 Creating an IC-MS configuration with Integrion HPIC system, Dionex AS-AP autosampler, one Dionex auxiliary AXP-MS pumps, and ISQ EC mass spectrometer.

a) The configuration includes: Dionex Integrion HPIC system, Integrion HPIC pump wellness module, Dionex AS-AP autosampler, Dionex AXP-MS Auxiliary pump, ISQ EC mass spectrometer.

b) Add Integrion module by right-clicking on computer name, select *Add an Instrument*, and enter an appropriate name (for example: Integrion_ISQ_Application).

c) Right-click on Instrument and select *Add a module, IC: Dionex Integrated Systems, Integrion HPIC System, and OK button. (Figure 9)*

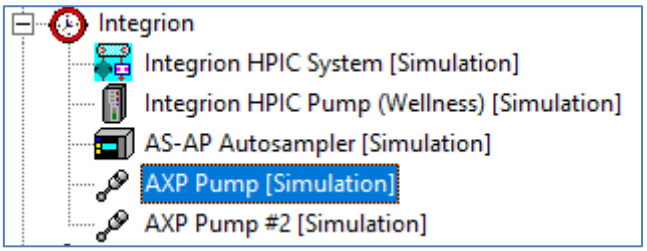


Figure 9: Adding modules to the Integrion instrument

d) Follow the instructions in Table 10 and select the OK button.

Tab	Action	Result
General	Link to USB address	
Inject Device	Click boxes InjectValve and HP_Valve	Activates both valves for use with IC-MS applications. The HP_Valve is used as a diverter valve between the IC and MS instruments
	Double left-click on InjectValve	Opens the device configuration information on the injection valve
	Select Autosampler	Changes control of the injection valve from the autosampler to a programmed command by the Integrion IC
	HP_Valve	Used as a diverter valve
TTL Inputs	Click on box TTL_1_In,	Active TTL_1_In channel
	Double click on TTL_1_In, select Inverted Edge on drop down menu. Click box Pump On.	Selects pump to turn off when activated

Table 10: Add Dionex Integrion HPIC module

e) Add the Dionex Integrion HPIC Pump Wellness module by selecting *Add a module, IC: Dionex Integrated Systems, Integrion HPIC (pump) Wellness, and OK button*

f) Follow the instructions in Table 11 and select the OK button.

Tab	Action	Result
General	Link to USB address	
Devices	Click box Pressure Signal(s)	Activates monitoring of Integrion HPIC pump pressure channel

Table 11. Add Dionex Integrion HPIC Pump Wellness module

g) Add Dionex AS-AP Autosampler by selecting *Add a module, IC: Dionex Integrated Systems, AS-AP module, and OK button*

h) Follow the instructions in Table 12 and select the *OK button*.

Tab	Action	Result
General	Link to USB address	
Sharing		Select Instrument name(s). This option is available if more than one instrument is detected
Segments / Pump Link	Well Plate Sampler Segment Types	Select size and type of vials in each color segment, for example, 10 mL polystyrene vials or 1.5 mL vials for “Red”, “Blue”, and “Green”
Options	Injection Mode	Select Push
	Select Buffer size	Typically, 1200 µL
	Select Syringe size	Typically, 100, 250, or 1000 µL
	Loop size	Enter injection Loop size (µL)
	Temperature control	Mark box, set limits to include application temperature
Relays	Click on Relay 4 box	Activates Relay 4 for Contact Closure (only needed for SII for Xcalibur)

Table 12. Add Dionex AS-AP module

i) Add Dionex Auxiliary pump AXP-MS module by selecting *Add a module, IC: Dionex Modules, AXP Pump, and OK button (Figure 10).*

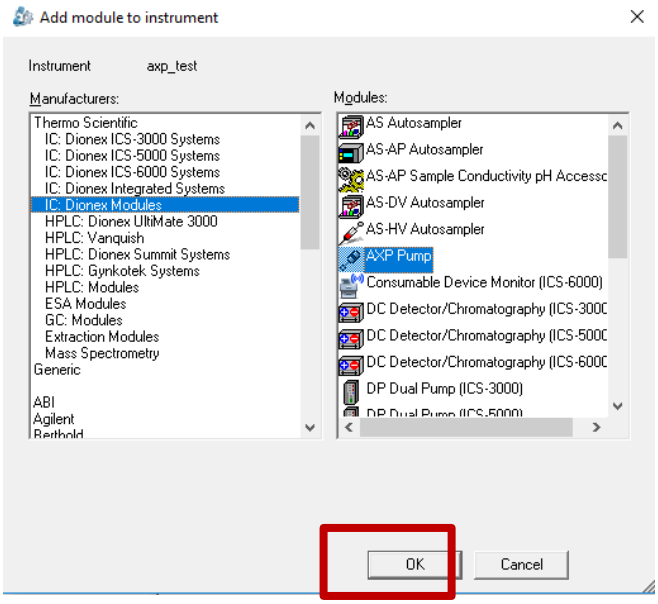


Figure 10. Adding AXP pumps to the instrument

j) Follow the instructions in Table 13, General Tab to select the correct COM channel, and select the *OK button*.

Tab	Action	Result
General	Select COM channel identified in Section 9.2. Select OK	If correct, no error messages will appear in Messages Instrument Controller within 1 min. If error messages occur
	Device Name	Rename pump to Pump_MS
Pump Type	Select AXP-MS	Sets the pump type and flow limits

Table 13: Add the Auxiliary AXP-MS pump module

k) After clicking OK, the connection will try to initiate the AXP driver and messages will appear in the Instrument Controller panel, while still in Instrument configuration. See Figure 11.

l) Wait approximately 30 seconds. If the initiation is correct, no messages will appear after Initiating Driver AXP Pump.

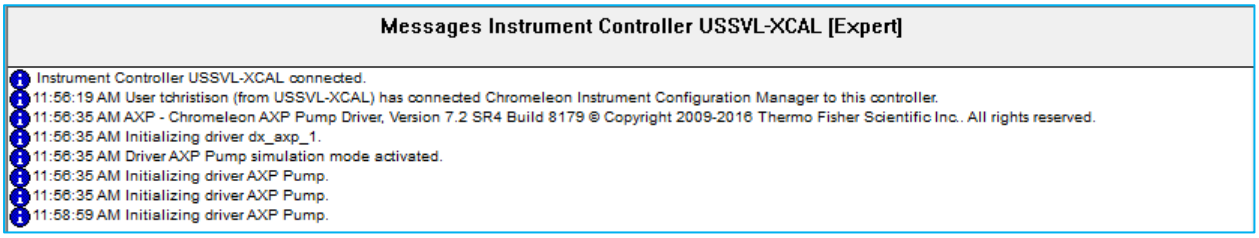


Figure 11: Initiating the AXP pump driver

m) If the initiation is incorrect, an error message will occur. Return to steps described Section 9.2 Finding the COM port. Select the other COM channel. Click **OK**. Wait about 30 seconds to ensure that no error messages occur.

n) Add ISQ EC mass spectrometer module by selecting *Add a module, Mass Spectrometry, ISQ ICMS family, and OK button*

o) Follow the instructions in Table 14, General Tab and select the *OK button*.

Tab	Action	Result
General	Deselect Hardware inject synchronization	Do not select. Chromeleon controls injection
	Select ActiveLow for Remote Start	Setting for Dionex AS-AP autosampler
	Select ISQ EC	Select the correct MS

Table 14: Add the ISQ ICMS module

9.4 Chromeleon: IC-MS system with Dionex Integrion coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer

9.4.1 Find the COM port the AXP and AXP-MS pumps by following Section 9.2. Rename them with unique names.

9.4.2 Open the Chromeleon Configuration Manager program, as described in 9.3.2

9.4.3 To create the IC-MS configuration

a) Add the following IC modules: Dionex Integrion HPIC system, Dionex Integrion HPIC Pump Wellness module, two AXP pump modules (AXP-MS and AXP pumps).

b) Specific information for each module is described in Section 9.3.3

b) Add the TSQ mass spectrometer module by selecting *Add a module, Mass Spectrometry, Mass Spectrometer, and OK button*

9.5 Chromeleon: IC-MS system with Dionex ICS-5000+ or ICS-6000 coupled with Thermo Scientific ISQ EC mass spectrometer

9.5.1 Find the COM port the AXP and AXP-MS pumps by following Section 9.2

9.5.2 Open the Chromeleon Configuration Manager program by following Section 9.3.2

9.5.3 Creating an IC-MS configuration with Dionex ICS-5000+ or ICS-6000 system, an AXP-MS pump, and ISQ EC single quadrupole mass spectrometer.

a) The configuration includes: Dionex ICS-5000+ or ICS-6000 HPIC system, including the DP, EG, DC modules, Dionex AS-AP autosampler, Dionex AXP-MS Auxiliary pump, Dionex AXP pump, ISQ EC mass spectrometer.

b) Add ICS-6000 system by right-clicking on computer name, select *Add an Instrument*, and enter an appropriate name (for example: ICS-6000_ISQ_Application).

c) Right-click on Instrument name and select *Add a module, IC: ICS-6000 Systems, DP Dual Pump, and OK button (Figure 12).*

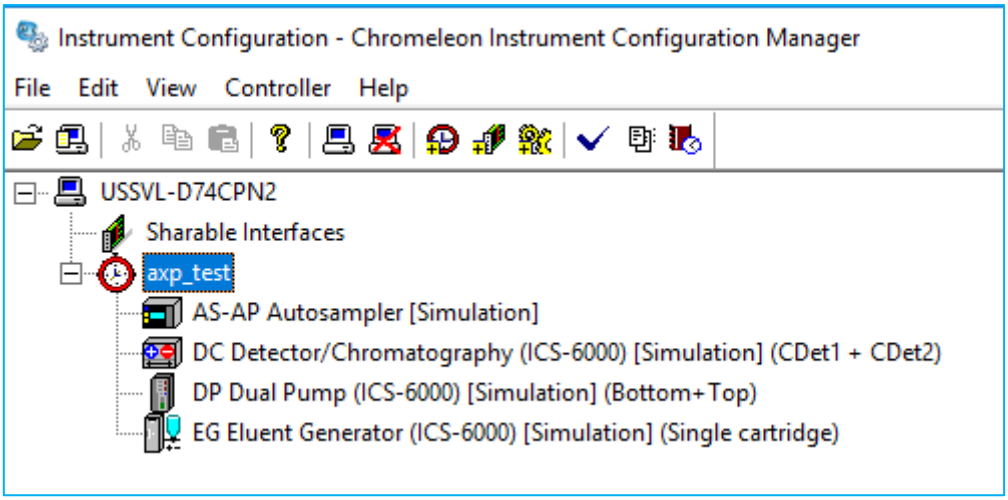


Figure 12. Adding modules to the instrument

d) Follow the instructions in Table 15 and select the OK button.

Module	Tab	Action
DP	General	Select Browse, select serial number to link module to Instrument
	Device	Link pump to instrument
EG	General	Select serial number to link module to Instrument
	Cartridges	Link to instrument. Check EGC_1 and link to Pump 1
DC	General	Select serial number to link module to Instrument, Select Instrument
	Detectors	Double click on CD, Link to Pump 1
	Thermo Controls	Check Compartment_TC and Column_TC
	Suppressors	Double click Suppressor1, Link to Pump 1
	High Pressure Valves	Double click InjectValve1, Link to Pump 1, select control by autosampler. Double click InjectValve2, Link to Pump1, select control by DC
	Low Pressure valves	Not used in this application
AS-AP Autosampler	General	Select serial number to link module to Instrument
	Sharing	This option is present, select Instrument
	Segments / Pump Link	Select 10 mL PolyVials or 1.5ml vials for “Red”, “Blue”, and Green. Leave the pump and TTL links empty.
	Options	Select 1200 buffer loop, 125 µL syringe, enter “10” µL in sample loop, temperature control, push mode
	Relays	Not used in this application
	Inputs	Not used in this application
ISQ EC	General	Add the ISQ EC
		Deselect the hardware inject synchronization. Not needed as Chromeleon controls injection.
		Select ActiveLow for Remote Start. (Setting for Dionex AS-AP autosampler)

Table 15: Electronic Configuration Parameters for ICS-6000 coupled to ISQ EC Mass Spectrometer

e) Add the AXP-MS module according to the below Table 16 and as described in Section 9.3

Tab	Action	Result
General	Select COM channel identified in Section 8.4. Select OK	If correct, no error messages will appear in Messages Instrument Controller within 1 min. If error messages occur
	Device Name	Rename pump to Pump_MS
Pump Type	Select AXP-MS	Sets the pump type and flow limits

Table 16: Add the AXP-MS module

9.6 Chromeleon: IC-MS system with Dionex ICS-5000+ or ICS-6000 HPIC System coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer

9.6.1 Find the COM port the AXP and AXP-MS pumps by following Section 9.2

9.6.2 Opening Chromeleon Configuration Manager program, see 9.2.1

9.6.3 Creating an IC-MS configuration with ICS-5000 or ICS-6000 HPIC system, and TSQ triple quadrupole mass spectrometer.

a) The configuration includes: Dionex ICS-5000+ or ICS-6000 HPIC system, including the DP, EG, DC modules, Dionex AS-AP autosampler, Dionex AXP-MS Auxiliary pump, Dionex AXP pump, and generic mass spectrometer

b) Add ICS-6000 system by right-clicking on computer name, select *Add an Instrument*, and enter an appropriate name (for example: ICS-6000_TSQ_Application).

c) Right-click on Instrument and select *Add a module, IC: ICS-6000 Systems, DP Dual Pump, and OK button*

d) Add the other modules including the AXP-MS pump according to section 9.3.3

e) Add TSQ mass spectrometer module by selecting *Add a module, Mass Spectrometry, Mass Spectrometer, and OK button*

9.7 SII for Xcalibur: IC-MS system with Dionex Integrion coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer or Thermo Scientific Hybrid Orbitrap-Quadrupole High Resolution Mass Spectrometer

9.4.1 SII for Xcalibur is a Chromeleon program embedded into Xcalibur and serves as the interface between Xcalibur and Chromeleon.

9.4.2 Find the COM port the AXP and AXP-MS pumps by following Section 9.2

9.4.3 Creating an IC-MS configuration with Dionex Integrion HPIC system, Dionex AS-AP Autosampler, and TSQ triple quadrupole mass spectrometer.

a) Close all open Xcalibur programs

b) Open Xcalibur configuration program (gear symbol)

c) Click on Thermo Scientific SII for Xcalibur. See 13.

d) Click on Configure Device. See Figure 14.

-

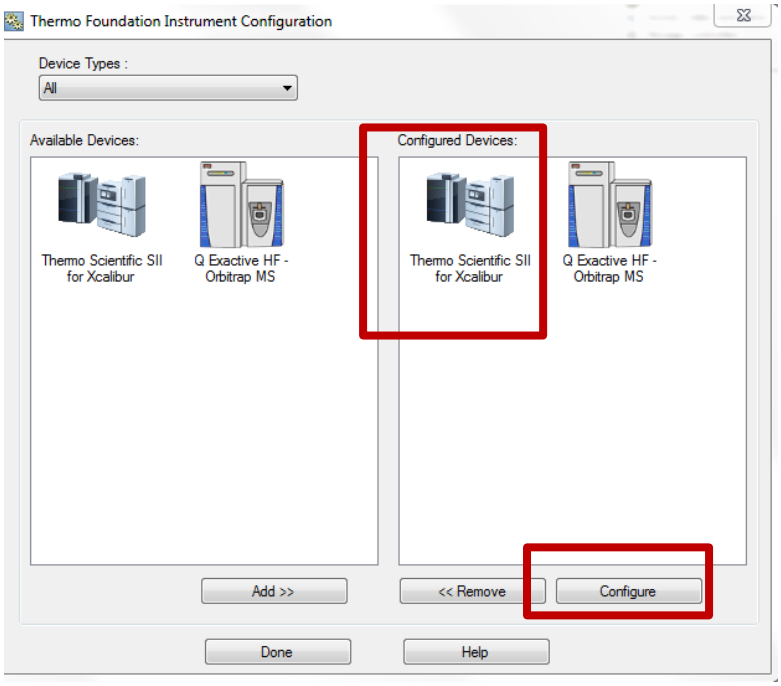


Figure 13. Configuring the instrument using SII for Xcalibur

e) Another SII for Xcalibur Configuration window will open. Click on **Configure Device**. See Figure 14. Click **OK**.

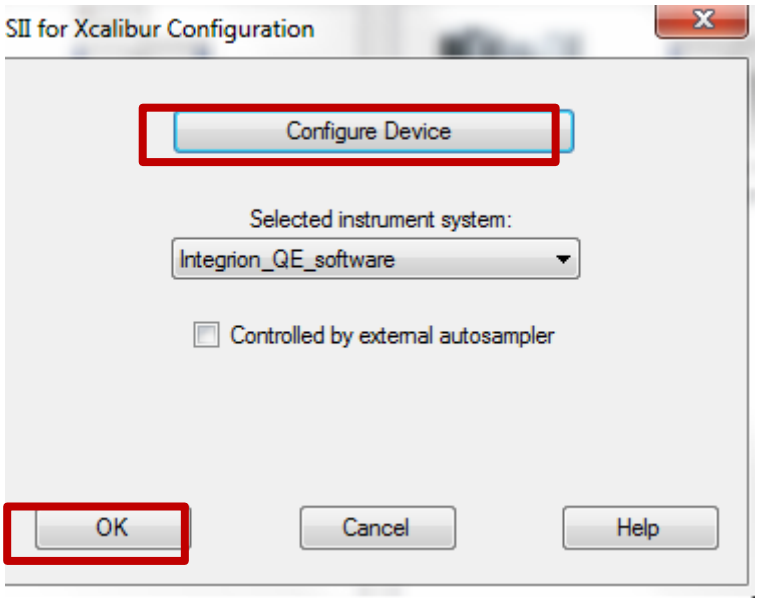


Figure 14. Configuring Devices

f) Create an Instrument Configuration by adding these modules: Integrion HPIC Instrument, Integrion HPIC Pump wellness, two auxiliary pump modules for the AXP and AXP-MS pumps, AS-AP autosampler and generic mass spectrometer module.
Follow Section 9.4 for detailed instructions.

Relay 4 is needed for Contact Closure and is activated in the Instrument Configuration, Dionex AS-AP autosampler module. To activate Relay 4, follow the instructions below in Table 17. Installation of the Contact Closure cable is discussed in Section 4.3.

Tab	Action	Result
General	Link to USB address	
Sharing		Select Instrument name(s). This option is available if more than one instrument is detected
Segments / Pump Link	Well Plate Sampler Segment Types	Select size and type of vials in each color segment, for example, 10 mL polystyrene vials or 1.5 mL vials for “Red”, “Blue”, and “Green”
Options	Injection Mode	Select Push
	Select Buffer size	Typically, 1200 µL
	Select Syringe size	Typically, 100, 250, or 1000 µL
	Loop size	Enter injection Loop size (µL)
	Temperature control	Mark box, set limits to include application temperature
Relays	Click on Relay 4 box	Activates Relay 4 for Contact Closure (only needed for SII for Xcalibur)

Table 17. Instrument Configuration of Dionex AS-AP autosampler module for Contact Closure

9.8 SII for Xcalibur: IC-MS system with Dionex ICS-5000+ or ICS-6000 HPIC System coupled with Thermo Scientific TSQ triple quadrupole mass spectrometer or Thermo Scientific Hybrid Orbitrap-Quadrupole High Resolution Mass Spectrometer

9.8.1 Find the COM port the AXP and AXP-MS pumps by following Section 9.2

9.8.2 Creating an IC-MS configuration with Dionex ICS-5000+ or ICS-6000 HPIC system, Dionex AS-AP autosampler, and TSQ triple quadrupole mass spectrometer.

a) Close all open Xcalibur programs

b) Open Xcalibur configuration program (gear symbol)

c) Click on Thermo Scientific SII for Xcalibur. See Figure

d) Click on **Configure Device**. See Figure . Click **OK**.

e) Create an Instrument Configuration by adding these modules: ICS-6000 HPIC system DP, EG and DC modules, two auxiliary pump modules for the AXP and AXP-MS pumps, AS-AP autosampler, and generic mass spectrometer module. See Section 9.5

f) Relay 4 is needed for Contact Closure and is activated in the Instrument Configuration, Dionex AS-AP autosampler module. To activate Relay 4, follow the instructions below in Table 18. Installation of the Contact Closure cable is discussed in Section 4.3

Module	Tab	Action
DP	General	Select Browse, select serial number to link module to Instrument
	Device	Link pump to instrument
EG	General	Select serial number to link module to Instrument
	Cartridges	Link to instrument. Check EGC_1 and link to Pump 1
DC	General	Select serial number to link module to Instrument, Select Instrument
	Detectors	Double click on CD, Link to Pump 1
	Thermo Controls	Check Compartment_TC and Column_TC

	Suppressors	Double click Suppressor1, Link to Pump 1
	High Pressure Valves	Double click InjectValve1, Link to Pump 1, select control by autosampler. Double click InjectValve2, Link to Pump1, select control by DC
	Low Pressure valves	Not used in this application
AS-AP Autosampler	General	Select serial number to link module to Instrument
	Sharing	This option is present, select Instrument
	Segments / Pump Link	Select 10 mL PolyVials or 1.5ml vials for “Red”, “Blue”, and Green. Leave the pump and TTL links empty.
	Options	Select 1200 buffer loop, 125 µL syringe, enter “10” µL in sample loop, temperature control, push mode
	Relays	Activate Relay 4 by clicking on the box
	Inputs	Not used in this application
MS	General	Add the generic mass spectrometer
		Deselect the hardware inject synchronization. Not needed as Chromeleon controls injection.
		Select ActiveLow for Remote Start. (Setting for Dionex AS-AP autosampler)

Table 18. Electronic Configuration Parameters for ICS-6000 coupled to ISQ EC Mass Spectrometer

9.9 Two-Software (Chromeleon + Xcalibur) Installation

For IC-MS installations it is common that the analyst may plan to switch from anions to cations or from IC-MS to HPLC-MS. As the MS can only accept one channel at a time, an effective solution would be to use a two-software installation: Chromeleon and SII for Xcalibur or Chromeleon and Xcalibur.

The software configuration can use SII for Xcalibur with Xcalibur by having two separate instrument configurations. However, the IC eluent generator system and the suppressor will undesirably turn-off when the configuration is switched to HPLC-MS. It is often more convenient to use Chromeleon independently for the IC-MS applications from Xcalibur for the HPLC-MS applications.

9.9.1 Case 1: Anion + Cation + HPLC with a single MS and single computer

Two software installation is typical when you need switch automatically between 2 or more lines connected to a single MS. This does not apply to the ISQ EC MS.

9.9.2 Case 2: IC and LC with MS and independently using two computers

In this example, the customer wants to operate with IC or LC with the MS but also stand alone. In this case, the MS is connected with one computer and chromatographic devices (IC and LC) are standalone systems controlled by a second computer.

All quantification and post-acquisition steps are managed by MS software. Chromeleon CDS is used only to control chromatographic hardware. Chromeleon CDS is installed on chromatographic computers without MS control

When using SII for Xcalibur the MS start command is generally performed by Relay 4 on the Dionex AS-AP autosampler. See Section 4.3 Installing Contact Closure Cables, 9.7 or 9.8 Electronic Configuration, and Section 13 Triggering Contact Closure. Figure 15 shows the Relay 4 commands entered in the Script Editor of the Instrument Method.

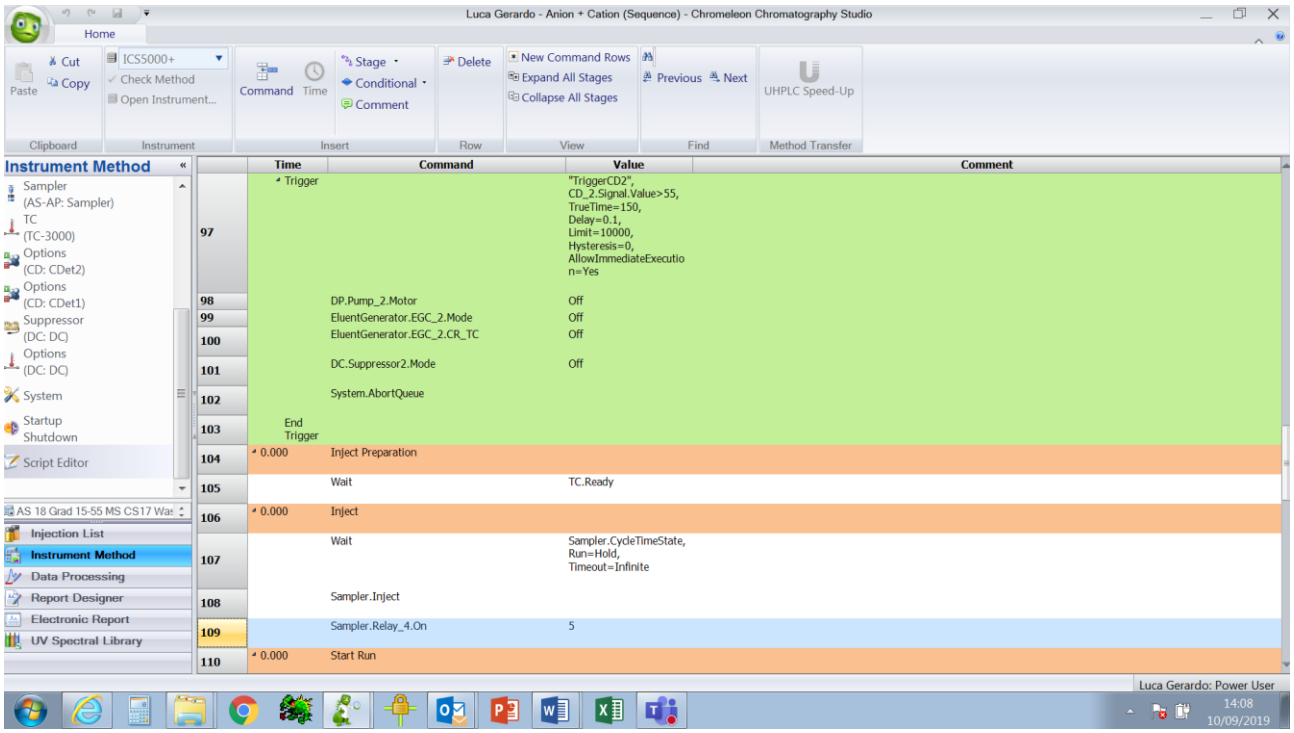


Figure 15. Sampler Relay commands

On Xcalibur instrument setup (MS PC), only MS is configured without IC or LC.
To start the sequence, select the “In Use Flag” (Figure 16).

Figure 16. Sampler Relay commands

9.9.3 Case 3: Automatic Switching to determine anions and cations by IC-MS

It is very common to run both anion and cation applications using the capabilities of the Dionex ICS-6000 HPIC modular dual channel IC system. To accommodate the single MS channel, the IC is configured with Chromeleon CDS on one or two timebases with the MS configured in Xcalibur.

Configure the sequence on Chromeleon CDS: typically, two lines on the same timebase, or 2 timebases

- Launch MS sequence (check that the numbers of lines will be equal to Chromeleon sequence)
  If using Chromeleon with two different timebases, the lines on MS sequence must be equal to Seq 1 + Seq 2 on Chromeleon.
  Xcalibur remains in “Waiting for contact closure” mode because flag on “start instrument” is not selected.
- Start Chromeleon sequence. “Sampler Relay 4_on” command starts the Dionex AS-AP autosampler, the autosampler injects to the IC, and then starts the IC and MS acquisition.
- The data from anion and cation chromatographic runs are directed to the MS software.

10. Using the Diverter Valve

10.1 Programming the Diverter valve to Direct IC Flow to MS

In analysis mode, the diverter valve should be directed to the MS. When the MS is not in analysis mode, direct the diverter valve flow to waste.

1. Click on the line below the end of DC instructions for convenience. This is highlighted in blue in Figure 17. Switching the valve is not enough to ensure safe operation. On the next injection, the valve will switch to the MS again and salt will run into the MS if the suppressor failed. This will happen until the trigger triggers again - in this case after 3 minutes. Thus, the stop of the analytical pump is necessary (or every other command that will set the system into "HOLD")

Time	Command	Value
◢ {Initial Time}	Instrument Setup	
◢ Trigger		"HighConductivity", CDet1.CD_1_Total.Signal>=1200, TrueTime=180, AllowImmediateExecution=Yes
	DC.InjectValve_2.LoadPosition	
	DP.Pump_1.Motor	off
	Click here to append a new command	
End Trigger		
	DC.Compartment_TC.AcquireExclusiveAccess	
	DC.Column_TC.AcquireExclusiveAccess	
	DC.Column_TC.Mode	On
	DC.Column_TC.TemperatureSet	30.00 [°C]
	DC.Compartment_TC.Mode	On
	DC.Compartment_TC.TemperatureSet	15.00 [°C]
	DC.IC_Cube2.Cube2_TC.Mode	On
	DC.IC_Cube2.Cube2_TC.TemperatureSet	35.00 [°C]
	DC.Suppressor1.Type	AERS_2mm
	DC.Suppressor1.Carbonate	0.0 [mM]
	DC.Suppressor1.Bicarbonate	0.0 [mM]
	DC.Suppressor1.Hydroxide	30.0 [mM]
	DC.Suppressor1.Tetraborate	0.0 [mM]
	DC.Suppressor1.OtherEluent	0.0 [mN]
	DC.Suppressor1.RecommendedCurrent	8 [mA]
	DC.Suppressor1.CurrentSet	8 [mA]
	DC.Suppressor2.Type	None
	DC.Suppressor2.Carbonate	0.0 [mM]
	DC.Suppressor2.Bicarbonate	0.0 [mM]
	DC.Suppressor2.Hydroxide	0.0 [mM]
	DC.Suppressor2.Tetraborate	0.0 [mM]
	DC.Suppressor2.OtherEluent	0.0 [mN]
	DC.Suppressor2.CurrentSet	0 [mA]
	EluentGenerator.EGC_1.Concentration	30.00 [mM]

Figure 17. Selecting a line to insert a command.

2. Click on “Insert Command” icon (Figure 18).

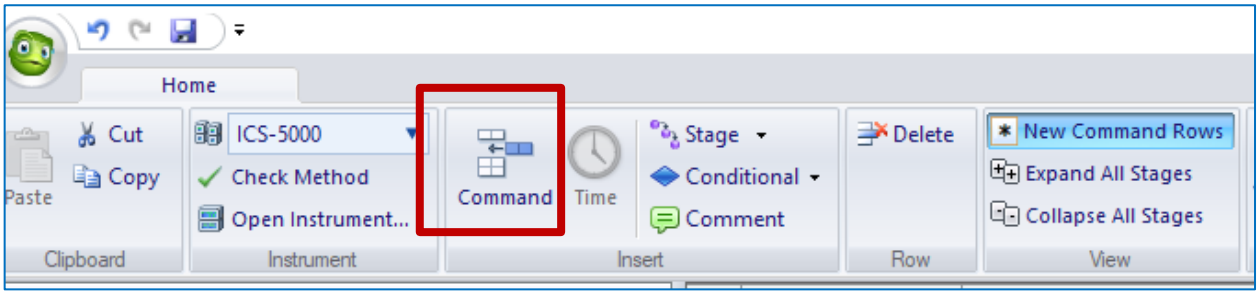


Figure 18. Insert Command

3. Enter command for diverter valve to be in the position to MS. Here I show the second injection valve of the ICS-6000 DC in the flow path to the MS (Table 19):

DC.InjectValve_2.LoadPosition

System	Valve	Command
ICS-6000	Extra valve	DC.InjectValve_2.LoadPosition
	Automation Manager	Enter AM_HP1.A
Integrion	Extra valve	Pump_ECD.HP_Valve.A

Table 19. Injection Valve Commands for Dionex ICS-6000 HPIC and Dionex Integrion HPIC Systems

See “Create Action,..” or “Add another action” for other valve commands.(Figure 19)

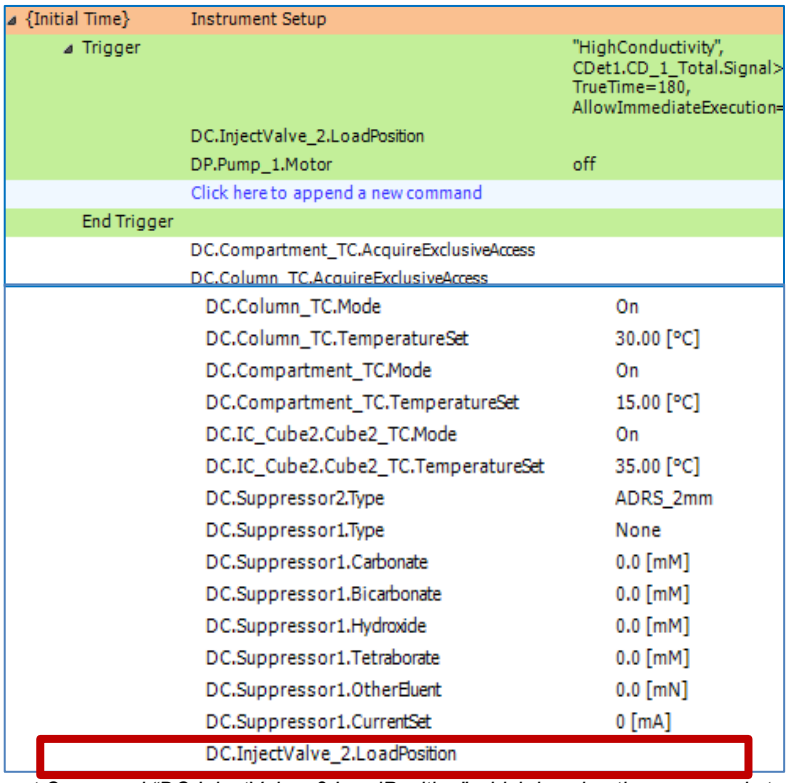


Figure 19. Insert Command “DC.InjectValve_2.LoadPosition” which is using the commands to use the second injection valve for Dionex ICS-6000 HPIC as the diverter valve.

10.2 Programming the Diverter valve to Direct IC Flow to Waste or Recycle

In analysis mode, the diverter valve should be directed to the MS. When the MS is not in analysis mode, direct the diverter valve flow to waste. Add commands near the end of the run time. Table 20 shows the valve commands based on the IC system and the valve selected as the diverter valve.

System	Valve	Command
ICS-6000	Extra valve	DC.InjectValve_2.InjectPosition
	Automation Manager	Enter AM_HP1.B
Integrion	Extra valve	Pump_ECD.HP_Valve.B

Table 20: Injection Valve Commands for Dionex ICS-6000 HPIC and Dionex Integrion HPIC Systems

1. Click on the line before the last time entrée and Stop Run (Figure 20).

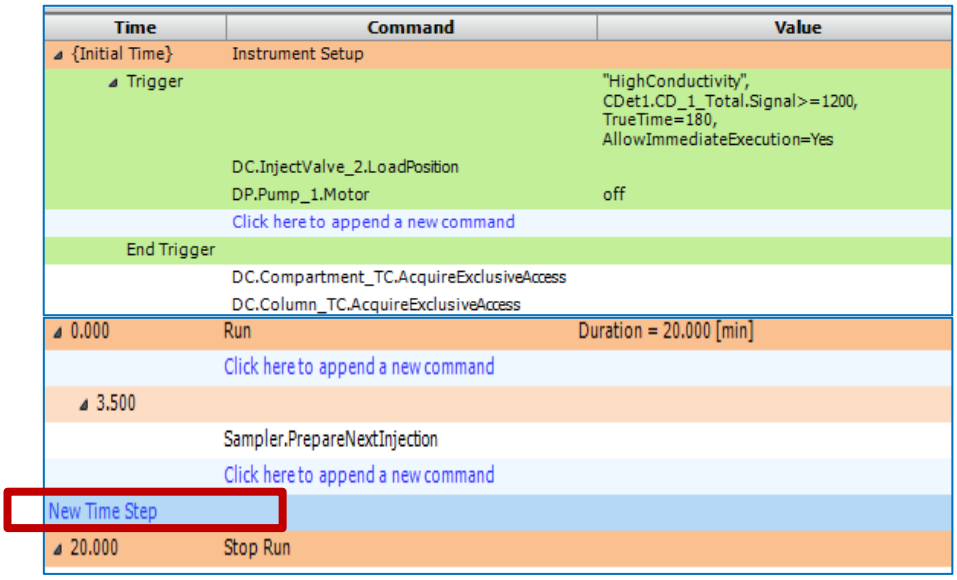


Figure 20. Insert "A new Time Step".

2. Right click and select "Insert Time"
3. Enter a time 0.1 – 1.0 min before the end of run.
   Note the Author entered 19.9 min
4. Enter command for diverter valve to be in the position to waste. Here I show the second injection valve of the ICS-6000 to direct the flow to waste or recycle mode:
   Example shown in Figure 21 uses the *DC.InjectValve_2.InjectPosition at 19.8 min*

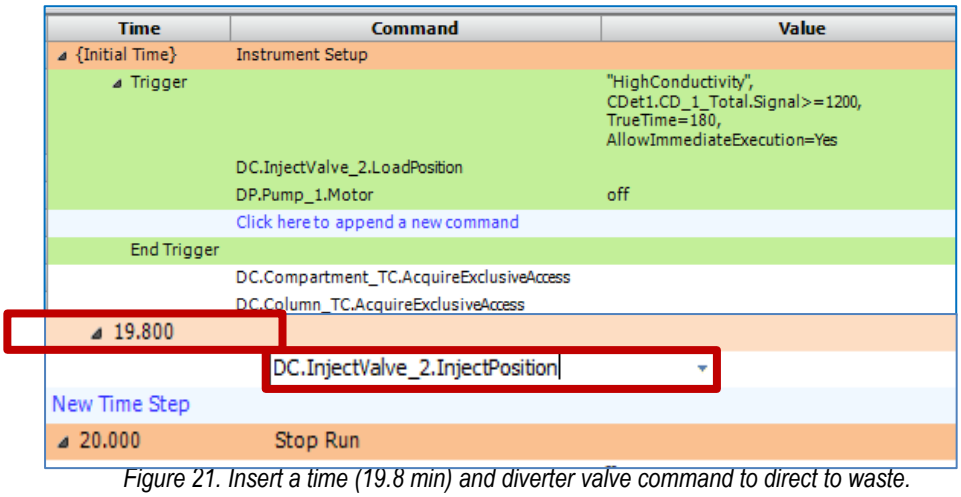


Figure 21. Insert a time (19.8 min) and diverter valve command to direct to waste.

5. Save file

11. Triggers, Emergency Methods and TTL connections

Triggers and TTL connections are used here to create emergency methods.

11.1 TTL Settings

Some devices are “normally closed” with running, that is the circuit is closed. Some devices are “normally open” when running. The TTL setting on the IC must match the devices’ type (Table 21)

TTL Setting	AXP Pins
Normal Edge	Normally Closed
Inverted Edge	Normally Open

Table 21: TTL Settings with Normally Open and Normally Closed Pin Positions

11.2 Using the AXP Low Pressure Error Trigger

AXP pump (since 2016):
Switching off the pump does NOT trigger the TTL. The TTL triggers when it is turned on instead.

Conditions:
Instrument is in idle
System is running
Sequence hasn’t started, completed, or interrupted.
Suppressor is in external water mode
AXP loses pressure (water runs out, stalls, loses prime, bubbles lodge in pump)
Error: the AXP pump issues a low-pressure error
Trigger: Turn-off eluent pump and therefore turn-off electrolytic devices, and stop flow to MS. See Table 22.

IC system	TTL	AXP pump (pumping suppressor regen)	AXP pump	Action
Integrion	TTL_1_In, Inverted Edge, Pump On	Pin 6 Common relay (black), Pin 8 Normally Open (Red)	Normally open	AXP Low Pressure triggers Integrion pump to turn-off
ICS-6000	TTL_2_In, Pin 11 ground (black wire), Pin 23 (red wire)	Pin 6 Common relay (black), Pin 8 Normally Open (Red)	Normally open	AXP Low Pressure triggers both pumps to turn-off

Table 22. TTL Settings for AXP Pump to Turn-off the IC Pump

Example shown in Figure 22 shows activating the TTL commands in Instrument Configuration (to use with “Normally open” connected relay)

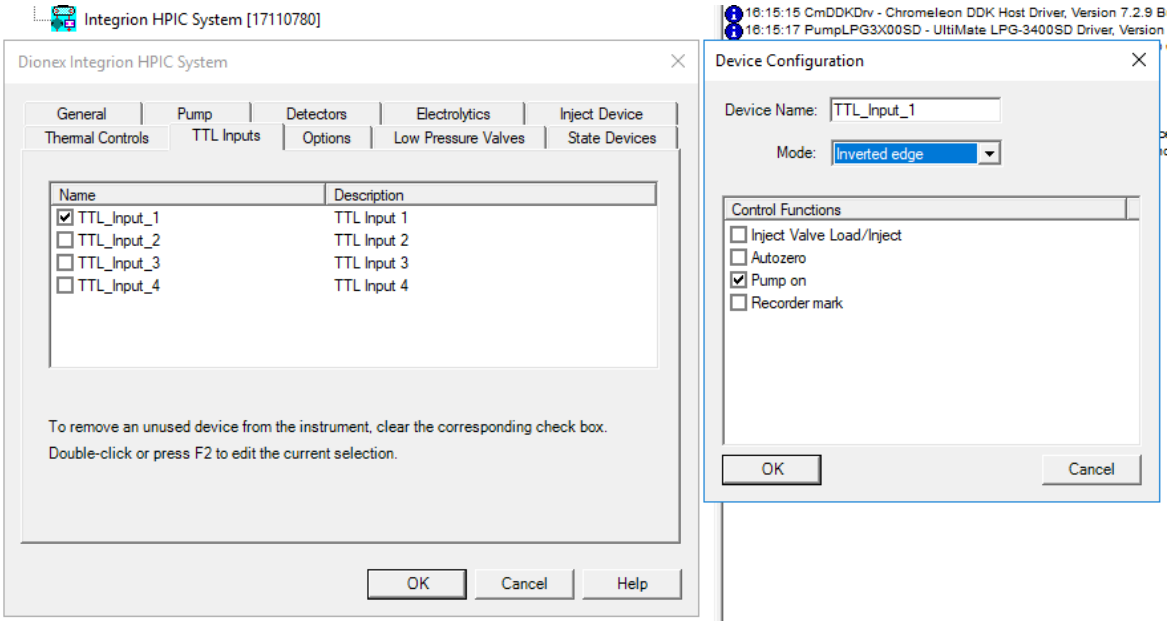


Figure 22. Activating the Integrion TTL_Input_1 channel the AXP low pressure limit trigger

11.3 Using A High Conductivity Trigger

Conditions:

Instrument is running a sequence

AXP fails to deliver regen water to suppressor resulting in the suppressor failing or Suppressor fails in another mode

Total conductivity increases to a high level and doesn't return to baseline

Trigger: High conductivity conditions are met. Triggers action to execute, in this case rotates diverter valve to direct eluent flow to waste or to recycle mode.

Second actions: Turn-off eluent pump and therefore turn-off electrolytic devices. Can also be directed to turn ISQ EC MS vaporizer temperature to "0" (room temperature).

Setting the system to "HOLD" is mandatory if no emergency method is controlling the system.

Open Script Editor

1. Open instrument program
2. Select Script Editor
3. Select the number 4 on line under {Time} (Figure 23).

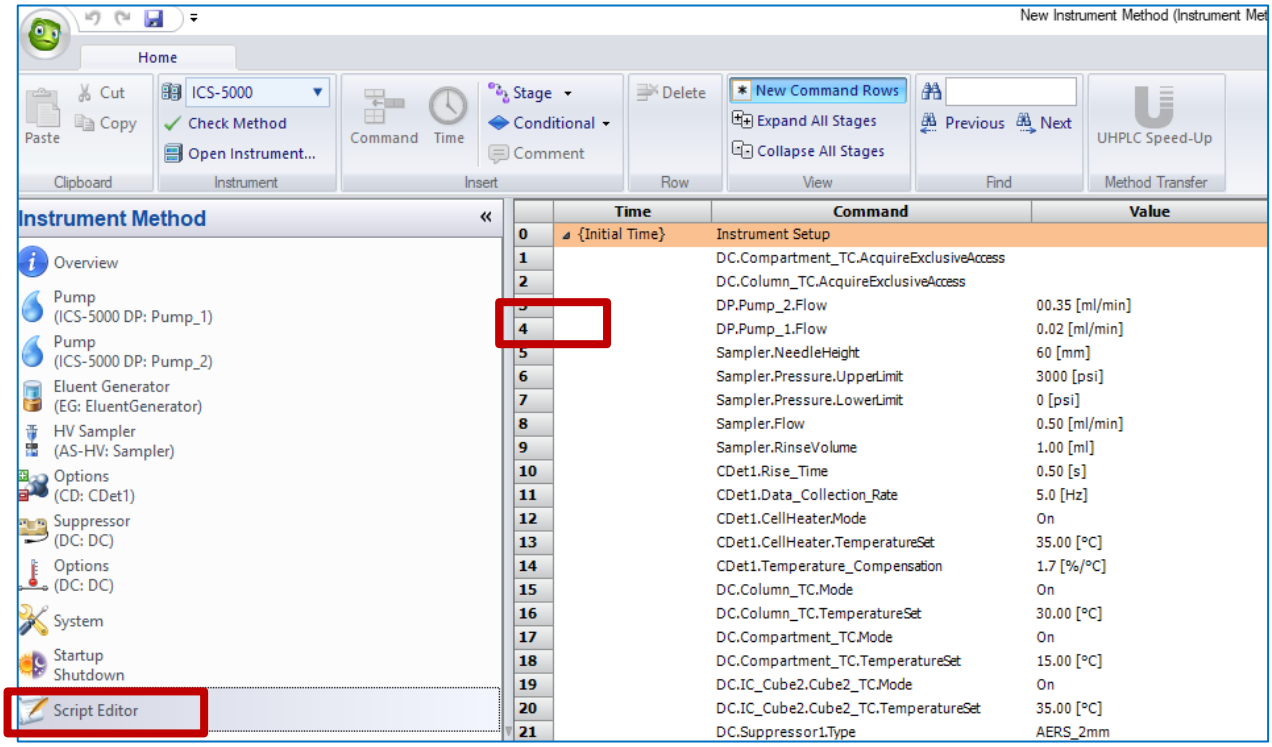


Figure 23. Insert a New Command to Create a High Conductivity Trigger

Table 23 lists the different commands used by the ICS-6000 and the Integrion IC systems for Conductivity Detection signal.

System	Conditions	Time	AllowImmediate
ICS-6000	CDet.CD_1_total.signal>=50	180	yes
Integrion	CDet.CD_total>=50	180	yes

Table 23. Commands for the Conditions section of the High Conductivity Trigger

Note: This trigger is not suitable for acetate eluents. For acetate eluents, conductivity may reach up to 1000 µS (caused by acetic acid).

Creating a "HighConductivityTrigger" (Figure 24)

1. Select Conditional and pull down menu. Select "Insert Trigger"
2. Enter conditions for trigger
3. Name: "HighConductivityTrigger"
4. Conditions
5. Press OK

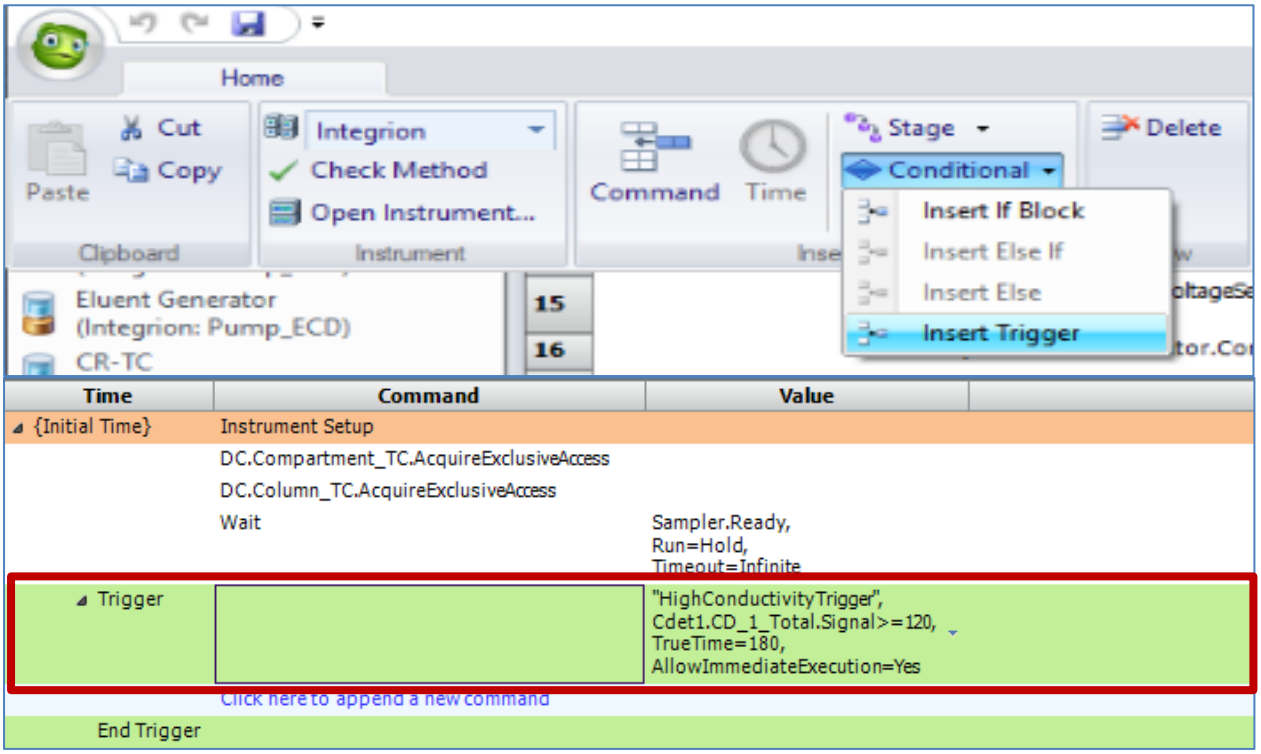


Figure 24. Using a Conditional and Insert Trigger commands to create the High Conductivity Trigger.

Note: Preferred total conductivity would be "50". This can be set higher if you have high chloride and sulfate in the sample. Adjust it lower as soon as your sample preparation consistently brings the total conductivity signal (CD_total) to a lower valve.
Note2: I am assuming that CD1 is your ICS-6000 detector. If it is CD2, change command to Cdet.CD_2_total.signal>=50

Create Action if Trigger Conditions are met

Table 24 lists the different commands used by the ICS-6000 and the Integrion IC systems to program the valve selected as the diverter valve to rotate and direct the eluent flow away from the MS (waste or recycle mode). Setting the system to “HOLD” is mandatory if no emergency method is controlling the system.

System	Valve	Command
ICS-6000	Extra valve	DC.InjectValve_2.InjectPosition
	Automation Manager	Enter AM_HP1.B
Integrion	Extra valve	Pump_ECD.HP_Valve.B

Table 24. Action Commands for the High Conductivity Trigger

1. Click on asterisk line (“click here,…”
2. Example in Figure 25, command for first high pressure valve in Automation Manager that is acting as the diverter valve.

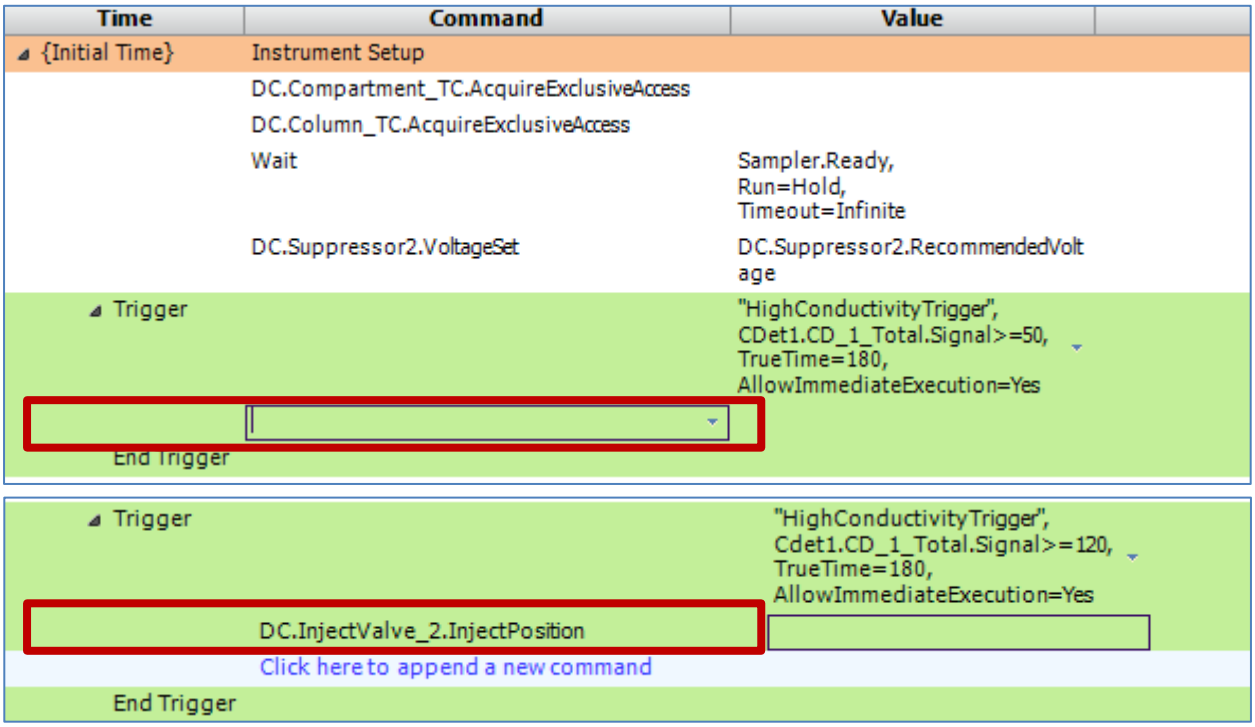


Figure 25. Using the High Conductivity Trigger to rotate the diverter valve to the Inject Position (DC.InjectValve_2.InjectPosition when the trigger conditions are met

Note: Check Automation Manager valve to verify that position A is to MS and position B is to waste.
Note2: command may be DC.AM_HP1.B

Add another Action

Multiple commands can be used as actions after meeting the High Conductivity Trigger conditions. It is recommended to execute the diverter valve rotation to waste first. Followed by any other commands in any order. The exception is any action that stops the eluent pump flow must be the last command. Any actions after initiating a stop pump or pump off command will be disregarded as the IC goes to "hold".

System	Action	Command	Value
Chromeleon CDS Software only with ISQ EC Mass Spectrometer			
Dionex Integrion HPIC with ISQ EC	Cools down MS vaporizer to standby conditions	ISQEC.ISQEC_MethodSettings.VaporizerTemperture	0
Chromeleon CDS Software and SII for Xcalibur software			
Dionex Integrion HPIC	Stops Integrion pump flow	System.Stop.Flow	
Dionex ICS-6000 HPIC	Stops Pump_1 or Pump_2 which in turn, stops eluent generation, CR-TC trap column, and suppressor	DP.Pump_1.motor	off
Dionex ICS-6000 HPIC		DP.Pump_2.motor	off

Table 22. Additional commands Using the High Conductivity Trigger to turn-off the DP_1 pump when the trigger conditions are met

Example in Figure 26 shows adding an extra action command to turn off the DP_1 pump motor. In this example the second injection valve (InjectValve_2) is being used as the diverter valve. DP_1 is the eluent pump.

Click on asterisk line ("click here,…"

1. To turn-off pump which will turn-off EGC, CR-TC trap column and suppressor,
    1. Enter DP.Pump_1.Motor
    2. Enter "off" in Value column
2. Save instrument program

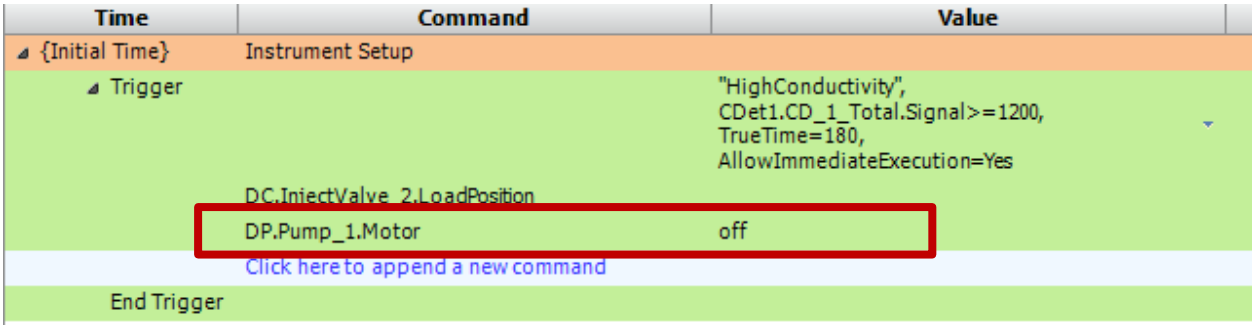


Figure 26. Using the High Conductivity Trigger to turn-off the DP_1 pump when the trigger conditions are met

Note: I am assuming that Pump 1 is the eluent pump.

Note2: The pump-off command must be the last command. Other commands following this command will not be initiated during an emergency shutdown.

Reset the trigger

To reset the trigger, clear the error by refilling the DI water containers, priming the AXP pump as discussed in Section 8 and the eluent pumps. Restart the IC system.

11.4 Creating a Virtual Channel to monitor suppressors

A Virtual Channel, programmed into the Instrument Method, creates a new stream of continuous data on a defined parameter. In this case, Virtual Channel will be used to monitor the voltage draw of the ERS 500e suppressor. A suppressor's voltage will increase over time as the suppressor ages, accumulates contamination, or starts to fail. A significant deviation from the voltage of the suppressor at installation is an indication that the suppressor needs cleaning or might be failing.

Suggested limit: The suppressor may need to be cleaned when the voltage is > 2 V higher than the initial voltage at installation.

Applicable to Suppressor Types:

- Monitor voltage in constant current suppressors: ERS 500, ERS 500e, and DRS 600 in constant current mode (legacy mode)
- Monitor current in constant voltage suppressors: DRS 600 in constant voltage mode (dynamic mode)

Dionex DRS 600 Dynamic Suppressors: A Virtual Channel can also monitor the current draw. Limits are to be defined in the future.

Limitations:

- At this time, only defined for ERS 500e suppressors.
- The voltage limit will likely be changed in the future. Chromeleon automatically limits the current draw to the max. current allowed for the suppressor if it is exceeded. CM automatically switches to constant current mode and resumes to constant voltage mode if the max. current of the suppressor is no longer exceeded
- The Virtual Channel is not programmed to turn-off the IC pump.
- A Virtual Channel may not visible in SII for Xcalibur.

The commands needed to create a Virtual Channel for the constant current or constant voltage suppressors in the Integrion and ICS-6000 IC systems are shown in Table 23.

System	Suppressor	Mode	Formula	Units*

Dionex Integrion HPIC	ERS 500, 500e	Legacy or constant current	Electrolytics.Suppressor.voltage	“V”
	DRS 600	Legacy or constant current	Electrolytics.Suppressor.voltage	“V”
	DRS 600	Dynamic or constant voltage	Electrolytics.Suppressor.current	“mA”
Dionex ICS-6000 HPIC	ERS 500, 500e	Legacy or constant current	dc.Suppressor1.voltage (Suppressor 1 used as an example)	“V”
	DRS 600	Legacy or constant current	dc.Suppressor2.voltage (Suppressor 1 used as an example)	“V”
	DRS 600	Dynamic or constant voltage	dc.Suppressor1.current (Suppressor 1 used as an example)	“mA”

*Type: Digital; Evaluate: No

Table 23: Virtual Column commands to monitor suppressors

To create a virtual channel:

1) Open the Script Editor on the Instrument Method pane.

2) Place cursor on row after 0.000 Start Run.

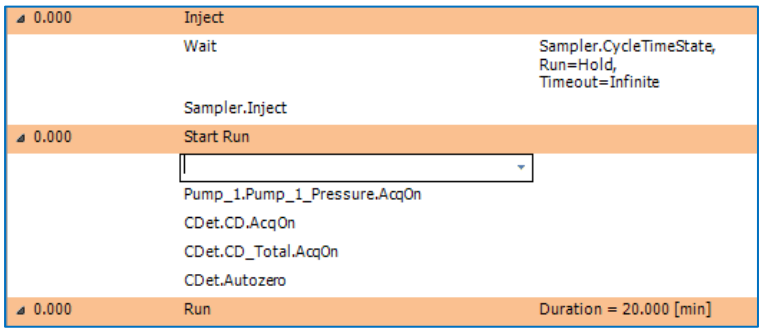


Figure 27. Creating a Virtual Channel, by creating a new command line

3) Select **Insert Command**. Insert a Command and Type in VirtualChannel.

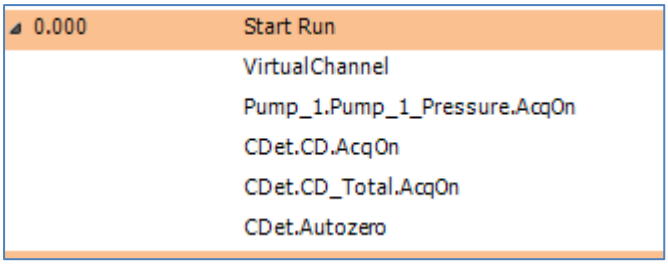


Figure 28. Creating a Virtual Channel, insert the command VirtualChannel

Example: Figure 29 shows the example of a constant current suppressor in the Integrion HPIC system.

4) Tab to the right, and type in

a. Name: “Suppressor_Voltage”
b. Formula: Electrolytics.Suppressor.voltage
c. Type: Digital
d. Unit: “V”
e. Evaluate: No

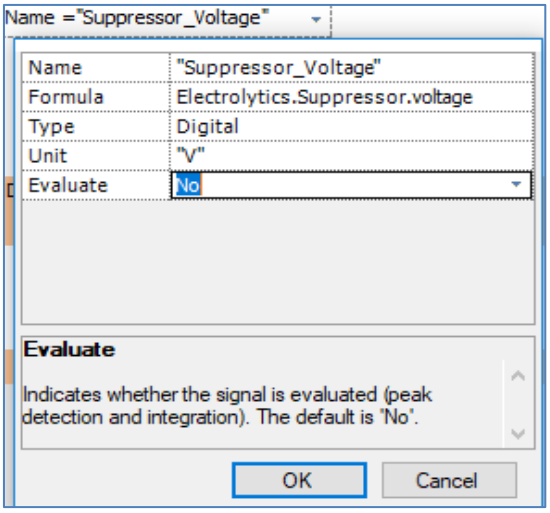


Figure 29. Entering the parameters for the Virtual Channel

5) Click **OK**.

12. Start Up and Shutdown Practices

This chapter describes good practices for startup and shut down of your IC system and then IC-MS system.

12.1 Start and condition IC system independently from the MS system

- Divert, disconnect, or stop any liquid flow from the IC to the MS system.
- Install the CD detector. Power on the modules. Start the Chromeleon or SII for Xcalibur. Create a configuration with Chromeleon Services Manager and Chromeleon Instrument Manager or SII for Xcalibur Configuration program.
- Install and condition the eluent generator cartridge and the CR-TC trap column according to the instructions under the Consumables download instructions on Chromeleon console (Chromeleon) or using Direct Instrument Control (SII for Xcalibur). Temporarily direct the flow to waste.
- Install and condition the guard and separation columns according to the product manual instructions which in most cases are using the QAR conditions. Temporarily direct the flow to waste.
- Hydrate and condition the suppressor without current or voltage, at < 10 mM eluent and 0.15 mL/min for 10 min. Allow 20 min for the membranes to swell, and then install the suppressor in external water mode into the IC.
- Turn on the IC pump and turn-on the eluent generator to the application flow rate and eluent concentration. If the column is a 4µm particle column, turn-on the column oven and increase the flow rate incrementally to the application conditions.
- **Within 10 min after pump flow**, turn-on the suppressor and the CR-TC trap column. Set suppressor to constant current with current setting or constant voltage with voltage setting from application conditions.
- Equilibrate the IC until the total conductivity < 2 µS/cm.
- Connect the flow exiting the IC diverter valve in the “A” position (from CD detector) to the MS.

12.2 Good start-up practices for IC-MS systems

- Verify that the IC is operating properly before directing the flow to MS.
- Check that the total conductivity is < 2 µS/cm.
- Mixing Tee: If a mixing tee is used in the application, rinse and check the mixing tee prior to analysis to ensure there are no flow restrictions or clogs.
- Verify the backpressure to the suppressor is < 150 psi prior to analysis.

12.3 Good shutdown practices for IC-MS systems

- At the end of the sequence while the MS is still running, it is advisable to rinse the MS probe by diverting the IC flow to waste, plumbing the desolvation solution or the external water directly to the grounding connector, and allowing 2-3 min of solvent (or water) to flow through the MS probe. Additionally, rinse the mixing tee so that it is ready for the next day.
- Rinse and check the diverter valve periodically for restriction or clogging.

13. Triggering Contact Closure

This chapter provides additional information about triggering contact closure using Chromeleon software.

13.1 Using Chromeleon software to trigger contact closure

Chromeleon controls the IC-MS system therefore Contact Closure is not needed to trigger the IC-MS system to start.

- Connect the USB cables from the MS to the computer or the switching box (which is connected to the computer).
- For Integrion, connect the USB cable from the autosampler to the Integrion system. Connect a USB cable from Integrion to the computer
- For the ICS-5000+ and ICS-6000 IC systems, connect the USB cables from the DP or SP module to the EG module, the EG module to DC module, and the DC module separately to the autosampler and to the computer.

13.2 Using SII for Xcalibur software to trigger contact closure

When using SII for Xcalibur software, Contact Closure is used to trigger the IC-MS system by triggering the Thermo Scientific™ Dionex™ ASAP Autosampler to start and run. After sample injection, the MS and the IC start acquiring data.

1) A twisted pair cable with two connectors is needed to connect the MS to IC autosampler (Section 4.3):
    a. Thermo Scientific™ Dionex™ AS-AP Autosampler:
        i. Use the 16-pin connector in the IC ship kits.
        ii. Use Relay 4 positions: Red wire in COM and black wire in NO (ground).
        iii. Activate Relay 4 in instrument configuration for the Dionex AS-AP autosampler module (Section 9.7 or 9.8)
    b. MS: usually uses the 2-pin connector
        i. For the Q Exactive MS family, connect the other end of the cable to the MS, Start In, Pins 1 and 2
        ii. For the TSQ Altis, Quantis, and Fortis MS family, connect the other end of the cable to the MS, Start In, Pins 1 (ground) and 4.
2) Connect the USB cables from the MS to the computer or the switching box (which is connected to the computer).
3) For Integrion, connect the USB cable from the autosampler to the Integrion system. Connect a USB cable from Integrion to the computer.
4) For the ICS-5000^{+} and ICS-6000 IC systems, connect the USB cables from the DP or SP module to the EG module, the EG module to DC module, and the DC module separately to the autosampler and to the computer.

13.3 Activating the Contact Closure Trigger in SII for Xcalibur

1) It not necessary to activate Relay 4 in instrument configuration (as previously done in the prior software interface).
2) Open Xcalibur.
3) Open **Sequence Setup**.
4) Click Ready to Download under the line Thermo Scientific SII for Xcalibur and click on Direct Control button. See Figure 30.

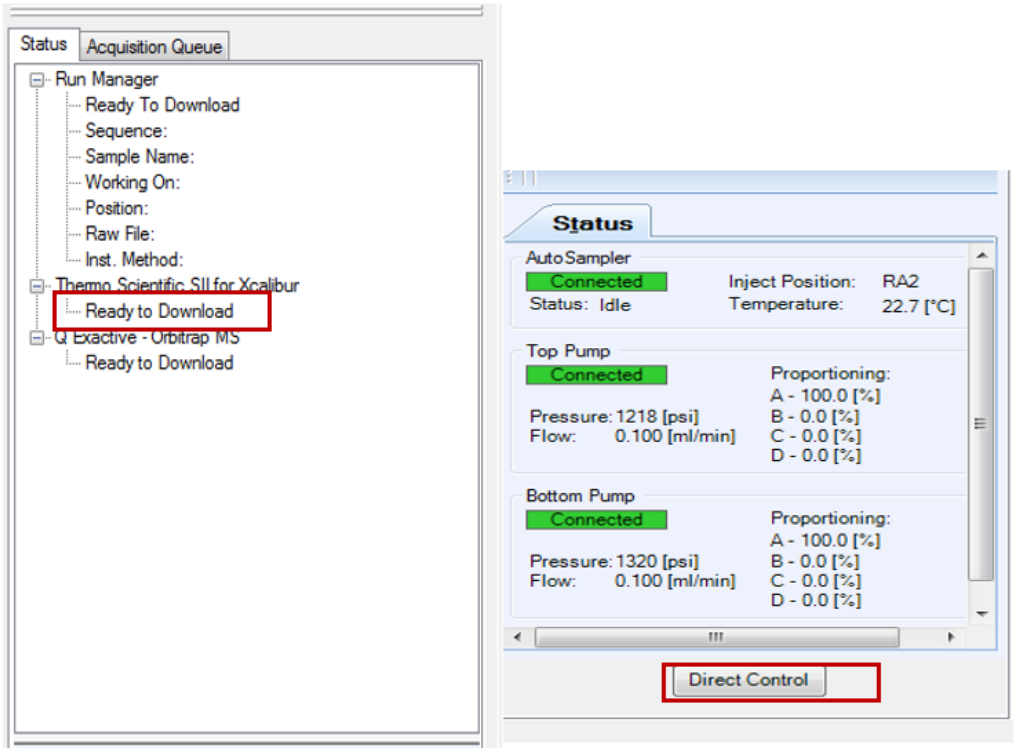


Figure 30. Open access to direct control of the IC instrument

5) This opens up Chromeleon 7 Console panels (Figure 31).

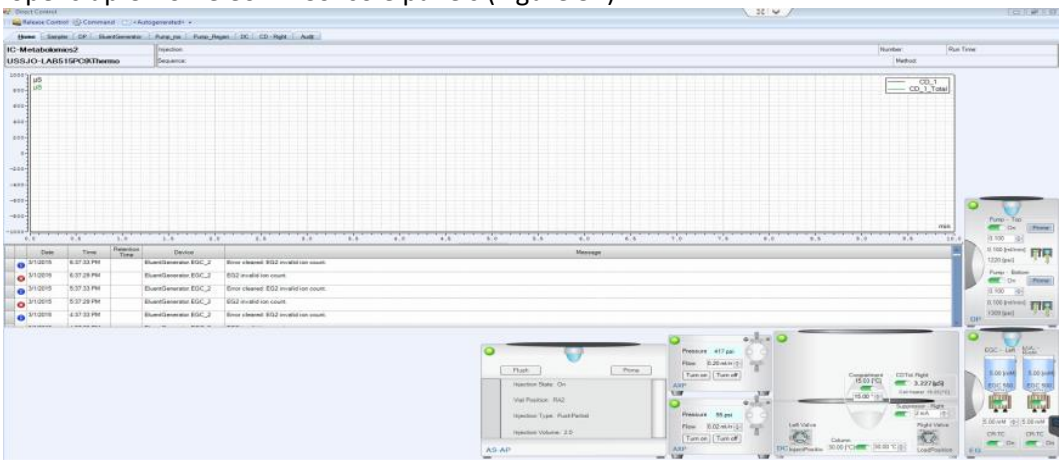


Figure 31: Opening IC instrument Console panels in Chromeleon

6) Select **Take Control**.
7) Press the F8 key on your keyboard, which pulls up manual commands.
8) Select **Sampler**.
9) Right-click on list to change view mode. Select **Expert** control level.
10) On the Relay 4 line, select "10sec_InjectOut" from pull down menu. See Figure 32.

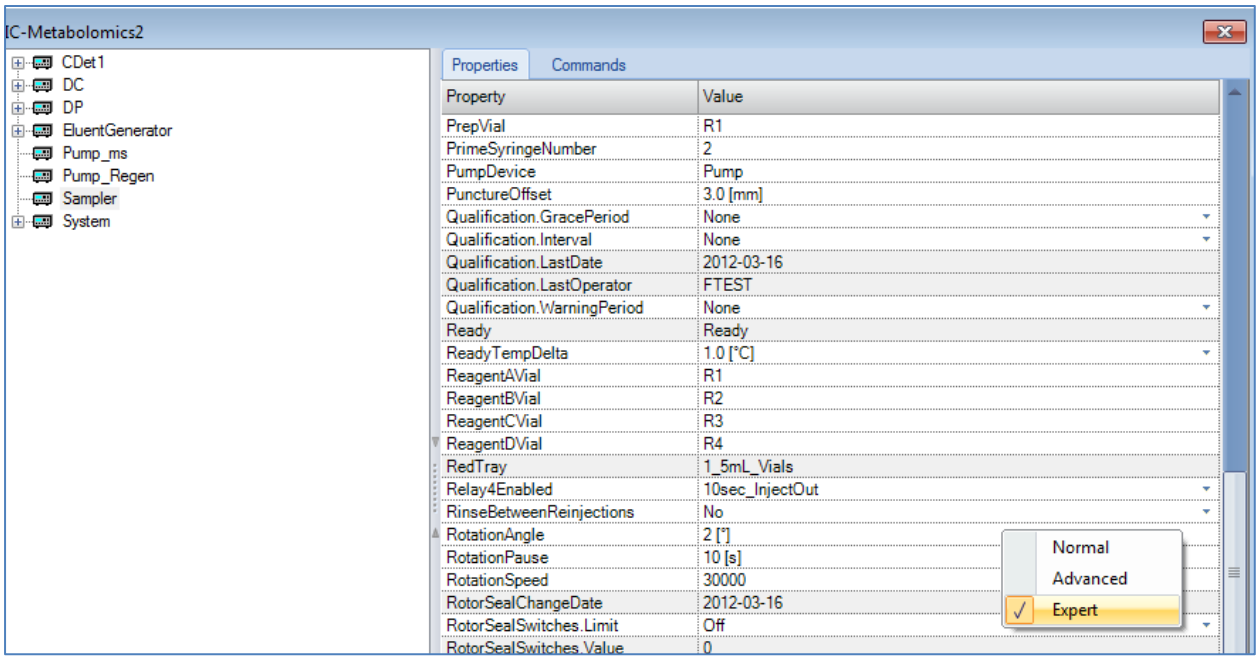


Figure 232: Using Expert mode, Enable Relay 4

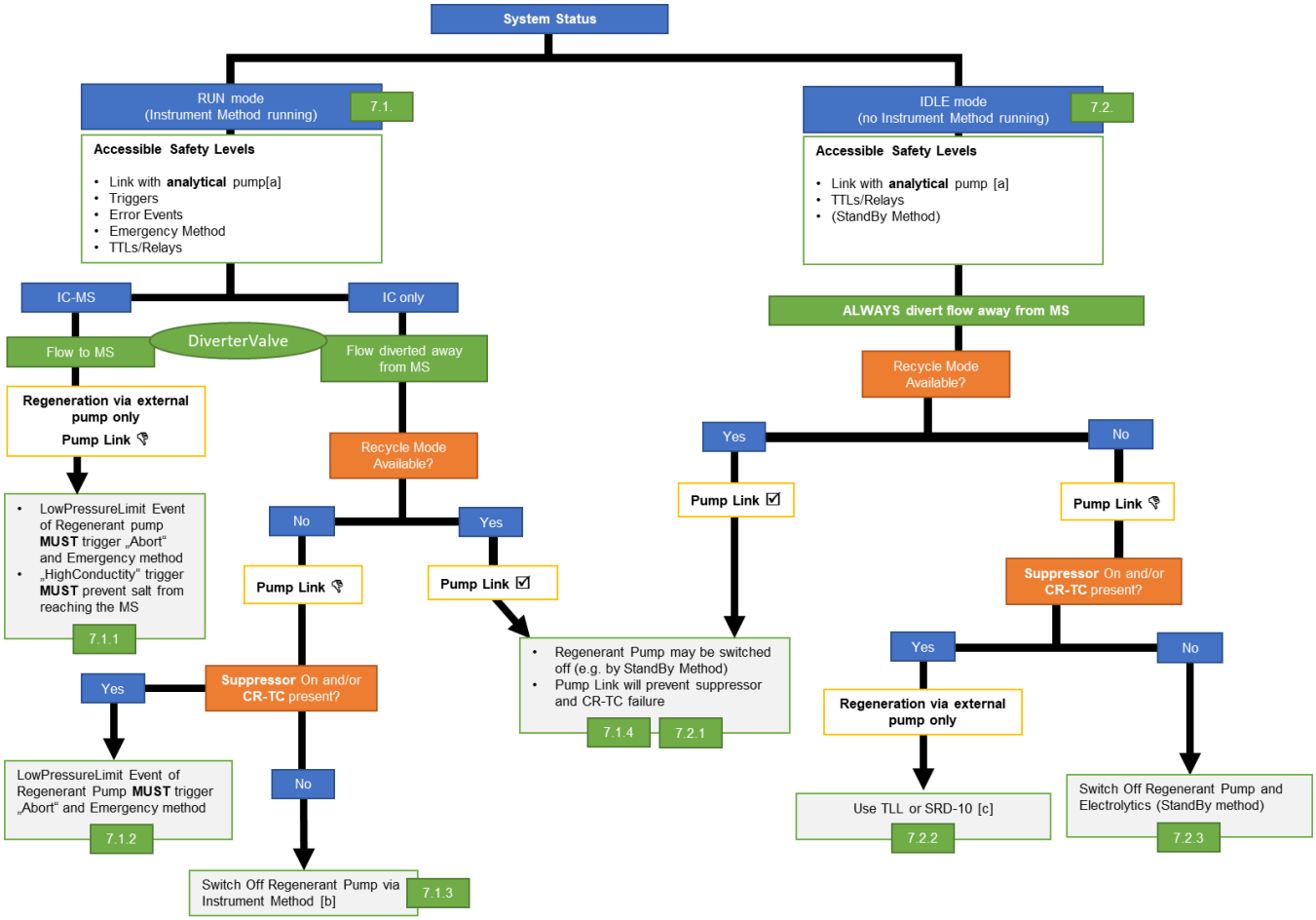
11) Select **Release Control**.
12) Return and complete the methods in Instrument Setup and complete the sample injection order, select the instrument methods in Sequence Setup.
13) Initiate the sequence start and select **SII for Xcalibur** to initiate start.
14) After the autosampler injects the sample, the IC and the MS will start collecting data.

13.4 Two software (Chromeleon + Xcalibur) Installation

For IC-MS installations it is common that the analyst may plan to switch from anions to cations or from IC-MS to HPLC-MS. As the MS can only accept one channel at a time, an effective solution would be to use a two-software installation: Chromeleon and SII for Xcalibur or Chromeleon and Xcalibur.

The software configuration can use SII for Xcalibur with Xcalibur by having two separate instrument configurations. However, the IC eluent generator system and the suppressor will undesirably turn-off when the configuration is switched to HPLC-MS. It is often more convenient to use Chromeleon independently for the IC-MS applications from Xcalibur for the HPLC-MS applications. See Section 9.9 for additional information

Appendix I: General Decision Chart for System Safety Operation



[a] The suppressor cannot be linked to an AXP pump

[b] This is important. If the pump fails (i.e. no solvent is left), the sequence will stop because of "Abort" error.

[c] This setup will usually be run in External water mode in IDLE and RUN mode. If a SP/DP pump is used for regeneration, electrolytics may be linked to this pump (no TTL is needed).

[d] SRD-10 is no longer recommended

Explanations

This chart is valid for all instrument setups that are discussed here. Its Intention is to help choosing the necessary safety options to be implemented depending on a given instrument setup/customer application.

Different safety options must be set up for the two possible system states: **RUN** mode and **IDLE** mode. Follow the flow chart in both states and check all "sub-states" according to the given system configuration/application. Implement all safety features as described.

Example: IC-MS System with CD and EGC cartridges

In RUN mode, there are two options for operation: **IC-MS** or **IC only** (depending on the position of the DiverterValve)

IC-MS:

The suppressor and CR-TC are supplied via regenerant pump**.** Thus, the pump link is not fully functional. Implement the following safety features:

Setup “low pressure limit” for the regenerant pump (“Abort” error)
Use Emergency Method (triggered by “Abort” errors)
Use a “HighConductivity” trigger in the Instrument Method to prevent salt from reaching the MS

IC-only:

If only CD measurements are performed, the system operates in Recycle Mode, thus the analytical pump link is operational. Always divert the flow away from the MS at the beginning of the Instrument Method (“Initial Time”). No further safety features need to be implemented. It is recommended to switch off the Regenerant pump to prevent an unintended “Abort” errors from this pump (e.g. caused by empty regenerant bottle).

IDLE mode:

In IDLE mode the system operates in Recycle Mode (Flow diverted away from MS, CR-TC and suppressor supplied by analytical pump). The pump link is fully operational. The Regenerant Pump may be switched (potential “abort” errors can’t occur by default). It is recommended to switch to IDLE via a StandBy Instrument Method.

System Status

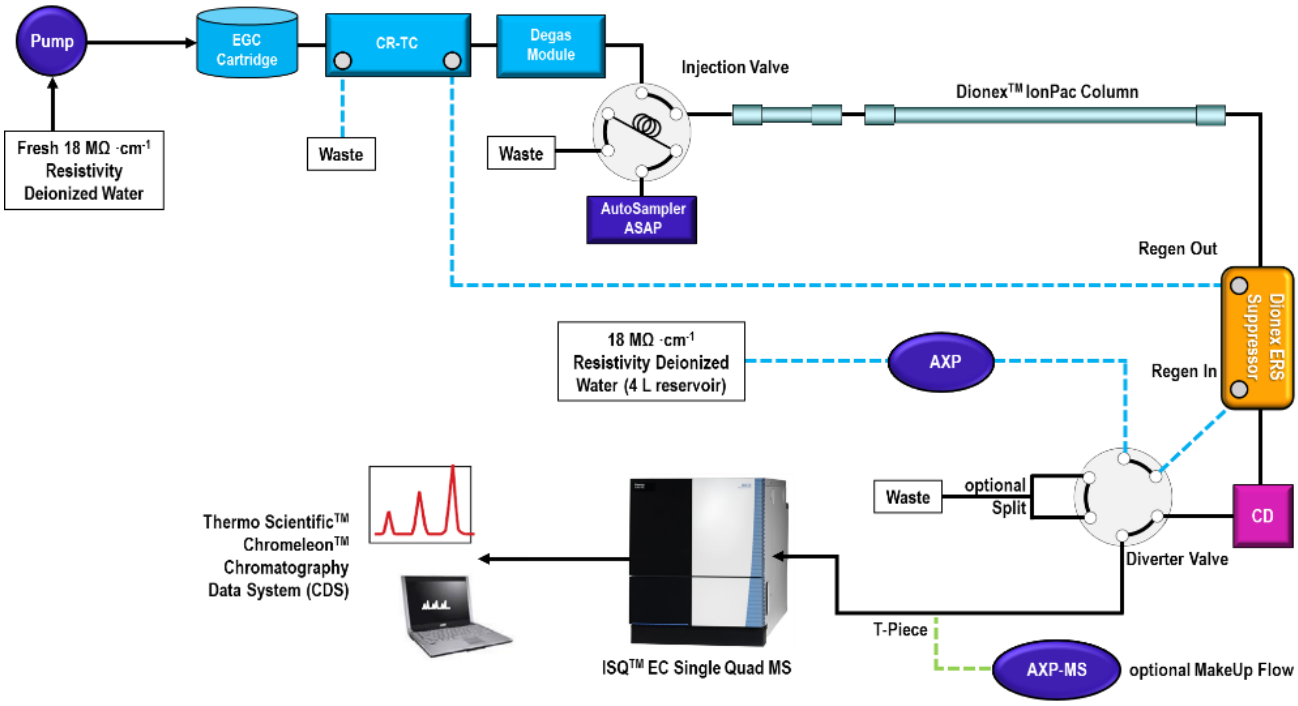
It must be assured that the system is safe in both modes: when the system is acquiring data (Instrument method running, RUN mode) and when in IDLE mode (no instrument method active)

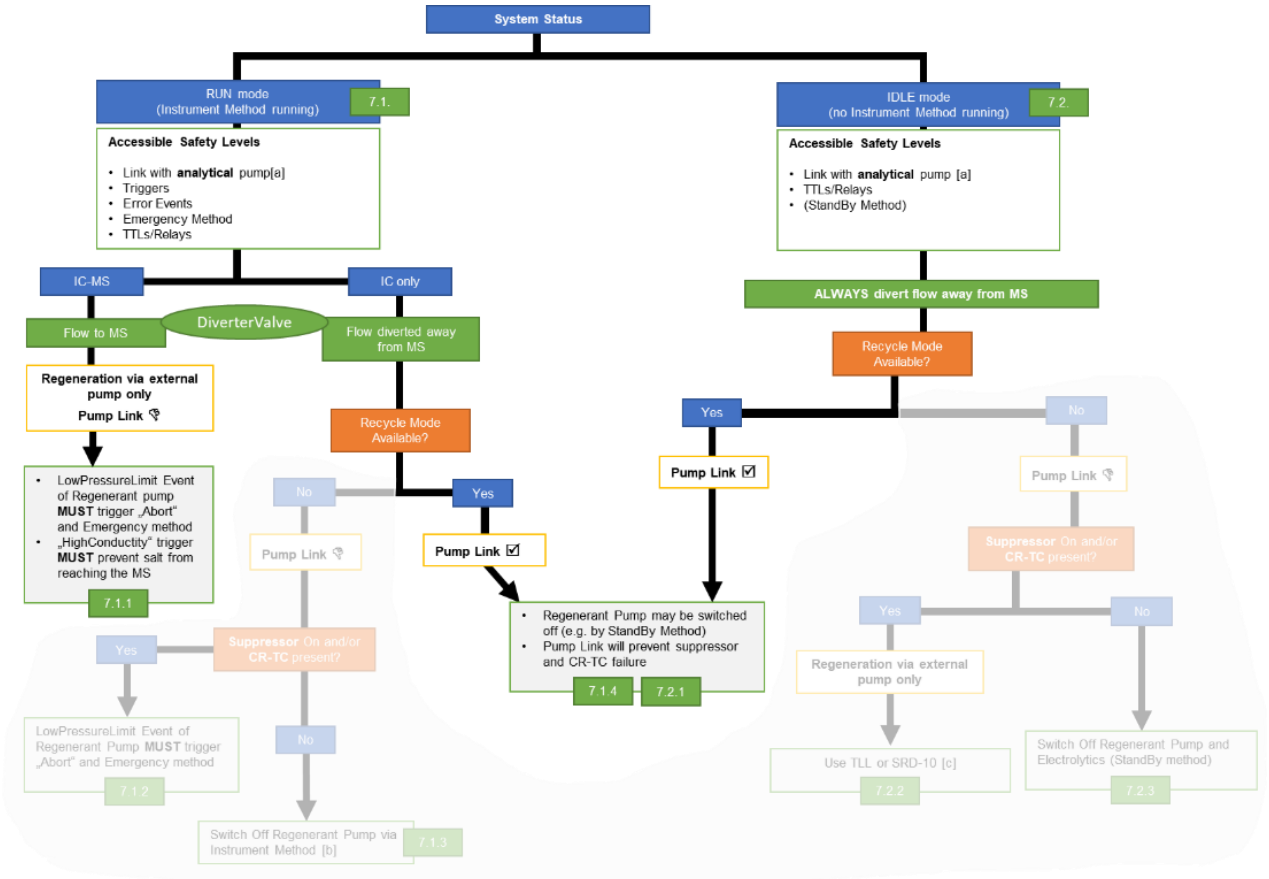
Depending on the system status, different safety options are available.

RUN mode: All the safety options are available

IDLE mode: Trigger cannot be applied

Errors such as reaching the lower pressure limit of a the AXP-pump are reported (Audit Trail), but no actions can be programmed to prevent damage of the electrolytics and/or MS Emergency Methods are not triggering.





Pump Link

The CR-TC is linked via the EG and thus always linked to the analytical pump. If an additional isocratic SP pump is available for external water regeneration, the suppressor can be linked to this pump. The

suppressor cannot be linked to an AXP pump (although the option is available on the ICS-6000 DetectorCompartment[1].

"Pump Link 👎": Pump Link is not working for at least one of the electrolytic devices (suppressor or CR-TC).

"Pump Link ☑": Pump Link is working.

RUN mode (Flow to the MS)

IC measurements with MS

The regeneration of the suppressor (and CR-TC) can only be accomplished by the Regenerant pump. Thus, Pump Link is not working (CR-TC and Suppressor are linked to the analytical pump).

The running method must include:

- A suitable LowPressureLimit for the regenerant pump (will cause "Abort")
- Error Handling for "Abort" errors. F Use a suitable Emergency Method to prevent damage from electrolytical devices and ISQ
- HighConductivityTrigger to prevent salt from reaching the MS
- Suitable error handling for the MakeUp pump if installed

IC only measurement with suppressor and/or CR-TC, but no recycle mode possible

Example Application:

- IC analysis using chemical regenerated suppressor or externally electrolytically regenerated suppressor (e.g. solvent containing eluents)
- "memory peaks" in RecycleMode
- Carbohydrates with PAD only and EGC (CR-TC needed, but supplied with AXP-pump)

The running method must include:

- OPTIONAL: The flow to the ISQ is diverted away from the MS at the {Initial Time}.
- Switch off unused electrochemical devices
- A suitable LowPressureLimit for the regenerant pump (will cause "Abort")
- Error Handling for "Abort" errors. F Use a suitable Emergency Method to prevent damage from electrolytical devices and ISQ
- Switch off MakeUp pump if installed

IC only measurement without suppressor and/or CR-TC, no recycle mode

Example Application:

- Carbohydrates with PAD

The running method must include:

[1] Attention! In the DC module of an ICS-6000, AXP pumps can be linked to suppressors in the Instrument controller. This link is NOT working properly (AXP pump revision 01 until 2016): CM7 ignores "AXP-Pump.State" (On/Off). Suppressor can always be switched On and does not switch off if "AXP-Pump.State = Off" (tested with Chromeleon 7.2.8, no change in the release notes of 7.2.9, AXP was connected via DB9 to USB converter, new AXP pump (since 2016) was not tested).

- OPTIONAL: The flow to the ISQ is diverted away from the MS at the {Initial Time}.
- Switch off unused electrochemical devices
- Switch Off the Regenerant Pump
- Switch off MakeUp pump if installed

The regenerant pump is not needed in this mode and will do no harm to the instrument but may cause unwanted “Abort” errors if a failure occurs.

IC only measurement with Recycle Mode

Example Application:
- All “standard” IC applications

The running method must include:
- OPTIONAL: The flow to the ISQ is diverted away from the MS at the {Initial Time}.
- (Switch off unused electrochemical devices)
- Switch Off the Regenerant Pump
- Switch off MakeUp pump if installed

The regenerant pump is not needed in this mode. Switch it off to avoid unwanted aborts of the sequence/injection if an error occurs on the regenerant pump.

The Pump Link will prevent failure of the electrolytical devices (recycle mode).

IDLE mode (Flow diverted away from MS)

Most IC systems are not switched off when not used over a short period of time. “Abort” error handling, Emergency methods and Triggers are not available in IDLE mode, safe system operation must be accomplished by the use of mandatory StandBy methods.

The StandBy method must
- Divert the flow away from the MS
- Stop the MakeUp pump if installed
- switch of unused electrolytical devices

Optional:
- Flush solvent/water through the MS via regenerant or optional MakeUp pump for a few minutes
- Turn on StandBy for the ISQ
- Lower Vaporizer Temperature

In General:
In IDLE mode, the safety triggers (e.g. low and high pressure of the AXP-pump) are not “active”[2]. To prevent damage to the Electrolytics:

- **ALWAYS use “Auto-Regeneration”-Mode if possible**
- **and/or switch off not used electrolytical devices (see example configurations below).**
- **If both options are not possible use a TTL connection from the AXP pump to the analytical pump.**

---

[2] In contrast to the RUN Mode (where an “Abort Error” of the AXP-pump will start the **emergency method** to prevent damage to the system), an AXP-pump error will not switch off the electrolytical devices in IDLE Mode (no pump link possible).

IDLE system with Recycle Mode

This mode operates like a “standard” IC application. Regenerant Pump should be stopped. Pump Link will ensure safe IDLE operation

IDLE system without Recycle Mode, at least one electrolytical device needed

Example Application: Carbohydrate System with EGC

In IDLE mode the CR-TC is still needed, but CR-TC regeneration channel is connected to regenerant pump. A failure of the external water supply can only be recognized by a TTL or SRD-10 (“Abort” error handling not possible, no Pump Link possible).

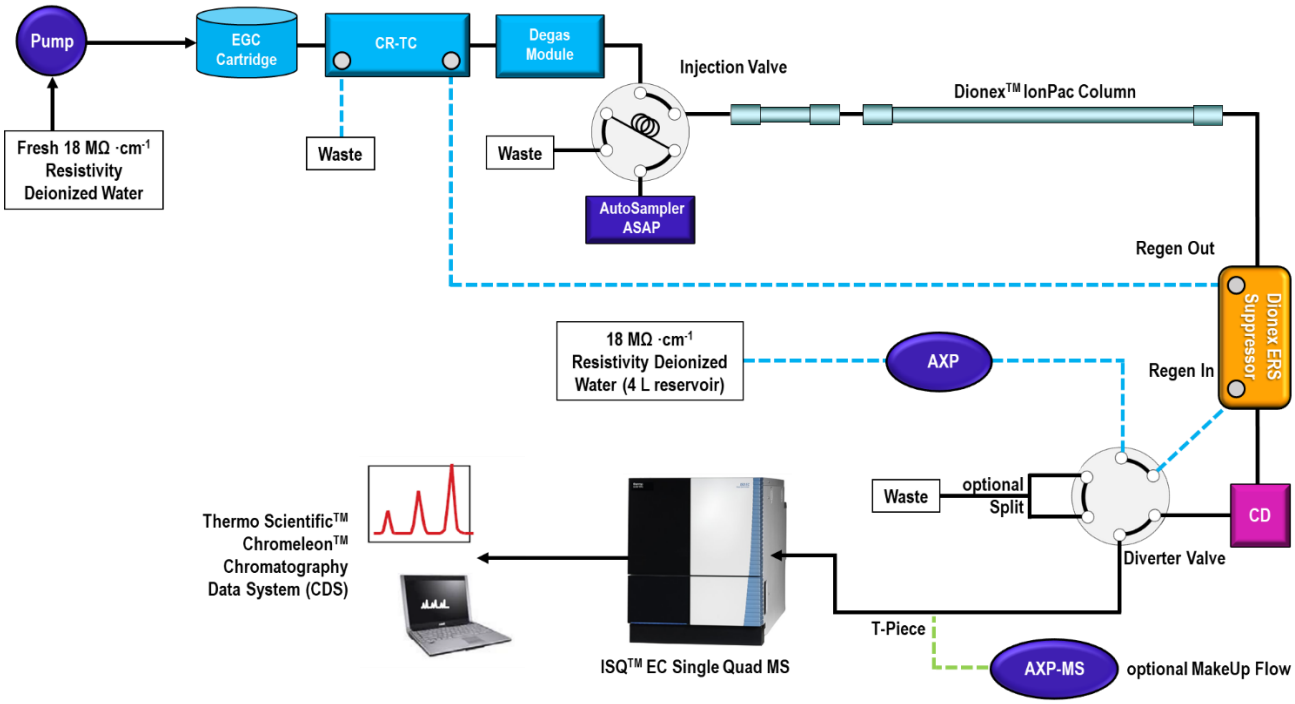
IDLE system without Recycle Mode, no Electrolytical devices needed

Example Application: Carbohydrate System with manual eluent preparation
Switch Off Regenerant Pump and all electrolytic devices

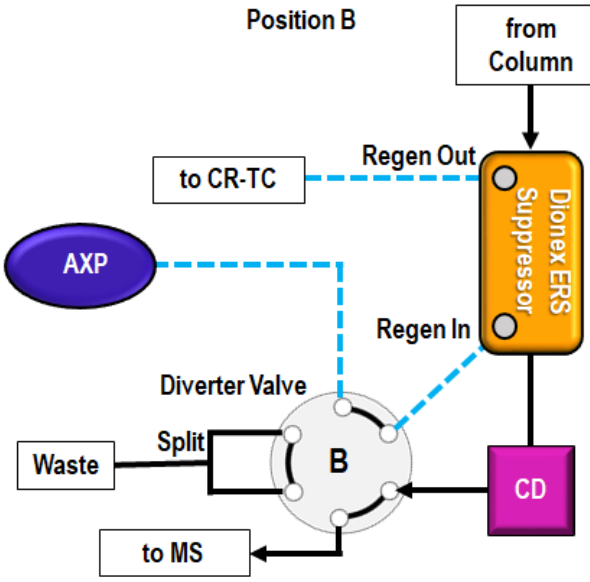
Appendix II. Configuration Schematics

The diverted valve is always directing the flow to Waste in position A (default valve position on system power up). Position B directs the flow to the MS.

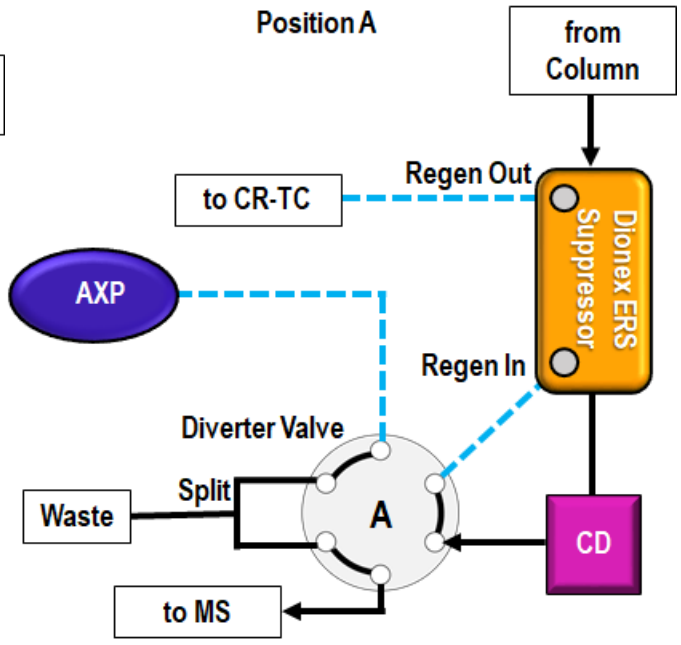
IC-MS with EGC and CD:



*Note: This setup can also be used for carbohydrates in RFIC mode with an Integrion System as long as no PAD is used (MS as sole detector).



Flow from CD to Suppressor & CR-TC (IDLE mode, Recycle Mode; RFIC)
AXP pump connected to MS (Jumper) or Waste



Flow from Conductivity Cell CD to MS (RUN mode)
AXP pump supplies Regenerant for Suppressor and CR-TC

Safety operations

- Link the Suppressor and CR-TC to the analytical pump
- Set "LowerPressure-Limits" for **all** pumps (**including** the AXP-pump for regenerant flow; per default a "low pressure" error will abort the running method and trigger the emergency method)
- In a "CD only" method, always turn Off the AXP-pump for regeneration and optional MakeUp-Flow pump*[3]
- Use "HighConductivity"-Triggers in all methods
- Use an emergency method

This setup ensures that the suppressor and CR-TC are supplied with water at all time. When the system is running IDLE recycle mode can be applied (DiverterValve always in position B). Only when an IC-MS method is running the valve is switched to Position A and the regenerant flow is provided by the AXP-pump. Here, the water supply is monitored via the AXP- pump pressure (error level for "fallen below LowerPressureLimit " is set to "Abort") which will trigger an emergency method switching the valve to position B (recycle mode).

[3] The AXP pump flows are not needed when no MS is used. Recycle Mode will ensure safe operation and no MakeUp-Flow is needed. Pump is even disadvantageous: an error triggered by theses pumps (e.g. "Fallen under LowerPressureLimit") will trigger an "Abort" of the method although the flow is not needed

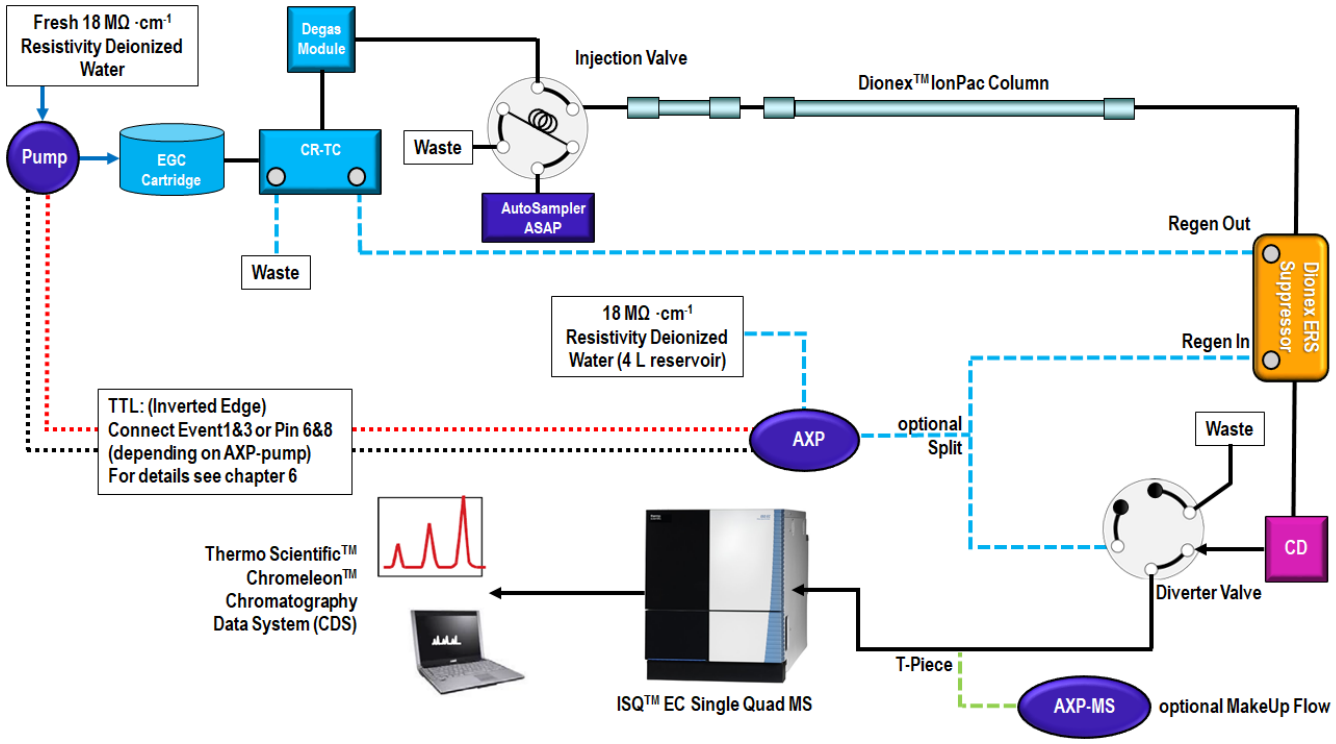
A TTL-link to the analytical pump or SRD-10 is not needed in this setup.

Optional split

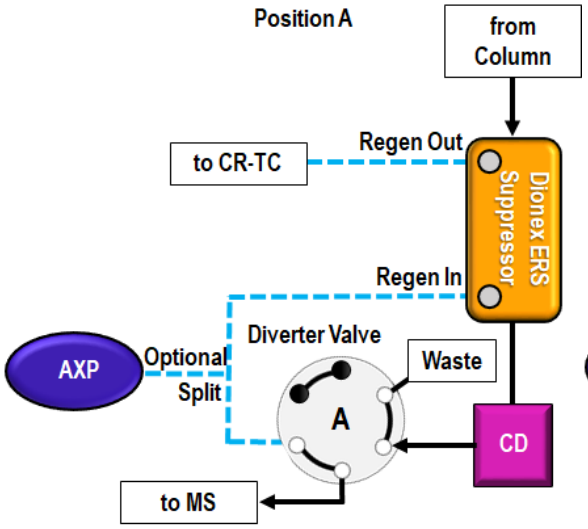
In IDLE mode the optional (adjustable) split allows the user to …

- direct the AXP-flow to Waste or MS (one flow-path completely closed)
- automatically reduce the AXP-flow to MS (both paths open) without the need to set the lower the flow via a method. This option is particularly useful when an error occurs

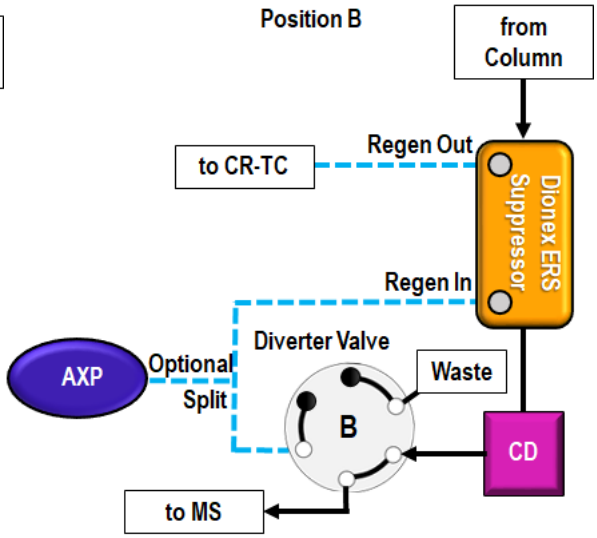
IC-MS with EGC and CD, with external regeneration of suppressor by default



Use when external regeneration is already required by default (without MS).



Analytical flow from CD directly to Waste
Suppressor and CR-TC supplied by AXP pump
Optional: AXP flushes MS via optional split



Analytical Flow to MS

Suppressor and CR-TC supplied by AXP pump

IC-MS with Carbohydrates:

Some considerations on Flow rates and Split ratios (Carbohydrate Setup):

Optimal Flow Rate to the MS:

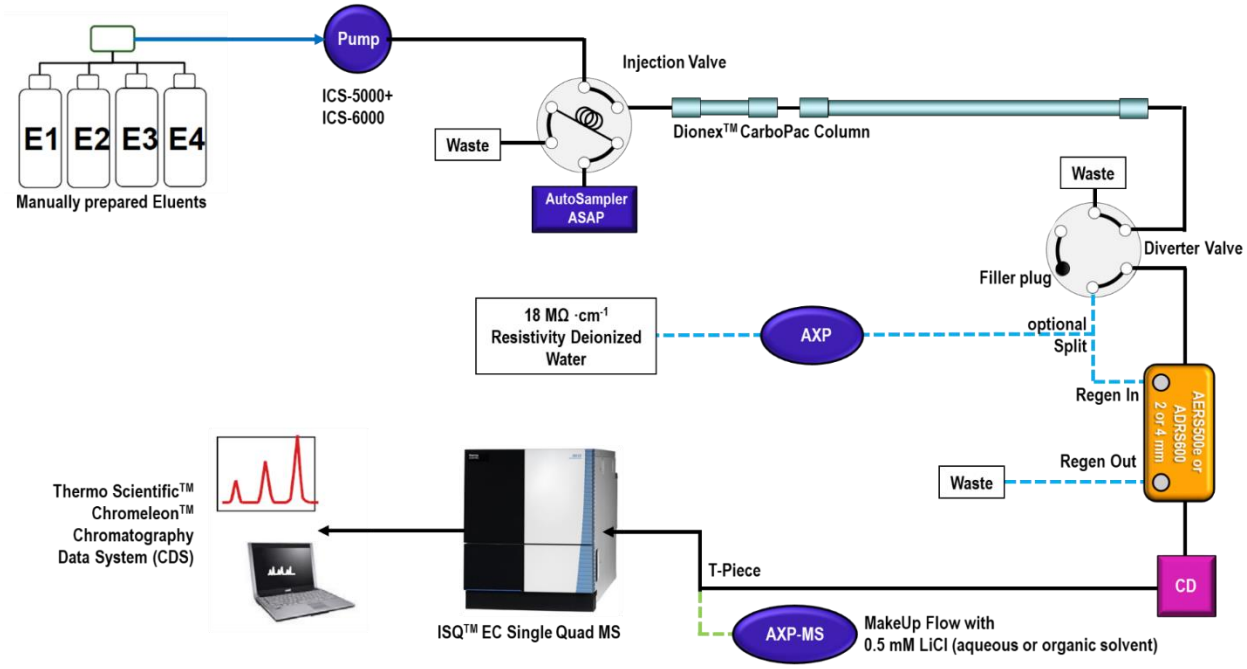
- ISQ operates at flow rates of 200 - 2000 µL/min (best: 200-500 µL/min = best ionization efficiency).
- Flow-rates below 200 µL/min cause an increased **Delay Volume** and **strong peak broadening** (reason: only black PEEK tubing (0.25 mm i.d.) or Viper (0.18 mm i.d.) must be used from Suppressor to the MS Probe to prevent high backpressure on the Suppressor; Delay Time from PAD to MS should be below 1.0 or better 0.5 minutes)
- Depending of analytical flow rate use a red/yellow PEEK capillary between the T-piece and PAD to adjust the flow to the MS to at least 150 to 200 µL/min (including the flow of an optional Make-Up Pump, see below)

- **Columns**
  Typical flow rates of 2 mm i.d. columns: 250 µL/min → only 100 µL/min flow to the PAD (which is sufficient in most applications, but do not exceed 100 µL/min to maintain peak shape in PAD cell)

- **Make-Up Pump**
  If a Make-Up pump is used, its flow rate adds to the flow rate directed to the MS. You may want to adjust the split ratio between PAD and MS to avoid excessive backpressure on the suppressor and to increase PAD signal

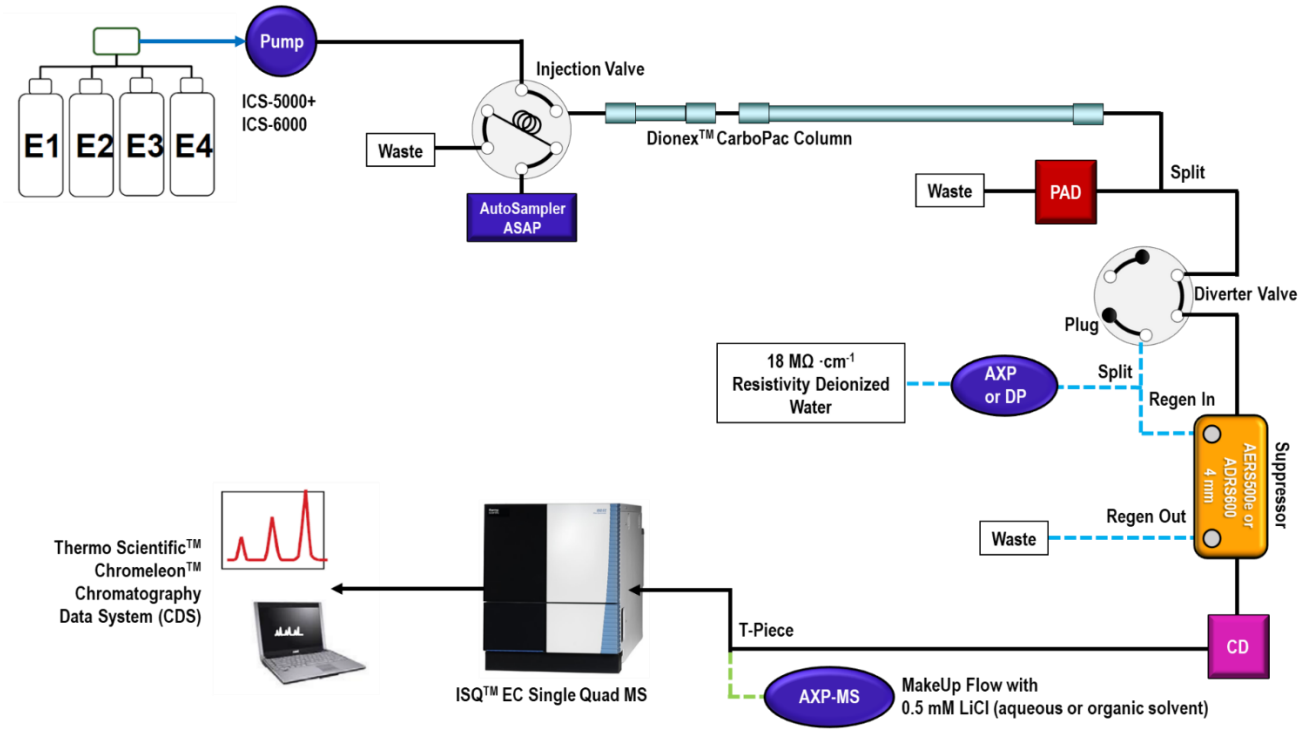
  Placement of T-Piece for MakeUp Flow (assumption: 50 µL/min MakeUp-Flow):
  - Flow rate from column to the MS > 200 µL/min => place the T-piece near the MS-probe (lower backpressure on Suppressor)
  - Flow rate from column to MS < 200 µL/min => place the T-piece near the suppressor (check backpressure!) to help with shorter Delay Time

MS-detection only (no PAD), Manually prepared Eluents (ICS-5000, ICS-6000 only)



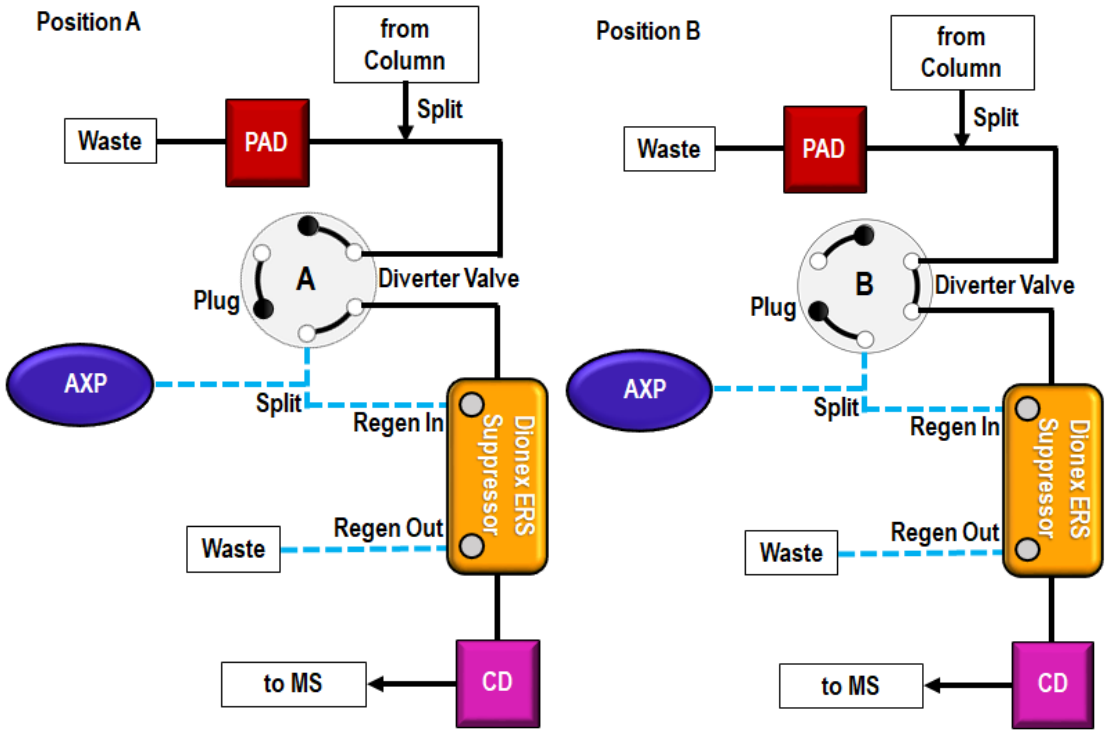
- Diverter Valve is installed before the Suppressor to avoid high salt concentrations (e.g. 1M NaOAc in washing steps)
- Split of regenerant flow allows Regeneration of Suppressor and Washing of Eluent path from the Diverter Valve to the MS
- 0.5 mM LiCl may improve signal of carbohydrates in positive mode and allows easier fragmentation by CID

IC-MS for Carbohydrates: MS-detection with PAD, Manually prepared Eluents (ICS-5000, ICS-6000 only)



- Diverter Valve is installed before the Suppressor to avoid high salt concentrations (e.g. 1 M NaOAc in washing steps)
- Split of regenerant flow allows Regeneration of Suppressor and Washing of Eluent path from the Diverter Valve to the MS
- 0.5 mM LiCl may improve signal of carbohydrates in positive mode and allows easier fragmentation by CID

Divert Valve Flow Scheme – All Carbohydrate Setups

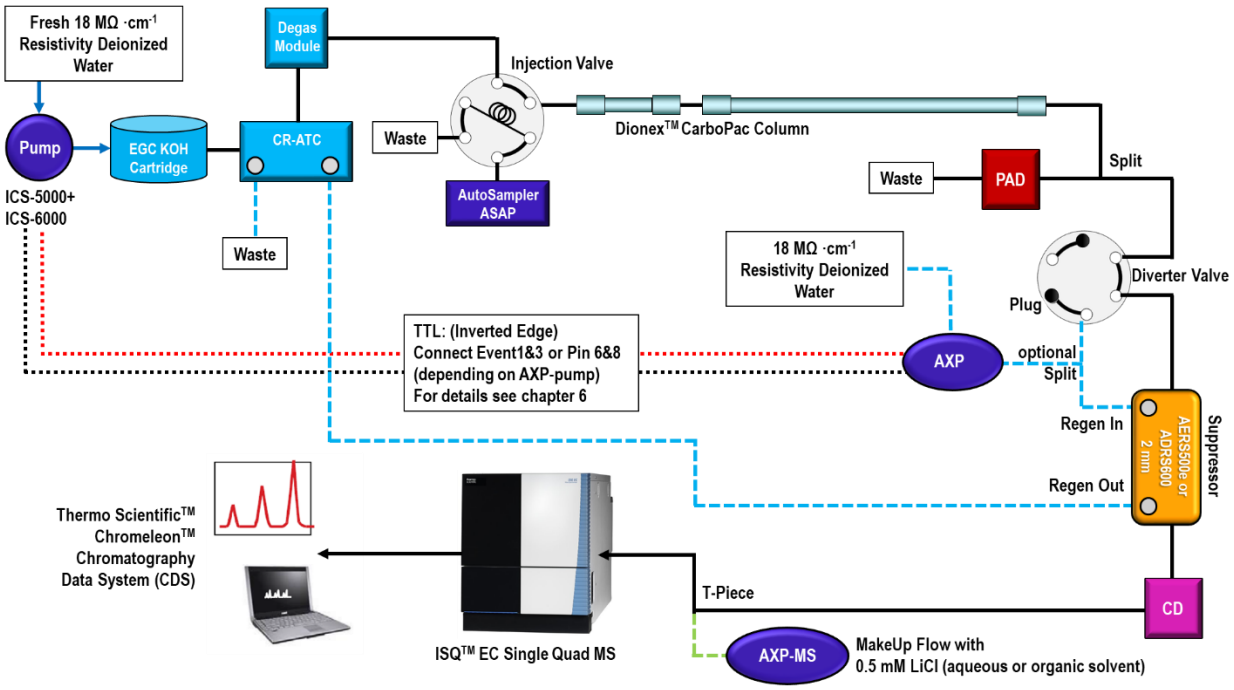


PAD and MS Detection
AXP pump supplies Regenerant for Suppressor

PAD Detection only
AXP pump supplies Regenerant for Suppressor and flushes Flow-path
No Suppressor Failure due to missing eluent flow

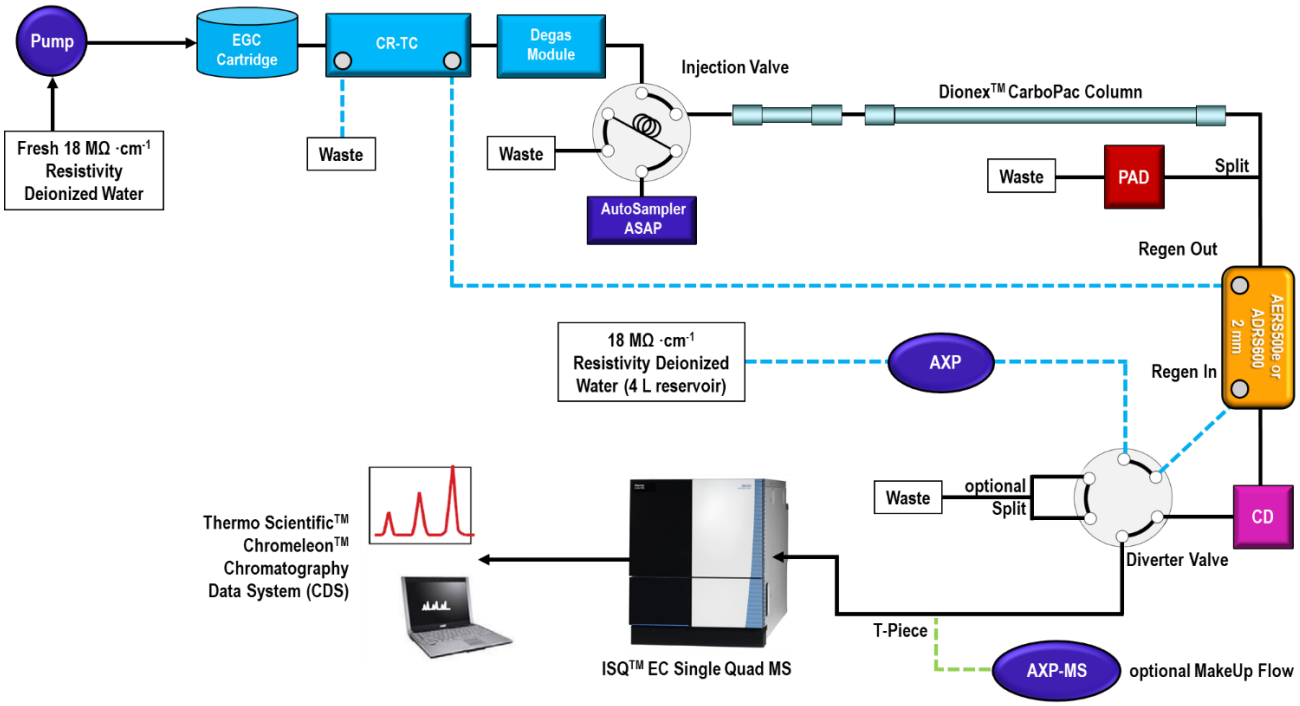
IC-MS for Carbohydrates: MS-detection with PAD, RFIC-generated Eluents

Option 1



If the optional regenerant flow split is not used, the eluent path of the suppressor will not be flushed when the analytical flow is diverted away from the MS. The suppressor has to be switched off to avoid damage to the suppressor, if this state is present for a longer time period (e.g. StandBy, IC-only methods)

Option 2: (adapted from SVL IC-MS Guide)



Advantages:

- Recycle mode available in IC-only MODE
- No TTL needed
- No need to switch off suppressor if flow directed away from MS

Disadvantages:

- Regenerant flow of suppressor and CR-TC reduced due to split