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EA IsoLink IRMS System for CNSOH

Including the Flash IRMS Elemental Analyzer

Operating Manual

31707010 Revision B December 2016

ThermoFisher SCIENTIFIC

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Release history:

Revision A, released October 2016, *"Original Instructions"*
Revision B, released December 2016

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Reader's Survey

EA IsoLink IRMS System for CNSOH, PN 31707010, Revision B

	Strongly Agree	Agree	Neutral	Disagree	Strongly Disagree
The manual is well organized.	1	2	3	4	5
The manual is clearly written.	1	2	3	4	5
The manual contains all the information I need.	1	2	3	4	5
The instructions are easy to follow.	1	2	3	4	5
The instructions are complete.	1	2	3	4	5
The technical information is easy to understand.	1	2	3	4	5
Examples of operation are clear and useful.	1	2	3	4	5
The figures are helpful.	1	2	3	4	5
I was able to operate the system using this manual.	1	2	3	4	5

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If not, please comment below. Attach additional sheets if necessary.

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- ❑ Pharmaceutical
- ❑ Research/Education
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Declaration

Manufacturer: **Thermo Fisher Scientific**

Thermo Fisher Scientific is the manufacturer of the instrument described in this manual and, as such, is responsible for the instrument safety, reliability and performance only if:

- installation
- re-calibration
- changes and repairs

have been carried out by authorized personnel and if:

- the local installation complies with local law regulations
- the instrument is used according to the instructions provided and if its operation is only entrusted to qualified trained personnel

Thermo Fisher Scientific is not liable for any damages derived from the non-compliance with the aforementioned recommendations.

Thermo Fisher Scientific (Bremen) GmbH
Hanna-Kunath-Str. 11; 28199 Bremen, Germany; Tel. +49 421 54930

Regulatory Compliance

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

Safety

This device complies with the following safety standards according to Low Voltage Directive 2014/35/EU.

- EN 61010-1:2010 (3ª ed.); IEC 61010-1:2010 (3rd ed.); CAN/CSA C22.2 No. 61010-1-12; UL 61010-1:2012; IEC 61010-2-010:2014 (3rd ed.); IEC 61010-2-081:2015 (2nd ed.)

Electromagnetic Compatibility

This device complies with the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI) according to directive 2014/30/EU:

- IEC 61326-1:2013 (2a ed.); IEC 51326-1:2012 (2nd ed.)

IMPORTANT: Class A equipment is intended for use in an industrial environment. In others environments there may be potential difficulties in ensuring electromagnetic compatibility, due to the conducted as well as radiated disturbances.

FCC Compliance Statement

THIS DEVICE COMPLIES WITH PART 15 OF THE FCC RULES. OPERATION IS SUBJECT TO THE FOLLOWING TWO CONDITIONS: (1) THIS DEVICE MAY NOT CAUSE HARMFUL INTERFERENCE, AND (2) THIS DEVICE MUST ACCEPT ANY INTERFERENCE RECEIVED, INCLUDING INTERFERENCE THAT MAY CAUSE UNDESIRED OPERATION.

CAUTION Read and understand the various precautionary notes, signs, and symbols contained inside this manual pertaining to the safe use and operation of this product before using the device.

Notice on Lifting and Handling of Thermo Scientific Instruments

For your safety, and in compliance with international regulations, the physical handling of this Thermo Fisher Scientific instrument ***requires a team effort*** to lift and/or move the instrument. This instrument is too heavy and/or bulky for one person alone to handle safely.

Notice on the Proper Use of Thermo Scientific Instruments

In compliance with international regulations: Use of this instrument in a manner not specified by Thermo Fisher Scientific could impair any protection provided by the instrument.

Notice on the Susceptibility to Electromagnetic Transmissions

Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

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WEEE Directive

2012/19/EU

Thermo Fisher Scientific is registered with B2B Compliance (B2Bcompliance.org.uk) in the UK and with the European Recycling Platform (ERP-recycling.org) in all other countries of the European Union and in Norway.

If this product is located in Europe and you want to participate in the Thermo Fisher Scientific Business-to-Business (B2B) Recycling Program, send an email request to weee.recycle@thermofisher.com with the following information:

- WEEE product class
- Name of the manufacturer or distributor (where you purchased the product)
- Number of product pieces, and the estimated total weight and volume
- Pick-up address and contact person (include contact information)
- Appropriate pick-up time
- Declaration of decontamination, stating that all hazardous fluids or material have been removed from the product

For additional information about the Restriction on Hazardous Substances (RoHS) Directive for the European Union, search for RoHS on the Thermo Fisher Scientific European language websites.

IMPORTANT This recycling program is **not** for biological hazard products or for products that have been medically contaminated. You must treat these types of products as biohazard waste and dispose of them in accordance with your local regulations.

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Directive DEEE

2012/19/EU

Thermo Fisher Scientific s'est associé avec une ou plusieurs sociétés de recyclage dans chaque état membre de l'Union Européenne et ce produit devrait être collecté ou recyclé par celle(s)-ci. Pour davantage d'informations, rendez-vous sur la page www.thermoscientific.fr/rohs.

WEEE Direktive

2012/19/EU

Thermo Fisher Scientific hat Vereinbarungen mit Verwertungs-/Entsorgungsfirmen in allen EU-Mitgliedsstaaten getroffen, damit dieses Produkt durch diese Firmen wiederverwertet oder entsorgt werden kann. Weitere Informationen finden Sie unter www.thermoscientific.de/rohs.

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15-Years Warranty for Furnace and Thermal Conductivity Detector

Thermo Fisher Scientific provides a 15-year warranty on the combustion and reduction furnaces (P/N 354 06100) of the Flash IRMS Elemental Analyzer. The combustion and reduction furnaces are assembled using the highest quality materials and operationally tested. Each furnace is supplied with a unique serial number to identify it.

If the combustion or reduction furnaces have a manufacturing or material defect during the 15-year warranty period, from the date of delivery of the system, they will be replaced free of charge by a service engineer.

What is excluded from the Furnaces Warranty?

Damage caused to the furnace as a result of improper use, which is defined as unwarranted maintenance and changes by the user, are not covered.

Thermal Conductivity Detector Warranty

The Thermal Conductivity Detector (P/N 419 07510) is assembled using the highest quality materials and operationally tested.
The operator must not touch the Thermal Conductivity Detector and in case of any suspected problems, should contact a service engineer for diagnosis.

Only in the case that a qualified service engineer determines that the Thermal Conductivity Detector has a manufacturing or material fault, there is a 15-year warranty covering replacement.

What is excluded from the Thermal Conductivity Detector Warranty?

Damage caused to the Thermal Conductivity Detector as a result of improper use, which is defined as unwarranted maintenance and changes by the user, are not covered.

Who to contact in case of problems?

In the event of Furnace or Thermal Conductivity Detector issue, please contact the Organic Elemental Analyzer Product Manager, Guido Giazzi at guido.giazzi@thermofisher.com.

Contents

Preface

This manual contains detailed information for the use of the Thermo Scientific™ EA IsoLink™ IRMS System for CNSOH.

Contents

About Your System

Thermo Scientific systems provide high-caliber elemental analysis instrumentation.

IMPORTANT Thermo Scientific systems optimize the separation and detection capabilities of EA IsoLink IRMS System for CNSOH providing high performance analytical capabilities for both research, and routine applications. More information about the use of this system can be found in related documentation sources, and by using the provided contact information.

WARNING Thermo Scientific systems operate safely and reliably under carefully controlled environmental conditions. If the equipment is used in a manner not specified by the manufacturer, the protections provided by the equipment might be impaired. If you maintain a system outside the specifications listed in this guide, failures of many types, including personal injury or death, might occur. The repair of instrument failures caused by operation in a manner not specified by the manufacturer is specifically excluded from the standard warranty and service contract coverage.

WARNING Operation of this system requires the use of chemical substances with different hazard specifications. Before using any chemicals, read the hazard indications and information reported in the Safety Sheet supplied by the manufacturer, referring to the relevant CAS (Chemical Abstract Service) number.

Power Rating

EA IsoLink IRMS System for CNSOH
- 230 Vac ±10%, 50/60 Hz ±2 Hz, 1400 VA

Detailed instrument specifications are in the Product Specifications Sheet.

Contacting Us

Thermo Fisher Scientific provides comprehensive technical assistance worldwide and is dedicated to the quality of our customer relationships and services.

Use http://www.thermofisher.com address for products information.

Service contact details are available under: www.unitylabservices.com

For other information please contact your local Thermo Fisher Scientific office or affiliate.

Safety Alerts and Important Information

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

IMPORTANT Highlights information necessary to prevent damage to software, loss of data, or invalid test results, or it might contain information that is critical for optimal performance of the system.

Note Emphasizes important information about a task.

Tip Provides information that can make a task easier.

Safety Symbols and Signal Words

All safety symbols are followed by **WARNING** or **CAUTION**, which indicates the degree of risk of personal injury, instrument damage, or both. Cautions and warnings are followed by a descriptor, such as **BURN HAZARD**.

A **WARNING** is intended to prevent improper actions that could cause personal injury. A **CAUTION** is intended to prevent improper actions that might cause personal injury and/or instrument damage. You find the following safety symbols on your instrument or in this guide:

Symbol	Description
	**BIOHAZARD:** Indicates that a biohazard *will*, *could*, or *might* occur.
	**BURN HAZARD:** Alerts you to the presence of a hot surface that *could* or *might* cause burn injuries.
	**ELECTRICAL SHOCK HAZARD:** Indicates that an electrical shock *could* or *might* occur.
	**FIRE HAZARD:** Indicates a risk of fire or flammability *could* or *might* occur.
	**GLOVES REQUIRED:** Indicates that you must wear gloves when performing a task or physical injury *could* or *might* occur.
	**MATERIAL AND EYE HAZARD.** Indicates you must wear eye protection when performing a task.
	**MATERIAL AND EYE HAZARD:** Indicates that eye damage *could* or *might* occur.
	**HAND AND CHEMICAL HAZARD:** Indicates that chemical damage or physical injury *could* or *might* occur.
	**HARMFUL.** Indicates that the presence of harmful material *will, could, or might* occur.
	**INSTRUMENT DAMAGE:** Indicates that damage to the instrument or component *might* occur. This damage might not be covered under the standard warranty.
	**LIFTING HAZARD.** Indicates that a physical injury *could* or *might* occur if two or more people do not lift an object.
	**READ MANUAL:** Alerts you to carefully read your instrument's documentation to ensure your safety and the instrument's operational ability. Failing to carefully read the documentation *could* or *might* put you at risk for a physical injury.
	**TOXIC SUBSTANCES HAZARD:** Indicates that exposure to a toxic substance could occur and that exposure *could* or *might* cause personal injury or death.
	**RADIOACTIVE HAZARD.** Indicates that the presence of radioactive material *could or might* occur.
	For the prevention of personal injury, this general warning symbol precedes the **WARNING** safety alert word and meets the ISO 3864-2 standard. In the vocabulary of ANSI Z535 signs, this symbol indicates a possible personal injury hazard exists if the instrument is improperly used or if unsafe actions occur. This symbol and another appropriate safety symbol alerts you to an imminent or potential hazard that *could cause personal injury*.

Instrument Markings and Symbols

The following table explains the symbols used on Thermo Fisher Scientific instruments. Only a few of them are used on the EA IsoLink IRMS System for CNSOH IRMS System, which are annotated with an asterisk below.

Table 1. Instrument Marking and Symbols

	Symbol	Description
		Direct current
*		Alternating current
		Both direct and alternating current
		Three-phase alternating current
		Earth (ground) terminal
		Protective conductor terminal
		Frame or chassis terminal
		Equipotentiality
*		On (supply)
*		Off (supply)
		Equipment protected throughout by DOUBLE INSULATION or REINFORCED INSULATION (Equivalent to Class II of IEC 536)
*		Instruction manual symbol affixed to product. Indicates that the user must refer to the manual for specific **WARNING** or **CAUTION** information to avoid personal injury or damage to the product.
		Caution, risk of electric shock
*		Caution, hot surface
*		Caution, biohazard
		In-position of a bistable push control
		Out-position of a bistable push control
		Jack socket
*		Symbol in compliance to the Directive 2012/19/EU on Waste Electrical and Electronic Equipment (WEEE) placed on the European market after August, 13, 2005.

Environmental Conditions

- For use and operation indoor only.
- Altitude up to 2000 meters.
- Operating temperature range from 15 to 35 °C.
- Maximum relative humidity between 30% and 85%.
- Voltage variations not exceeding ±10% of the nominal value.
- Transients according to installation categories **II**.
- Degree of pollution according to IEC 664 (3.7.3) **2**.

Safety Information

WARNING The instrument must be used according to the specifications of this guide. Improper use can adversely affect the instrument protection. If the equipment is connected to optional instruments, such as computer, balance, and so on, the degree of insulation of peripheral devices should be equivalent or higher (double or reinforced) than that of the EA IsoLink CNSOH IRMS System. The analyzer operation requires the use of chemical substances having different hazard specifications.
Before using chemicals, please read the hazard indications and information reported in the Material Safety Data Sheet supplied by the manufacturer referring to the relevant CAS (Chemical Abstract Service) number.

Use of Gases

The following gases are used with the instrument:

- **Helium** (**He**) as carrier gas.
- **Oxygen** (**O_2**) as gas for sample oxidation.
- **Gases Purity** — The EA IsoLink IRMS System for CNSOH uses helium, argon, and oxygen with **99.995%** minimum purity.
- **Maximum Pressure** — The maximum pressures of the gases to supply the Flash IRMS Elemental Analyzer is 700 kPa (7 bar).
- **Nominal Pressure of Gases** — The nominal pressure of the gases to supply the Flash IRMS Elemental Analyzer:
  - Maximum 250-300 kPa (2.5-3 bar) for helium (**He**).
  - Maximum 250-300 kPa (2.5-3 bar) for oxygen (**O_2**).

Safety Cut Off Device

When an alarm condition is detected, this device cuts off the power to the heating resistors of the oxidation, reduction furnaces and to the GC Oven. For more details see "Safety Cut Off Device" on page 5.

Gases Precautions

WARNING Before using gases, carefully read the hazard indications and information reported in the Material Safety Data Sheet (MSDS) supplied by the manufacturer referring to the CAS (Chemical Abstract Service) number. It is the user's responsibility to ensure compliance with all local safety regulations for the use of gases.

Precaution for Helium

Helium is a nontoxic, odorless, colorless, nonflammable gas stored in cylinders at high pressure. It can cause rapid suffocation when concentrations are sufficient to reduce oxygen levels below 19.5%. It is lighter than air and may collect in high points or along ceilings.

Precaution for Oxygen

Oxygen is an odorless, colorless, nonflammable gas stored in cylinders at high pressure. It is an oxidizing gas and vigorously accelerates combustion. Keep away from oils or grease. Rescue personnel should be aware of the extreme fire hazards associated with oxygen-enriched (greater than 23%) atmospheres.

Precaution for Hydrogen

Hydrogen is a colorless, odorless, highly flammable gas. The use of hydrogen requires the operator's strict attention and compliance with special precautions due to the hazards involved. Hydrogen is a dangerous gas, particularly in an enclosed area when it reaches a concentration corresponding to its lower explosion level (4% in volume). When mixed with air it can create an explosive mixture.

Precaution for Nitrogen

Liquid nitrogen is a colorless and odorless gas. It can cause rapid suffocation when concentrations are sufficient to reduce oxygen levels below 19.5%. A Self Contained Breathing Apparatus (SCBA) may be required. Oxygen concentrations must be monitored in the release area.

Precaution for Sulfur Dioxide

Sulfur dioxide is a colorless gas with an irritating odor. May be fatal if inhaled. Causes severe respiratory tract, eye and skin burns. Use only with adequate ventilation.

Precaution for Carbon Dioxide

Carbon dioxide is a colorless, cryogenic liquid. At low concentrations, is odorless. At higher concentrations carbon dioxide will have a sharp, acidic odor. At concentrations between 2 and 10%, Carbon dioxide can cause nausea, dizziness, headache, mental confusion, increased blood pressure, and increased respiratory rate. If the gas concentration reaches 10% or more, suffocation and death can occur within minutes.

Precaution for Carbon Monoxide

Carbon monoxide is a colorless gas. May be fatal if inhaled. Causes severe respiratory tract, Use only with adequate ventilation.

Hazardous Substances Precautions

WARNING Before using hazardous substances (toxic, harmful, and so on), read the hazard indications and information reported in the applicable Material Safety Data Sheet (MSDS.) Use personal protection according to the safety requirements.

Venting Toxic Gases

When analyzing toxic compounds be aware that during the normal operation of the EA some of the sample might be vented outside the instrument through the inlet and detector exits; therefore, make sure to vent the exhaust gases to a fume hood. Consult local Environmental and Safety Regulations for instructions in exhausting fumes from your system.

Residual Risks

Users of Flash IRMS Elemental Analyzer must pay attention to the following residual hazards.

High Voltage

WARNING DO NOT OPEN the electrical compartment because there are no user serviceable parts inside. Any operation inside the compartment must be carried out by authorized and trained Thermo Fisher Scientific personnel.

Hot Parts

WARNING Do not open the furnace compartment during operation due to the very high temperatures reached during operation. The protecting plate should only be removed when the temperature of the furnace is near that of room temperature.

Hazardous Chemical

WARNING Samples, consumables, reactors and filters filling materials might contain toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Avoid exposure to potentially harmful materials. Always wear protective clothing, gloves, mask, and safety glasses when you handle consumables, reactors and filters filling materials and during the cleaning of the MAS Plus's piston and the remotion of the ashes from the crucible when used.
Also contain waste streams and use proper ventilation. Refer to our supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular compound.

CAUTION Always use original Thermo Fisher Scientific materials and products. The use of materials not meeting the technical specifications of our products does not ensure a good operation of the instrument and may even damage it.

Maintenance Precaution

WARNING When, for technical reasons, it is necessary to work on instrument parts which might involve an hazard (moving parts, components under voltage, and so on), the authorized Technical Service must be contacted. This type of situations can be identified because access to these parts is possible only by using a tool. The removable protective covers bear a warning symbol suggesting to refer to the documentation accompanying the instrument. When a maintenance operation is performed, the operator must have received proper training to carry out specific actions.

CAUTION When the instrument is switched off, consider that its does not cool down immediately, but heat tends to concentration in the upper part of the furnaces area. It should be made clear that it is better to cool down the furnaces first before switching off the instrument. Switching it off means that the fan in the back does not remove the hot air concentrating at the top and the surface thus becomes very hot. The openings provided for the chamber aeration will cause a slow cooling of the same, which however, in the vicinity of the areas marked with the symbol "hot surfaces", might even reach temperatures higher than ambient temperature. Therefore in the minutes immediately following the instrument switching off, the operator must consider this risk and pay adequate attention during any maintenance operations following the use of the instrument.

Personal Protective Equipment

This manual can only give general suggestions for personal protective equipment (PPE), which protects the wearer from hazardous substances. Refer to the Material Safety Data Sheets (MSDSs) of the chemicals handled in your laboratory for advice on specific hazards or additional equipment.

- **Eye Protection** — The type of eye protection required depends on the hazard. For most situations, safety glasses with side shields are adequate. Where there is a risk of splashing chemicals, goggles are required.
- **Protective Clothing** — When the possibility of chemical contamination exists, protective clothing that resists physical and chemical hazards should be worn over street clothes. Lab coats are appropriate for minor chemical splashes and solids contamination, while plastic or rubber aprons are best for protection from corrosive or irritating liquids.

- **Gloves** — For handling chemical compounds, Thermo Fisher Scientific recommends the following gloves: white nitrile clean room gloves (Fisher Scientific P/N 19-120-2947B [size medium] or P/N19-120-2947C [size large]; Thermo Scientific P/N23827-0008 [size medium] or P/N 23827-0009 [size large]). For handling hot objects, gloves made of heat-resistant materials (for leather) should be available.
- **Mask** — For handling chemical compounds and filling materials for preparing and cleaning reactors and filters, and for removing the ashes from the crucible when used.

Training

Thermo Fisher Scientific offers worldwide training on instruments and software. Experience has shown that maximum results can be obtained from a scientific instrument, if the instrument operator receives an adequate training.

We recommend that the key operator undertakes training at Thermo Fisher Scientific Rodano - Milan (Italy), Thermo Fisher Scientific Bremen (Germany), at your site, or at one of the local Thermo Fisher Scientific offices. For information on training courses and enrollment, please contact your local Thermo Fisher Scientific office.

1

Getting Familiar with your EA IsoLink IRMS System for CNSOH

This chapter provides information to familiarize you with your Thermo Scientific™ EA IsoLink™ IRMS System for CNSOH. Here, a detailed description of the instrument's components is provided. The EA IsoLink IRMS System includes the Flash IRMS Elemental Analyzer, the Thermo Scientific™ ConFlo IV Universal Interface and a Thermo Scientific Isotope Ratio Mass Spectrometer.

Contents

Technical Features

Table 1 summarizes the major technical features of the EA IsoLink IRMS System for CNSOH.

Table 1. Technical Features of the Flash IRMS Elemental Analyzer

Features	Description
Detector	Thermal conductivity detector (TCD)
External interface	RS 232 serial line
Instrument control	Isodat Software Suite 3.0, Eager*Smart* Data Handling Software
Power supply	230 Vac ±10%, 50/60 Hz ±2 Hz, 1400 VA
Dimensions (cm)	Height 50 (54 with fittings); Width 59, Depth 58
Mass (kg)	65
Sound Pressure Level	Less than 70 db (A)

Instrument Configurations

The Flash IRMS Elemental Analyzer is able to be coupled to an Isotope Ratio Mass Spectrometer (IRMS) for the following determinations. See Table 2.

Table 2. EA IsoLink IRMS System for CNSOH Analytical Capabilities

Configuration	Description
H	Quantitative (weight%) and stable isotope analysis of hydrogen on H_2.
O	Quantitative (weight%) and stable isotope analysis of oxygen on CO.
N	Quantitative (weight%) and stable isotope analysis of nitrogen on N_2.
C	Quantitative (weight%) and stable isotope analysis of carbon on CO_2.
S	Quantitative (weight%) and stable isotope analysis of sulfur on SO_2.

IMPORTANT Oxygen and hydrogen analyses are performed using the **left** furnace (high temperature conversion). Nitrogen, carbon and sulfur analyses are performed using the **right** furnace for combustion.

Software Requirements

Thermo Scientific Isotope Ratio Mass Spectrometer series require the use of the Isodat Software Suite. In most cases the installation of Isodat Software Suite is sufficient to operate the EA IsoLink IRMS System for CNSOH. See "Isodat Software Suite" on page 64.
Table 3 describes the minimum hardware required to run the software.

ATTENTION If you want to operate with the EA IsoLink CNSOH as a stand-alone instrument, you need to install Eager*Smart* Data Handling Software on you computer. See Chapter 9, "Running the Flash IRMS as Stand-alone Instrument."

Table 3. Minimum Requirements for Personal Computer

Components	Description
Computer	Any PC can be used, including laptop computer
	Operating System: Windows® 2000 / XP* / Vista / 7* / 8
	Pentium Processor minimum 256 MHz
	Hard drive with at least 1 GB free
	One free COM port for instrument control
	One free COM port for balance, if required
	One free USB port
Monitor	Color 1024 x 768 or better
Printer	Any printer accepted by the operating system

* Required by Isodat 3.0

Instrument Basics

The Flash IRMS Elemental Analyzer is part of the EA IsoLink CNSOH IRMS System.
The Elemental Analyzer comprises:

- **Two furnaces** — The **left furnace**, for high temperature conversion analysis, contains a silicon carbide heater element. The heater element is immersed into a refractory material contained in a metallic compartment. This furnace is used only for the **H** and **O** isotope ratio determinations.
  The **right furnace** contains a quartz candle on which an electric resistance is wound.
  The candle is immersed into a refractory material contained in a metallic compartment.
  This furnace is used only for the **N**, **C** and **S** isotope ratio determination.
- **Furnace Temperature Regulation** — The temperature is monitored by a thermocouple of Pt-Pt/Rh type appropriately located in the furnace.
- **Furnace Cooling** — The cooling time varies according to the operating temperature setting and room temperature.
- **Thermal conductivity detector (TCD)** — Located in a thermostatic chamber at controlled programmable temperature. This chamber also accommodates the analytical column.
- **Chromatographic columns** — The chromatographic column performs the chromatographic separation of the reaction products generated during the combustion or high temperature conversion process. The chromatographic column for high temperature conversion is placed in the thermostatic chamber of the TCD detector according to the instrument configuration.
  The analytical column for the combustion side (N, C and S analysis) is placed in a separate ramped GC oven which can be heated and cooled in a fast way.

Description	Qty	Part number
H/O separation column (SS; 1 m; 1/8-in. OD)	1	260 08240
IRMS separation column (SS; 60 cm; 1/8-in. OD)	1	260 08241
IRMS NC separation column (SS; 60 cm; 1/8-in. OD)	1	260 08242
Sulfur separation column (Teflon®; 80 cm; 1/8-in. OD)	1	260 08243

- **Adsorption filters** — They can be made of glass or Plexiglas® according to the analytical configuration.
- **Reactors** — The H and O configurations require the use of a special cylindrical ceramic reactor filled with special catalyst.

  There are two different sizes available:

  – reactor of 18 mm OD

  – reactor of 25 mm OD (Macro Reactor)

> **Note** The EA IsoLink IRMS System for CNSOH comes standardized for a 18 mm OD reactor. For 25 mm reactor tubes the MAS Plus and the top of the EA must be modified. See the section "Upgrading EA for 25 mm OD Macro Reactor" on page 133.

❖ To set the temperature

The following procedure is recommended for heating up the furnace of the combustion reactor when operating with chromium oxide (Cr_2O_3) at 1020°C:

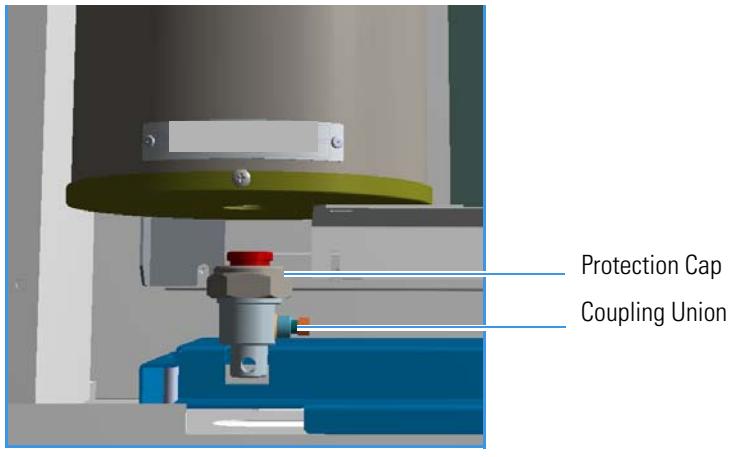
1. Increase the temperature from room temperature to 400°C and check the background signals on the Mass Spectrometer. Check the system for leaks.
2. Increase the temperature from room 400°C to 900°C in steps of 100°C and hold at 900°C for at least 15 min. This avoids melting the copper within the reactor, which will cause poor performance.
3. Increase it to the operating temperature of 1020°C..

WARNING Immediate temperature increase to 1020 °C can cause copper melting. Cooling down in steps can avoid reactor breaking. Alternatively, you can use the HeatUp and CoolDown script in Isodat. Right-click on the Flash IRMS visualization window in Isodat and choose the appropriate context menu item. See page 82.

- **Protective Cap**

IMPORTANT Before the EA IsoLink IRMS System for CNSOH is shipped, the pneumatic circuit is protected by a cap mounted on the coupling union located on the base of the left furnace compartment as shown in Figure 1 on page 4. This is to avoid humidity in the chromatographic column. The cap must be removed prior to the reactor installation.

Figure 1. Protective Cap



- **Autosampler** — Performs the automatic injection of samples into the reactor.
- **Pneumatic compartment** — Consists of two pressure reducers, two pressure gauges, and of several lines fitted with an thermo-regulator electronic flow controller (EFC-t), which ensures the switching between carrier gas and oxygen, and controls the flow values.
  - **He^M device** — Reduces the consumption of helium.
  - **Automatic switching box** — Switches automatically from **combustion** N,C, S configurations to **pyrolysis** O, H configuration, and vice-versa.
- **Electrical compartment** — Comprises the electronic boards for the instrument power supply and control.
- **User interface** — The instrument is not provided with independent keyboard and display. A synoptic on the instrument front allows you to monitor the instrument status.

Safety Information

CAUTION The instrument must be used according to the specifications of this guide. Improper use can adversely affect the instrument protection. If the equipment is connected to optional instruments, such as computer, balance, and so on, the degree of insulation of peripheral devices should be equivalent or higher (double or reinforced) than that of the EA IsoLink IRMS System for CNSOH. The analyzer operation requires the use of chemical substances having different hazard specifications. Before using chemicals, please read the hazard indications and information reported in the Material Safety Data Sheet supplied by the manufacturer referring to the relevant CAS (Chemical Abstract Service) number.

Safety Cut Off Device

When an alarm condition is detected, this device cuts off the power to the heating resistors of the furnaces and to the EA Oven.

Note For more details, see the section "Safety Cutoff" on page 138.

Labels Location on the Instrument

The following illustrations show the location of the safety labels attached on the instrument.

Figure 2. EA IsoLink IRMS System for CNSOH: Analyzer Model, Serial Number and Electrical Data

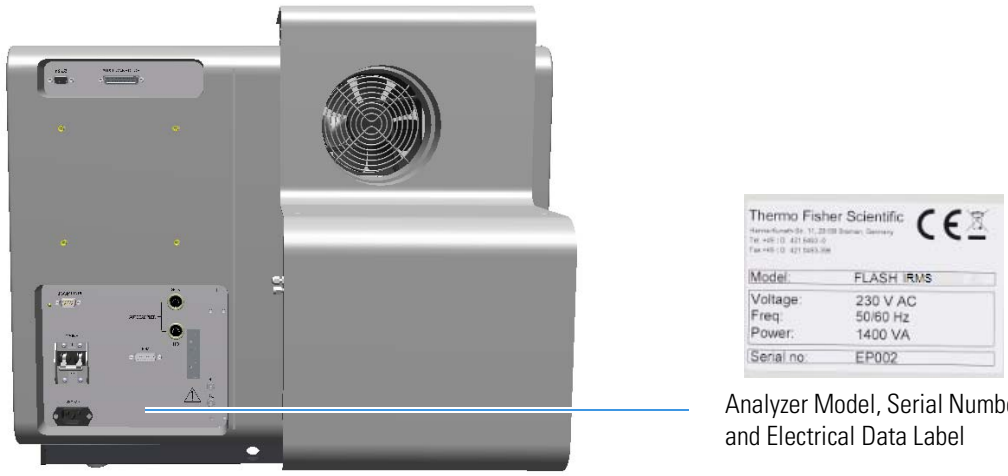


Figure 3. EA IsoLink IRMS System for CNSOH: Alert Labels

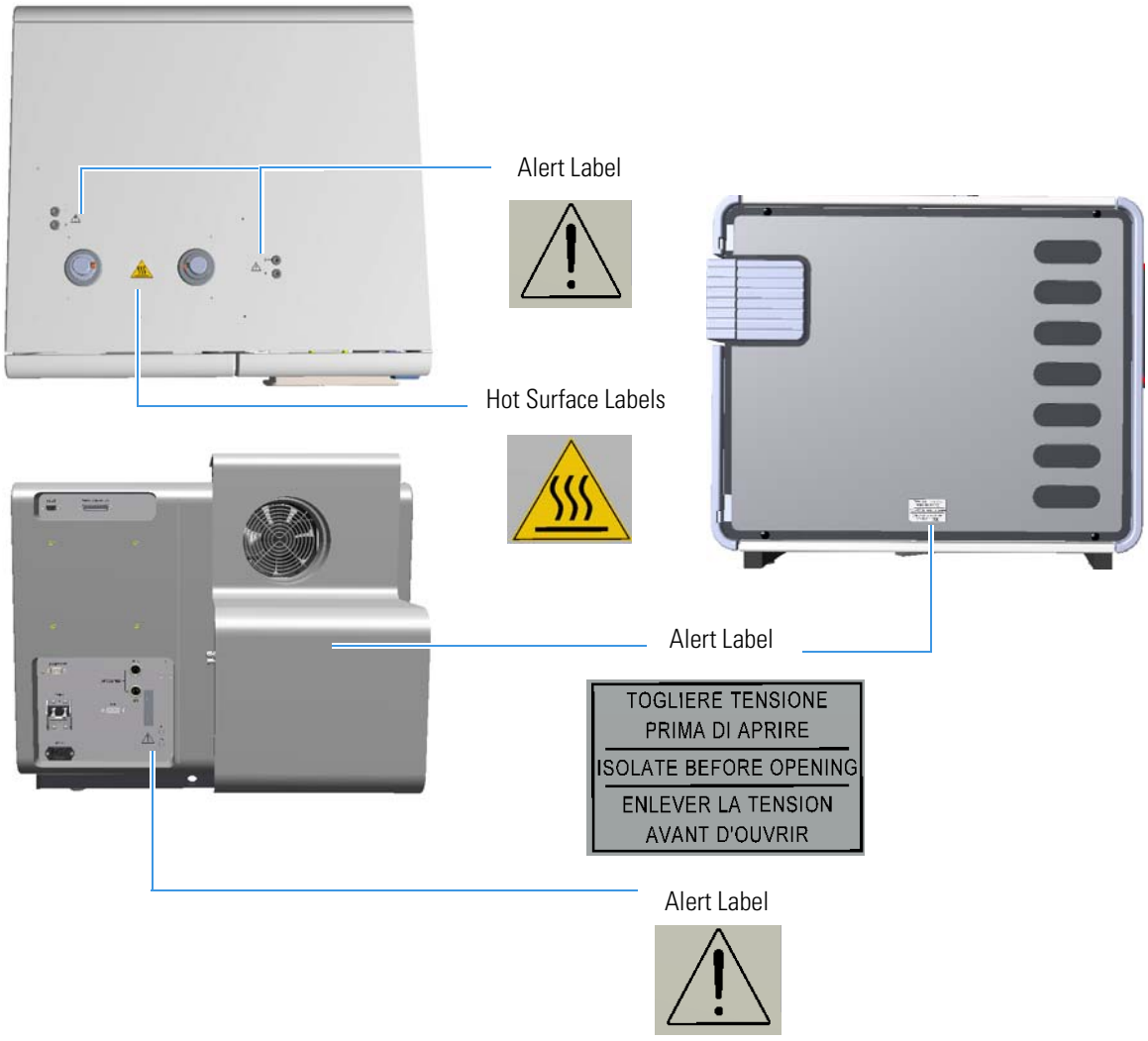
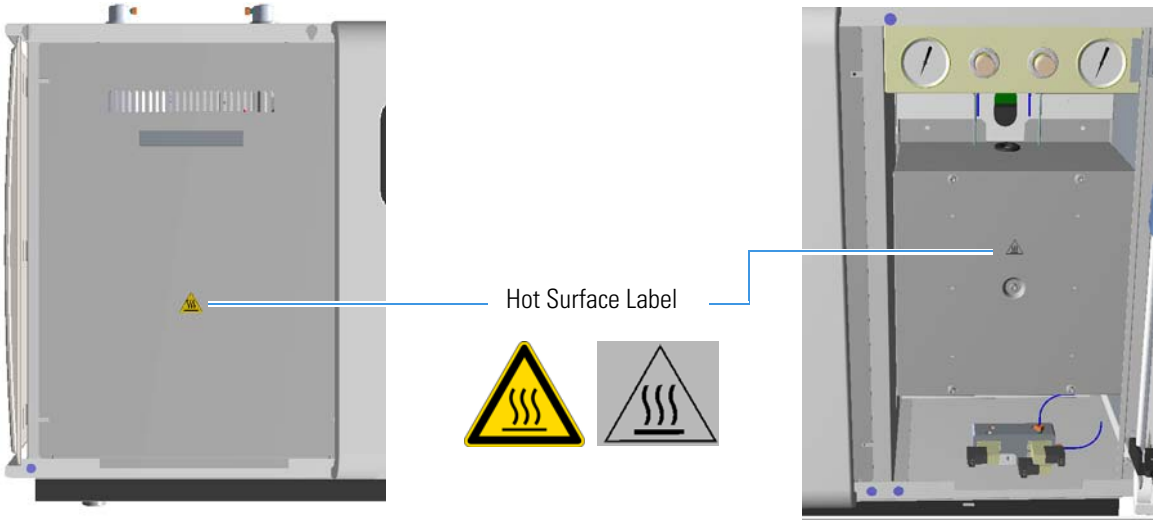


Figure 4. EA IsoLink IRMS System for CNSOH: Hot Surface Labels



Use of Gases

CAUTION Before using gases, carefully read the hazard indications and information reported in the Material Safety Data Sheet (MSDS) supplied by the manufacturer referring to the CAS (Chemical Abstract Service) number.
It is the your responsibility to see that all local safety regulations for the use of gases are obeyed.

Gas Supply

The Flash IRMS Elemental Analyzer uses the following gases:

- Helium (He) as carrier gas and purge gas [*CAS number 7440-59-7*]
- Oxygen (O_2) as gas for sample oxidation [*CAS number 7782-44-7*]

Note ConFlo II/III/IV and IRMS Delta models (**TF Bremen manufacturing**) use the following gases:

- Helium (He) as carrier gas and purge gas [*CAS number 7440-59-7*]
- Carbon Monoxide (CO) as standard gas [*CAS number 630-08-0*]
- Hydrogen (H_2) as standard gas [*CAS number 1333-74-0*]
- Nitrogen (N_2) as standard gas [*CAS number 7727-37-9*]
- Carbon Dioxide (CO_2) as standard gas [*CAS number 124-38-9*]
- Sulfur Dioxide (SO_2) as standard gas [*CAS number 7446-09-5*]

Gas Purity

The EA IsoLink IRMS System for CNSOH requires the use of ultra-high purity gas chromatography grade (99.999%) gases.

Maximum Pressure

The maximum pressure of the Elemental Analyzer gas supplies is 700 kPa (7 bar).

Nominal Pressure

The nominal pressure of the Flash IRMS EA gas supplies are:

- 250-300 kPa (2.5-3 bar) for He
- 250-300 kPa (2.5-3) for O_2

Note For the nominal pressure of the gases used by ConFlo II/III/IV and IRMS Delta models (**TF Bremen manufacturing**), refer to the relevant manuals.

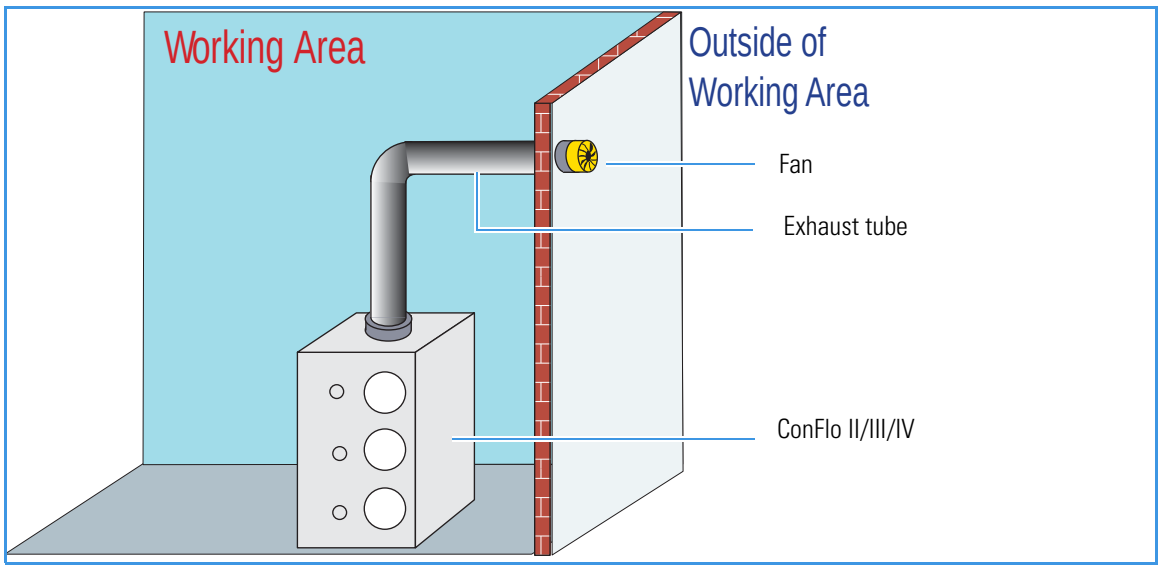
Note The pressure must be adjusted to about **4 kPa** through the reducing valves mounted at the cylinder.

Precaution for Carbon Monoxide

ATTENTION Carbon Monoxide is toxic!
When working with this gas good ventilation is essential. Otherwise, the gas can be hazardous to your health! The use of a CO detector with an alarm is strongly recommended.

Install an exhaust tube on top of your ConFlo II/III/IV, as shown in of the example of Figure 5, to remove the toxic carbon monoxide (CO) from inside the ConFlo II/III/IV out of your working area.

Figure 5. Exhaust Tube Connection



Precaution for Sulfur Dioxide

ATTENTION Sulfur Dioxide is toxic!
When working with this gas good ventilation is essential. Otherwise, the gas can be hazardous to your health!

Sulfur dioxide is s colorless gas with a distinctive pungent odor that will alert the user of a leak in ample time before irritation can occur. It is somewhat denser then air and will sink to low-lying spaces. Because of the density and expansion factors, never place sulfur dioxide containers in direct sunlight or exposed to other direct or indirect heating sources.
Sulfur dioxide is corrosive to many common metals and other substances.

Using Hydrogen

CAUTION The use of hydrogen requires the operator's strict attention and compliance with special precautions due to the hazards involved. The use of an H_2 detector with an alarm is strongly recommended.

Hydrogen is a dangerous gas, particularly in an enclosed area when it reaches a concentration corresponding to its lower explosion level (4% in volume). When mixed with air it can create an explosive mixture.

Use the following safety precautions when using hydrogen:

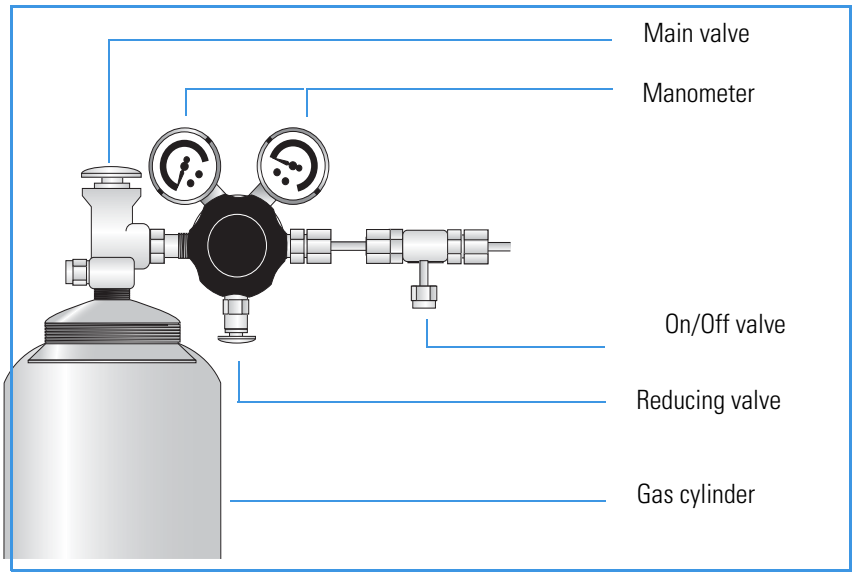
- Ensure that the hydrogen cylinder complies with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure the gas supply is turned completely off when connecting hydrogen lines.
- Perform a *bubble test* to ensure that the hydrogen lines are leak-tight before using the instrument as described in the paragraph "Leak Test." Avoid spraying any electrical components during the bubble test.

Leak Test

Before starting the system, a leak check has to be performed as described in the following "Performing a leak check," operating sequence.

❖ **Performing a leak check**

Figure 6. Gas Cylinder



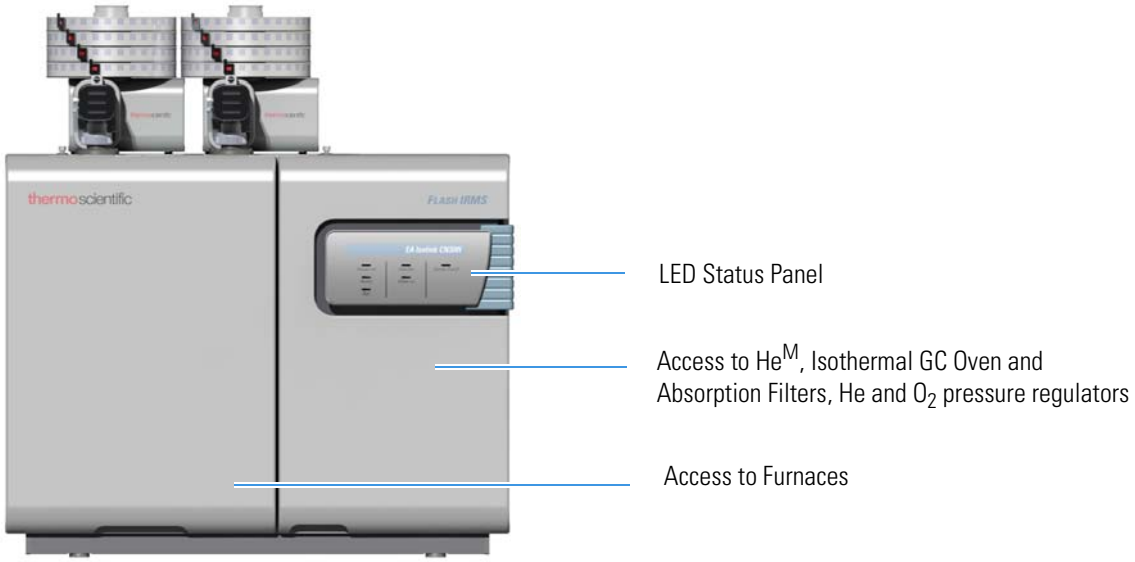
Referring to Figure 6, proceed as follows:

1. After mounting the reducing valve to the gas cylinder both “on/off-valve” and “reducing-valve” must be open.
2. Open the main valve for two or three seconds to let the gas purge the whole valve system.
3. Close the on/off-valve, then close the main-valve.
4. Mark manometer positions of the on/off-valve and main-valve, and wait for 10 - 15 min.
5. A leak may be present if the manometer positions have changed.
6. To detect a leak, use soap solution on all valves and connections. Check for bubble formation. Remove soap solution quickly and carefully after test.

Front Panel

The front panel of the Elemental Analyzer is shown in Figure 7.

Figure 7. Instrument Front Panel

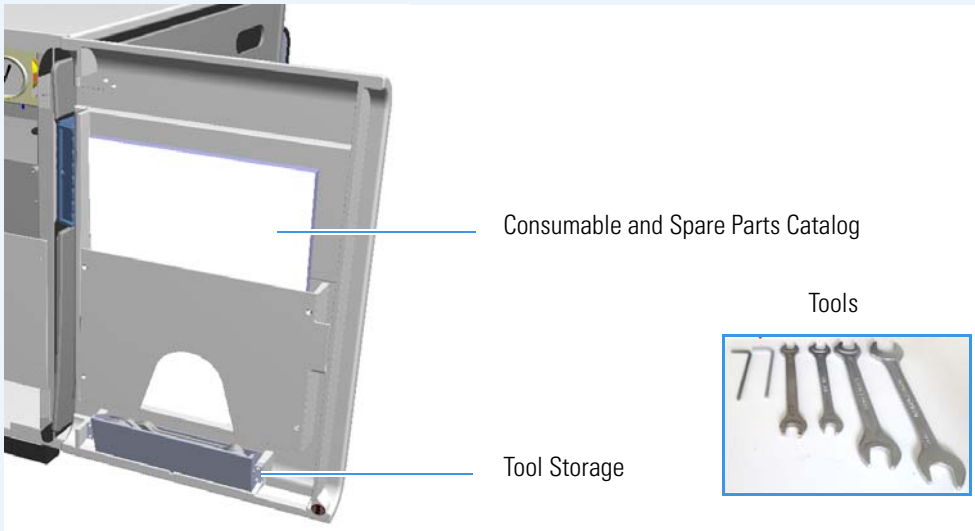


The front panel comprises:

- A furnaces compartment. Also see the section "Furnaces Compartment" on page 13.
- A LED status panel. Also see the section "Status Panel" on page 25.
- Oven compartment including the pressure regulators, pressure gauges, adsorption filters, isothermal GC oven housing, the TCD detector, the gas chromatographic column, and the He^M module. See the section "Oven Compartment" on page 16.

Note On the internal wall of the front door you will find holders designed for a paper copy of the Consumables and Spare Parts Catalog, and tools for maintenance. See Figure 8.

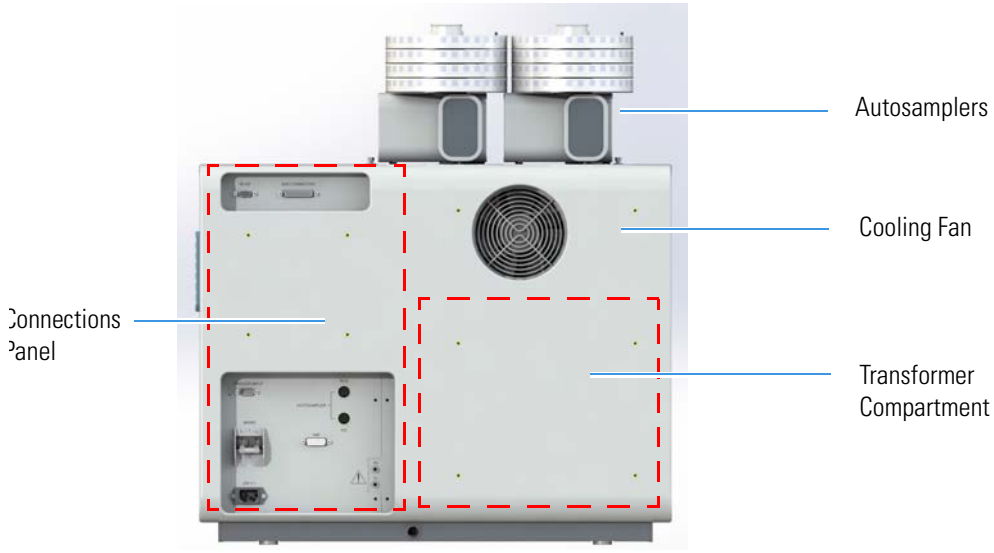
Figure 8. Right Door Internal View



Back Panel

The back panel of the Elemental Analyzer is shown in Figure 9.

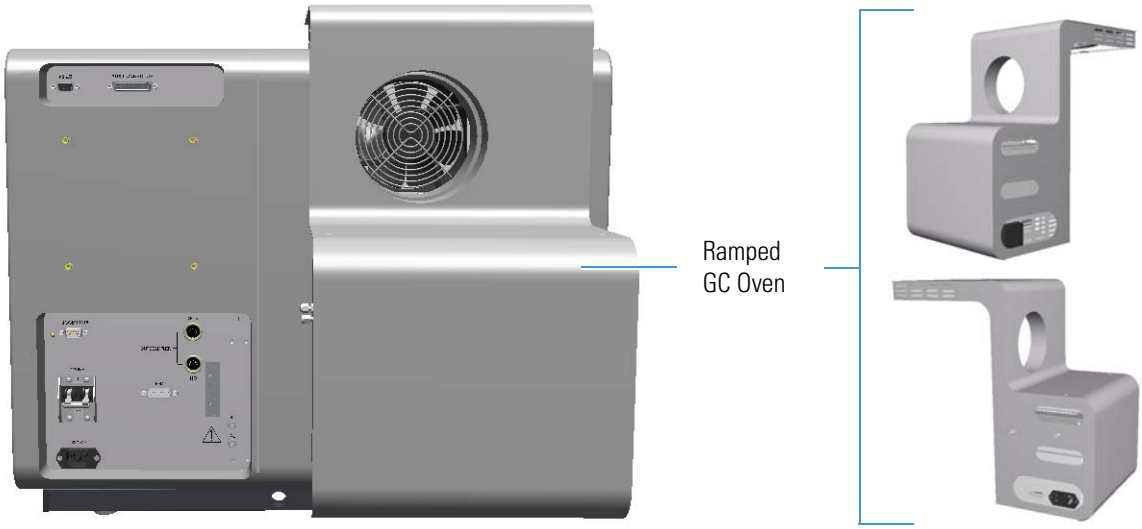
Figure 9. Instrument Back Panel



On the back panel, you will find:

- The cooling fan.
- Interface, gas inlets, and electrical connections. See the section "Connections Panel" on page 21.
- Transformers compartment. Access to the transformer compartment is obtained by removing the back panel cover. See the section "Transformers Compartment" on page 23.
- Place holders for the ramped GC Oven for EA IsoLink CNSOH. See Figure 10 and the section "Ramped GC Oven" on page 28.

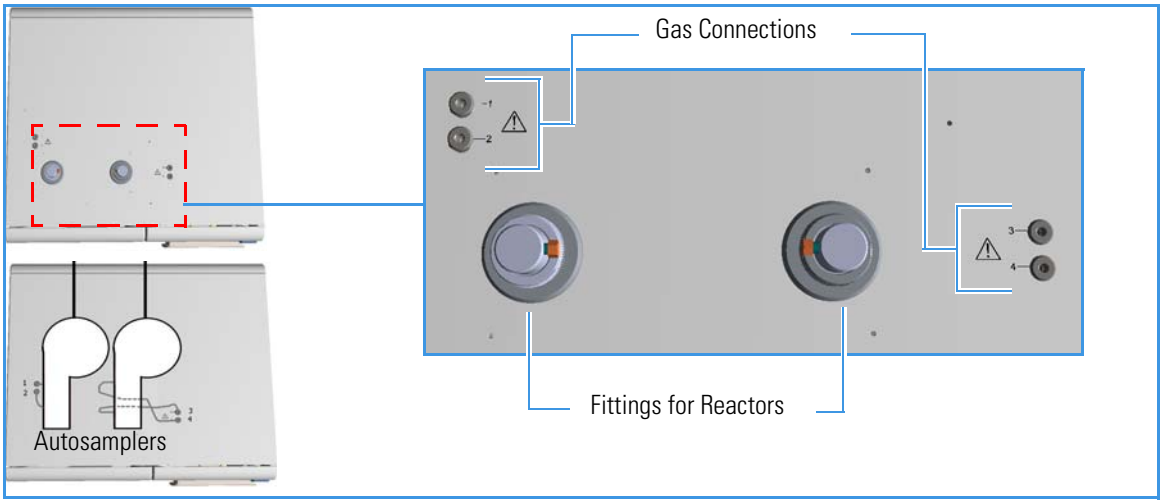
Figure 10. Instrument Back Panel with the Ramped GC Oven



Top Panel

The top panel of the Elemental Analyzer is shown in Figure 11.

Figure 11. Instrument Top Panel



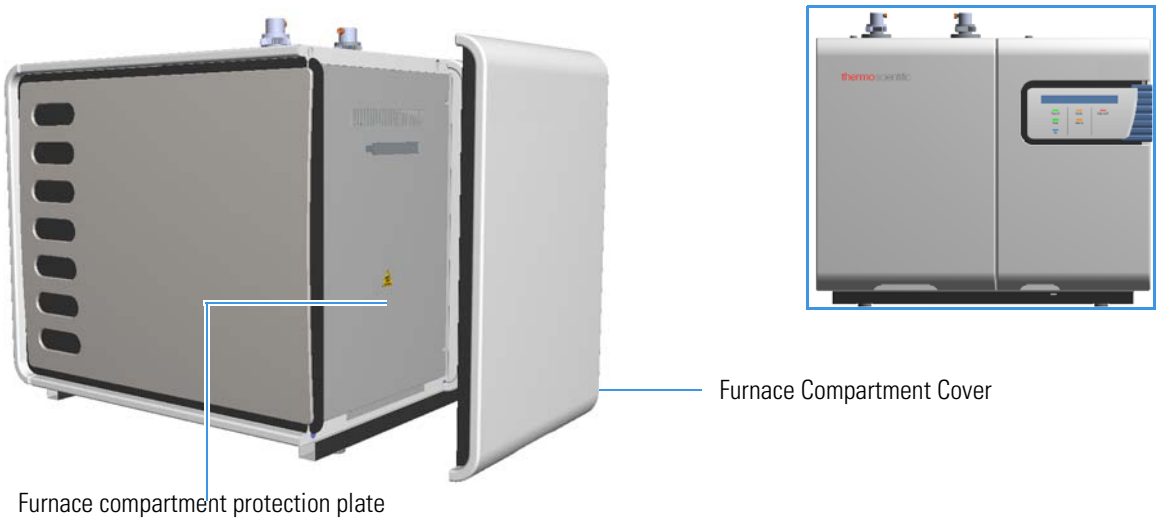
The top panel comprises:

- Fittings for mounting and securing reactors and autosamplers.
- Fittings for Helium carrier gas and ConFlo™ Universial Interface™ connection.

Furnaces Compartment

The furnaces compartment can be accessed from the instrument front by removing (lifting) the cover. See Figure 12.

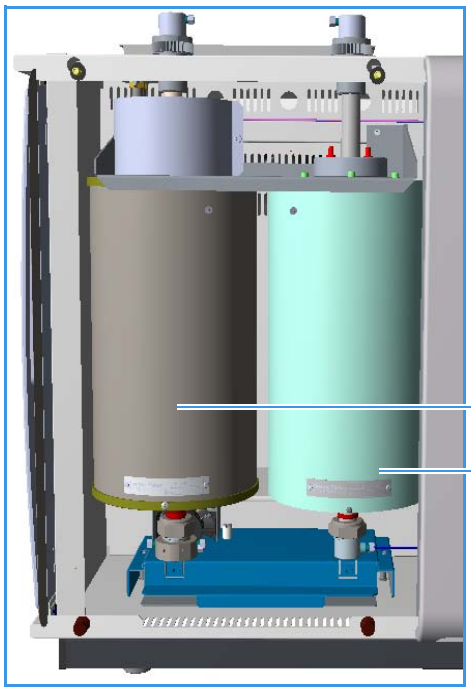
Figure 12. Furnace Compartment with Protection Plate

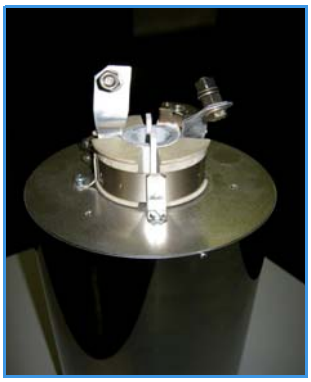


WARNING Do not open the furnace compartment during operation due to the very high temperatures reached during operation. The protecting plate should only be removed when the temperature of the furnace is near that of room temperature.

The furnaces are accessible by removing the protecting plate. See Figure 13.

Figure 13. Furnaces Compartment





In the software, the furnaces are limited to the following maximum temperatures to avoid reduced lifetime and damage:

- LEFT Furnace: 1450 °C
- RIGHT Furnace: 1100 °C

The furnace temperature is monitored by a thermocouple located inside the furnace. The furnaces are cooled when required by the operator or if there is a safety cut-off. The cooling time depends on the operating temperature and the room temperature.

❖ Instruction for operation of the furnace

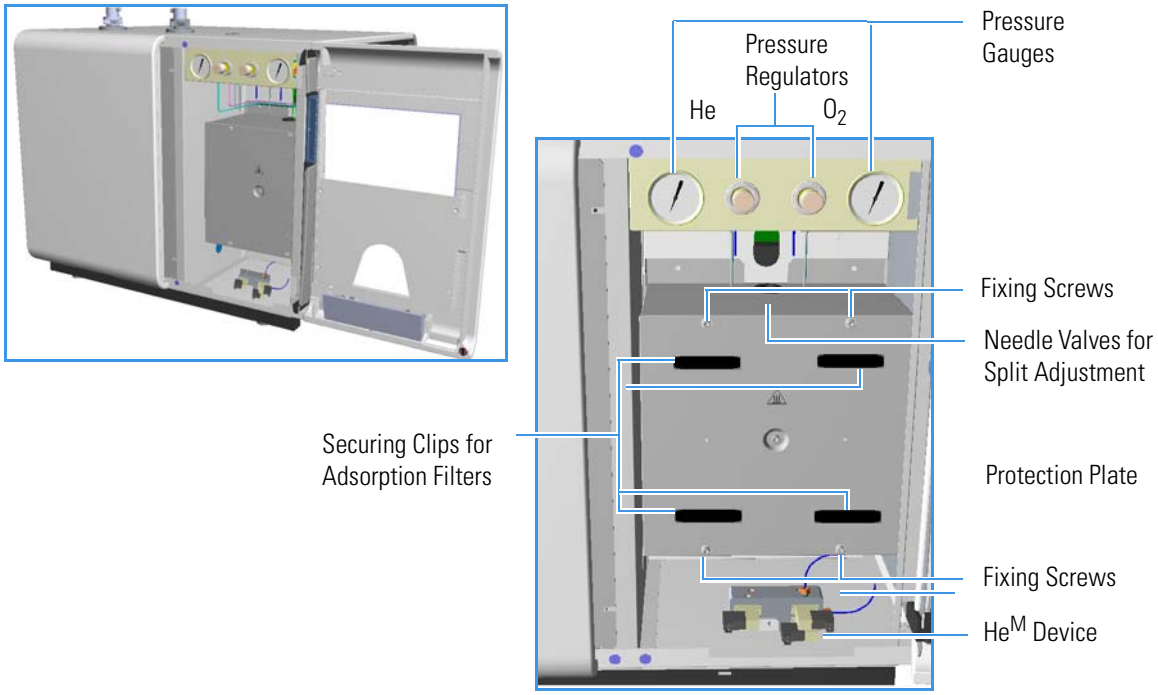
The furnace temperature is monitored by a thermocouple located inside the furnace. The furnaces are cooled when required by the operator or if there is a safety cut-off. The cooling time depends on the operating temperature and the room temperature.

1. The furnaces should not be operated above the maximum temperature stated in the operating instructions, which is 1100 °C and is limited by the software.
2. When the Elemental Analyzer is not being using for sample analysis, either for periods of several hours (e.g. overnight) or periods lasting several days or longer, the furnaces should be placed into Stand-By Mode as defined by the Standy-By Mode which can be activated automatically at the end of a sample sequence.
    a. If the instrument is not used for a long period of time (e.g. several weeks) it is recommended to switch off the furnaces. The furnaces should be cooled down using the furnace Cool Down procedure in the Isodat Software Suite.
    b. After a Stand-By period or period where the furnaces are switched off, the furnaces should be brought back to operating temperature following the furnace heat-up procedure defined in the manual.
3. The operator must not cause a short-circuit with the furnace heating element or it's connections, or the furnace thermocouple. Only a qualified service engineer should check or test this. Operator interference on these parts is not covered by the warranty.

Oven Compartment

The oven compartment is located behind the right front door of the instrument and can be accessed by opening the front door. Figure 14 shows the inside of the oven compartment.

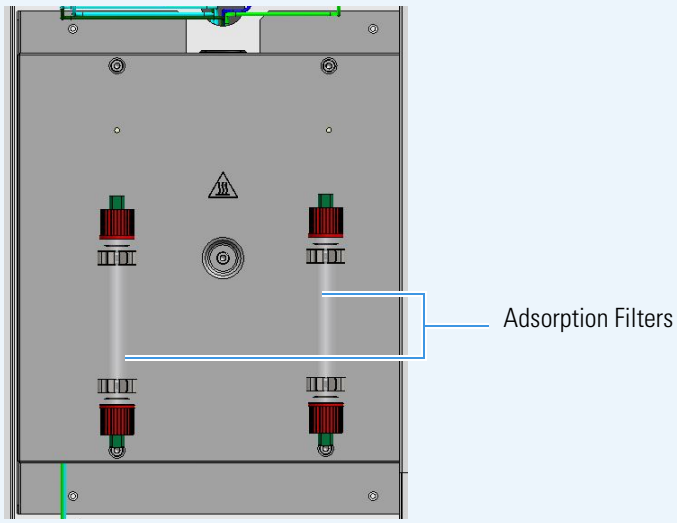
Figure 14. Oven Compartment Internal View



The oven compartment houses the helium (He) and oxygen (O_2) pressure regulators and pressure gauges, and the thermostatic chamber containing the thermal conductivity detector (TCD) and the gas chromatographic column located behind the protecting plate. See "To access to the thermostatic chamber" on page 17. The adsorption filters are housed in this compartment and are attached to the protecting plate using securing clips.

Note Two adsorption filters are required as shown in Figure 15. One filter is required for the High Temperature Conversion analytical channel and one filter for the combustion analytical channel.

Figure 15. Adsorption Filters Installed



❖ **To access to the thermostatic chamber**

1. Open the right side door, which can be moved 180°.
2. To access the TCD and GC Columns, first remove the adsorption filters from the fastening clips.
3. Then, remove the four fixing screws on the protecting plate. See Figure 16.

Figure 16. Access to Thermostatic Chamber

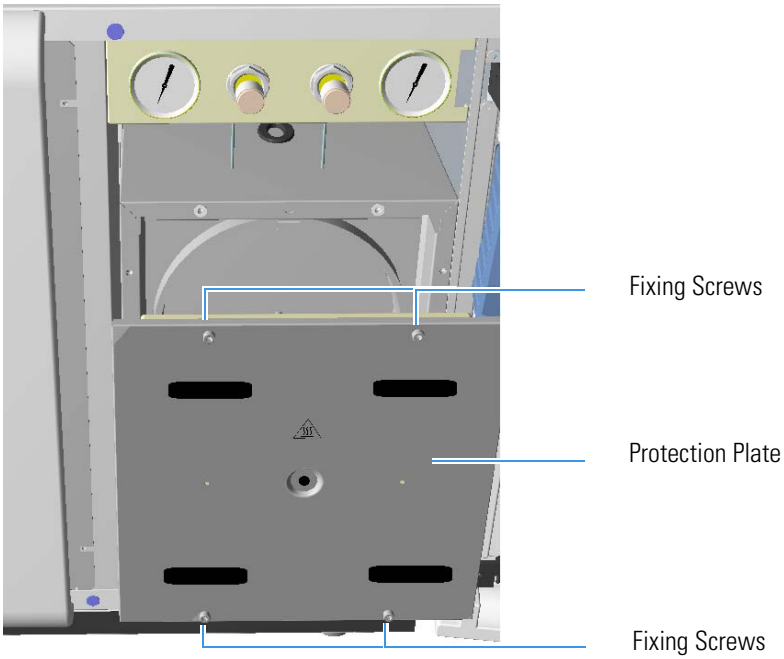
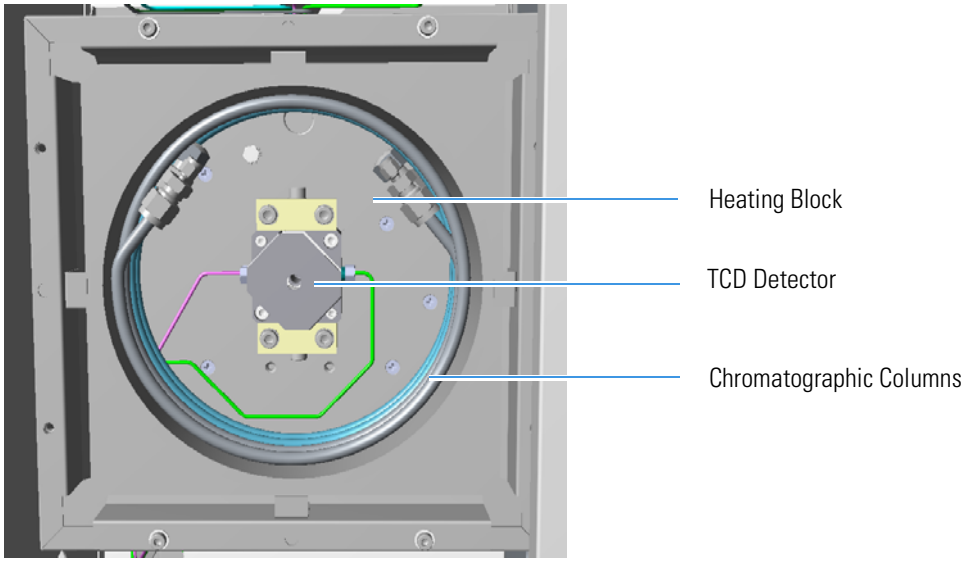


Figure 17 shows the detector compartment, the heating block surrounding the thermal conductivity detector (TCD), and the gas chromatographic column.

Note One chromatographic columns is installed for OH analysis with the high temperature furnace. A second chromatographic column may be installed for combustion if no ramped GC oven is used.

Figure 17. Thermostatic Chamber Internal View



Detection System Description

It consists of a thermal conductivity detector (TCD) sensitive to any substance with thermal conductivity other than that of the carrier gas used.

The detector consists of a stainless steel block provided with two pairs of filaments (generally of tungsten/rhenium) having the same electrical resistance. The detector is housed in a thermally insulated metal block (detector oven) and maintained at constant temperature.

The two pairs of filaments are electrically connected according to a Wheatstone bridge circuit powered at constant voltage. The first pair of filaments is fed with pure carrier gas (reference channel), whereas the second pair is fed with the gas flowing from the reactor (analytical channel). When the bridge is powered, the filaments heat at a temperature (resistance) that is a function of the thermal conductivity of the gas feeding the filaments. The reference channel is exposed only to pure carrier gas, whereas the analytical channel is exposed to the reactor effluents (carrier gas + sample).

When pure carrier gas flows through both the reference and the analytical channels, a constant temperature gradient is established between the elements and the detector walls, and the Wheatstone bridge is balanced, namely there is no output signal. As a component is eluted, a change in heat transfer occurs, with consequent variation of the filaments temperature. Since electrical resistance is a function of temperature, the bridge unbalances and the detector generates a signal proportional to the difference in thermal conductivity between the eluted component and the carrier gas. The output signal is then sent to the data acquisition board.

Note The filaments are powered constantly at 5 V and are electrically protected if their temperature exceeds 220 °C (Safety Cut Off).

Electrical Compartment

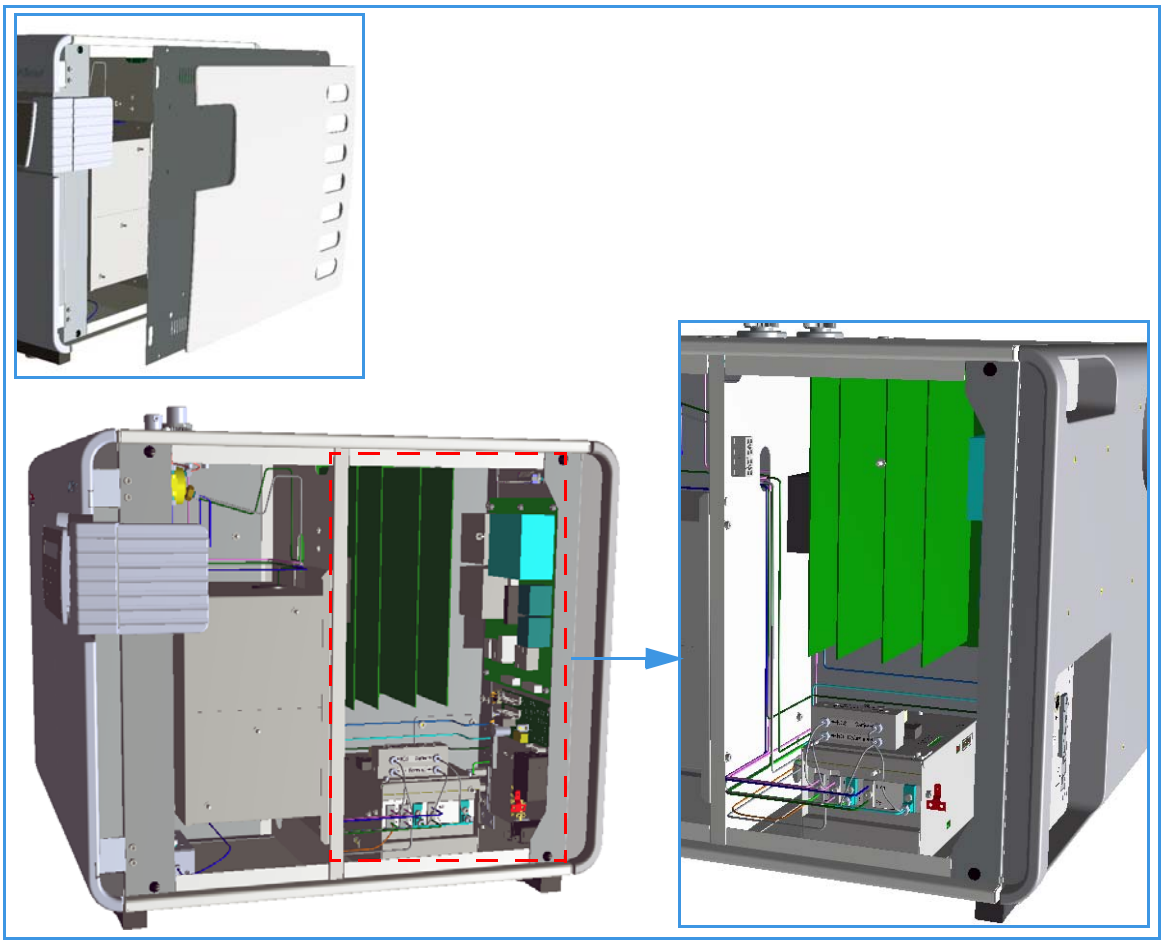
It is located on the right part of the instrument, and it is accessible by removing the right side cover. Behind the electrical compartment, there is the Connections Panel. For more details, see the section "Connections Panel" on page 21.

WARNING DO NOT OPEN the electrical compartment because there are no user serviceable parts inside. Any operation inside the compartment must be carried out by authorized and trained Thermo Fisher Scientific personnel.

The electrical compartment, shown in Figure 18, comprises:

- Low voltage compartment
- High voltage compartment
- EFC electronic flow controller for gas regulation

Figure 18. Electrical Compartment Internal View



Low Voltage Compartment

It contains the electronic boards to operate and control the instrument. These boards are interlocked through a mother board.

Table 4 reports the function of each electronic board present in the low voltage section:

Table 4. Description of the Function of the Electronic Boards (Sheet 1 of 2)

Board	Function
MB 1112	Mother board. It provides interlocking between low voltage boards and with the rest of the instrument. This board can be connected to a NiCd 3,6 V; 280 mA/h rechargeable battery located nearby. The rechargeable battery replacement must be performed by specialized technical personnel
CPU 1112	This board has full control of the instrument operation. It controls the communication between operator and machine through Eager*Smart* Data Handling Software. Actuates the Safety Cut Off device, which puts the instrument in safe conditions, when an alarm condition occurs.
HWD 1112	Provides power supply to the TCD detector filaments. Allows the detector oven thermo-regulation and also amplifies and converts the detector signal to send it to the PC.

Table 4. Description of the Function of the Electronic Boards (Sheet 2 of 2)

Board	Function
TCR 1112	Operates and controls the furnaces thermoregulation.
PWR 1112	Receives voltage supplies from the TRF 1112 transformers board. Generates voltage supply for the electronic control circuits.
FP 1112	Synoptic panel

Main Voltage Compartment

It contains the mains power circuits and the Safety Cut Off device.

Table 5 details the function of each component present in the mains voltage section.

Table 5. Description of the components of the Main voltage compartment

Component	Description
TRF 1112 Transformers Board	Receives the mains power and supplies it to the following devices: • Cooling fan • Furnaces transformers • Heater of the detector thermostatic chamber Six fuses are provided on the board. See Table 6.
AC 1112 Furnaces Power Supply	Supplies 48 Vac power to the furnaces. It contains the SSR relays for the furnaces control. Also see the section "Devices for the Furnaces Control" on page 25. Two fuses are provided on the board. See Table 6.
Breaker	Instrument On/Off main switch.

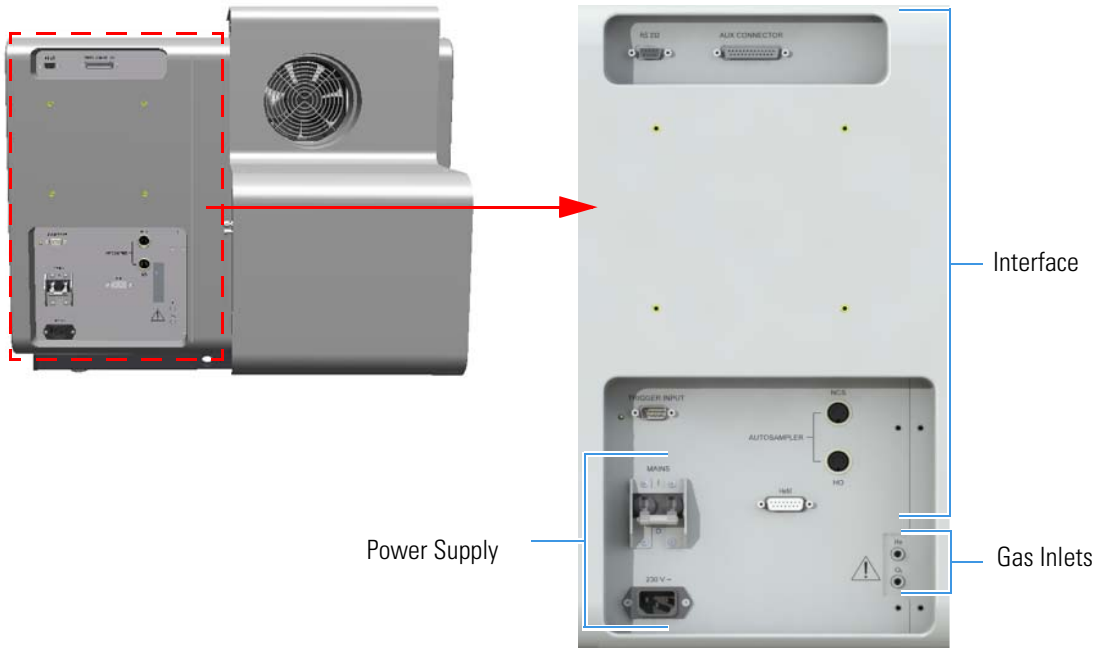
Table 6. Fuses of the High voltage compartment

Board	Fuse	Type	Protection
TRF 1112	F1	F1A; IEC 127/I (5 x 20 mm)	Power supply to LEFT and RIGHT furnaces transformers
	F2	F0.315A; IEC 127/I (5 x 20 mm)	Fan
	F3	F1.6A; IEC 127/I (5 x 20 mm)	Main power (Breaker)
	F4	F1A; IEC 127/I (5 x 20 mm)	LEFT and RIGHT furnaces transformers
	F5	F0.315A; IEC 127/I (5 x 20 mm)	Fan
	F6	F1,6A; IEC 127/I (5 x 20 mm)	Mains power (Breaker)
AC 1112	F1	FF12 A; IEC 269 (1.3 x 38 mm)	LEFT Furnace power circuit
	F2	FF12 A; IEC 269(1.3 x 38 mm)	RIGHT Furnace power circuit

Connections Panel

The connections panel is shown in Figure 19.

Figure 19. View of the Connections Panel



The connections panel is subdivided into the following connecting areas: **interface**, **power supply**, and **gases supply**.

Interface Area

The **interface** area comprises the connectors for connecting the autosamplers and the helium management device, and the automatic switching valve. See Figure 20.

Figure 20. Interface Area

- A 9-pin connector marked **RS 232** to dialog with the computer via serial line.
- A 25-pin connector marked **Aux Connector** for the remote start of the instrument.
- A 2-pin connector marked **Autosampler HO** for the MAS Plus autosampler installed on the **left channel** for HO determinations.
- A 2-pin connector marked **Autosampler NCS** for the MAS Plus autosampler installed on the **Right channel** for NCS determinations.
- A 9-pin connector marked **Trigger Input** for the control of the automatic switching valve.

- A 15-pin connector marked **He**^{M} for the control of helium management device.

Power Supply Area

The **power supply** area comprises the following components. See Figure 21.

Figure 21. Power Supply Are

- Breaker marked **Mains** to power the instrument ON/OFF.
  - Position I = instrument powered ON; — Position O = instrument powered OFF.
- 230 V; 50/60 Hz mains connector.

Gases Supply Area

The **gas supply** area comprises the gas inlet ports. See Figure 22.

Figure 22. Gases Supply Area

- The **helium** and **oxygen** gas inlet ports, marked **He** and **O**$_2$ are directly connected to the pressure regulators. Table 7 details the pressure value to be set for each gas inlet port.

Table 7. Gas Inlet Ports and Pressure Setting

Port	Description	Set TO:
He	Inlet port for helium or argon	250 kPa (2.5 bar, 36 psig)
O_2	Inlet port for oxygen	250-300 kPa (2.5-3 bar, 36-44 psig)

Gas pressures must be set and controlled through the pressure regulators and the pressure gauges of the instrument. Table 8 provides indications on the most currently used units of pressure.

Table 8. Pressure Units Conversion

To convert	Into	Multiply By:
kPa	bar	0.01
	psi	0.145
bar	kPa	100
	psi	14.51
psig	kPa	6.89476
	bar	0.0689476

Transformers Compartment

Located in the right bottom part of the instrument, the compartment of the transformer is accessible firstly by removing the back panel of the instrument, then by removing the relevant protection plate. See Figure 23.

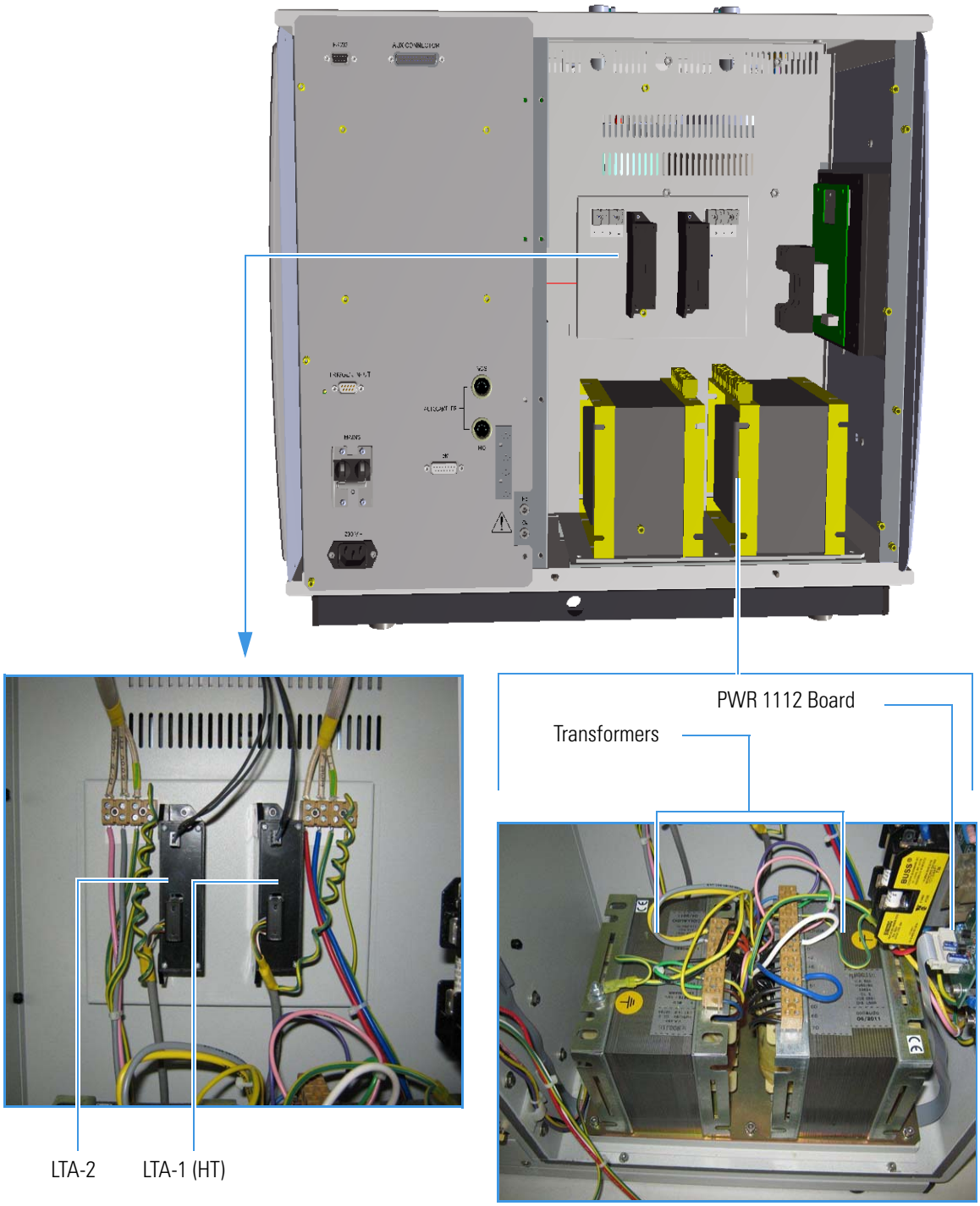
Figure 23. Accessing the Transformer Compartment

The compartment contains the electrical devices for powering the furnaces and controlling their temperature.

WARNING DO NOT OPEN the transformers compartment, there are no user serviceable parts inside. Any operation inside the compartment must be carried out only by authorized and trained Thermo Fisher Scientific personnel.

Figure 24 shows the devices contained in the compartment.

Figure 24. Transformer Compartment Internal View



Devices for the Furnaces Control

Table 9 describes the function of each device:

Table 9. Description of the Devices Controlling the Furnaces

Device	Function
LTA-1 LEFT LTA-1 RIGHT	They read the values of the thermocouple present in the relevant furnace and send the signals to the **TCR 1112** board.
SSR LEFT SSR RIGHT	Solid State Relays (SSR) contained in the **AC 1112** board. Each SSR is coupled with a proper safety sensor, which detects any malfunction. The SSR control the power supply to the relevant furnace and cut off power to the heating resistor when the thermocouple detects temperature values exceeding the setpoint.

Devices Supplying the Furnaces

Table 10 details the function of each device:

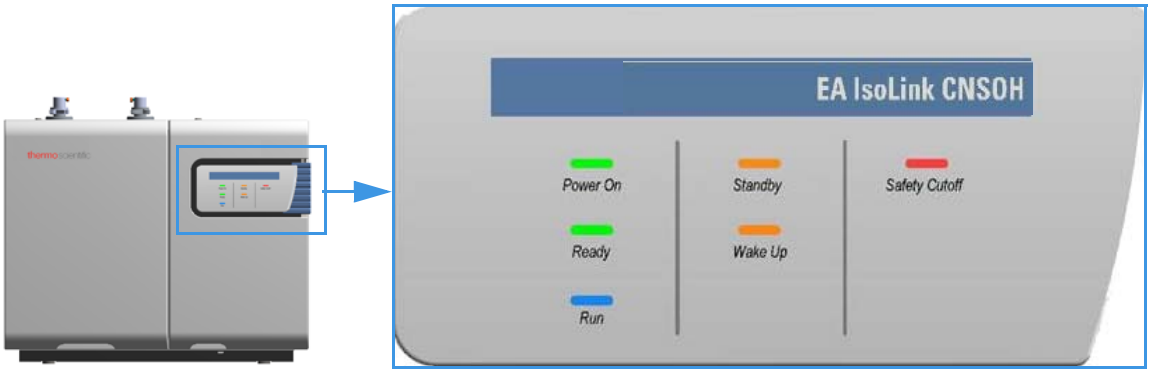
Table 10. Description of the Devices Supplying the Furnaces

Device	Function
T1 Transformer	Supplies 48 V voltage to the right furnace resistor. It is provided with a safety thermal protection, which cuts off power in case of overheating.
T2 Transformer	Supplies 48 V voltage to the left furnace resistor. It is provided with a safety thermal protection, which cuts off power in case of overheating.

Status Panel

The LED status panel shows the instrument operating conditions, and it is located on the right side of the instrument front panel. See Figure 25.

Figure 25. LED Status Panel



Each LED lights up when the relevant function is active. Table 11 illustrates the meaning of each function:

Table 11. Status LED Description

LED	Meaning
Power On	When lit, the instrument is powered on.
Ready	When lit, the instrument is ready to run analyses.
Run	When lit, an analysis is in progress.
Standby	When lit, the instrument is in standby condition. During this condition, helium gas flows are decreased to 10 mL/min, and the furnace temperatures reduced to 50% of the set value.
Wake Up	When lit, the instrument has been programmed for a timed automatic startup (Ready Condition).
Safety Cutoff	When lit, the instrument is in safety shut-off. Gas flows are stopped, furnaces are switched off and TCD is switched off.

Autosamplers

The EA IsoLink IRMS System for CNSOH can be configured with the following autosamplers: **MAS Plus** for solid samples, and **AI 1310/AS 1310** for liquid samples.

MAS Plus Autosampler for Solid Samples

It is the standard autosampler for solid samples mounted directly on the connecting fitting of the concerned channel provided with the proper reactor tube. See Figure 26.

Figure 26. MAS Plus Autosampler for Solid Samples

Its modular structure allows to run up to 125 unattended analyses. The base unit is provided with one 32-position sample tray. It can accommodate three additional 32-position trays to reach a capacity of 125 samples. Each sample tray is installed in a specific position defined by the numbering. Therefore, they are not interchangeable. The sample numbering is detailed in Table 12.

Table 12. Sample Tray Numbering

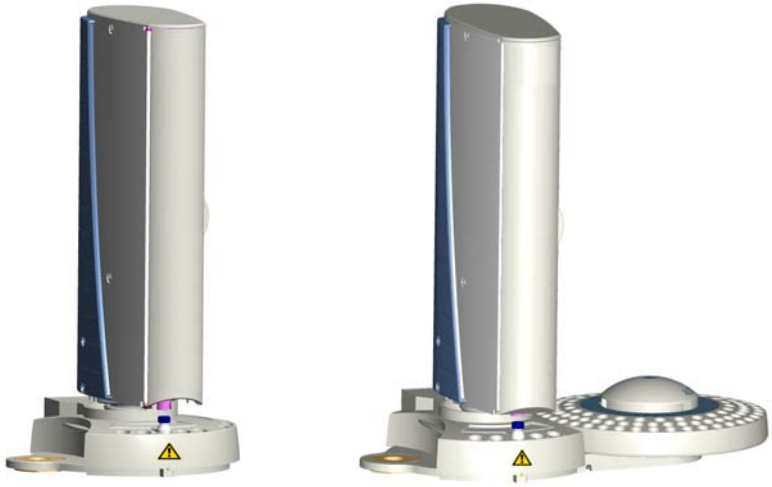
Sample Tray	Locating Mark	Numbering
#1	Seat marked 1 (one)	from 1 to 32
#2	Seat marked 0 (zero)	from 33 to 63
#3	Seat marked 0 (zero)	from 64 to 94
#4	Seat marked 0 (zero)	from 95 to 125

Note For installing the MAS Plus autosampler on your EA IsoLink IRMS System for CNSOH see Chapter 4, "Installing MAS Plus Autosampler."

AI 1310 /AS 1310 Autosampler for Liquid Samples

These are optional autosamplers for the analysis of liquid samples. It is mounted on the Flash IRMS EA by means of the appropriate support. See Figure 27.

Figure 27. AI 1310 and AS 1310 Autosamplers for Liquid Samples



AI 1310 AS 1310

The autosampler consists of:

- A sampling unit
- An 8-position (AI 1310) or 105-position (AS 1310) sample tray

Note For installing the AI 1310/AS 1310 autosamplers for liquids on your EA IsoLink IRMS System for CNSOH see Chapter 5, "Installing AI 1310/AS 1310 Autosampler."

For operation with the AI 1310/AS 1310 autosampler refer to the *AI 1310/AS 1310 for FLASH Elemental Analyzers User Guide*.

Ramped GC Oven

The Ramped GC Module for the temperature control of the chromatographic column.

Figure 28 shows the Ramped GC Oven installed on the back of the Flash IRMS EA.

Figure 28. Ramped GC Oven

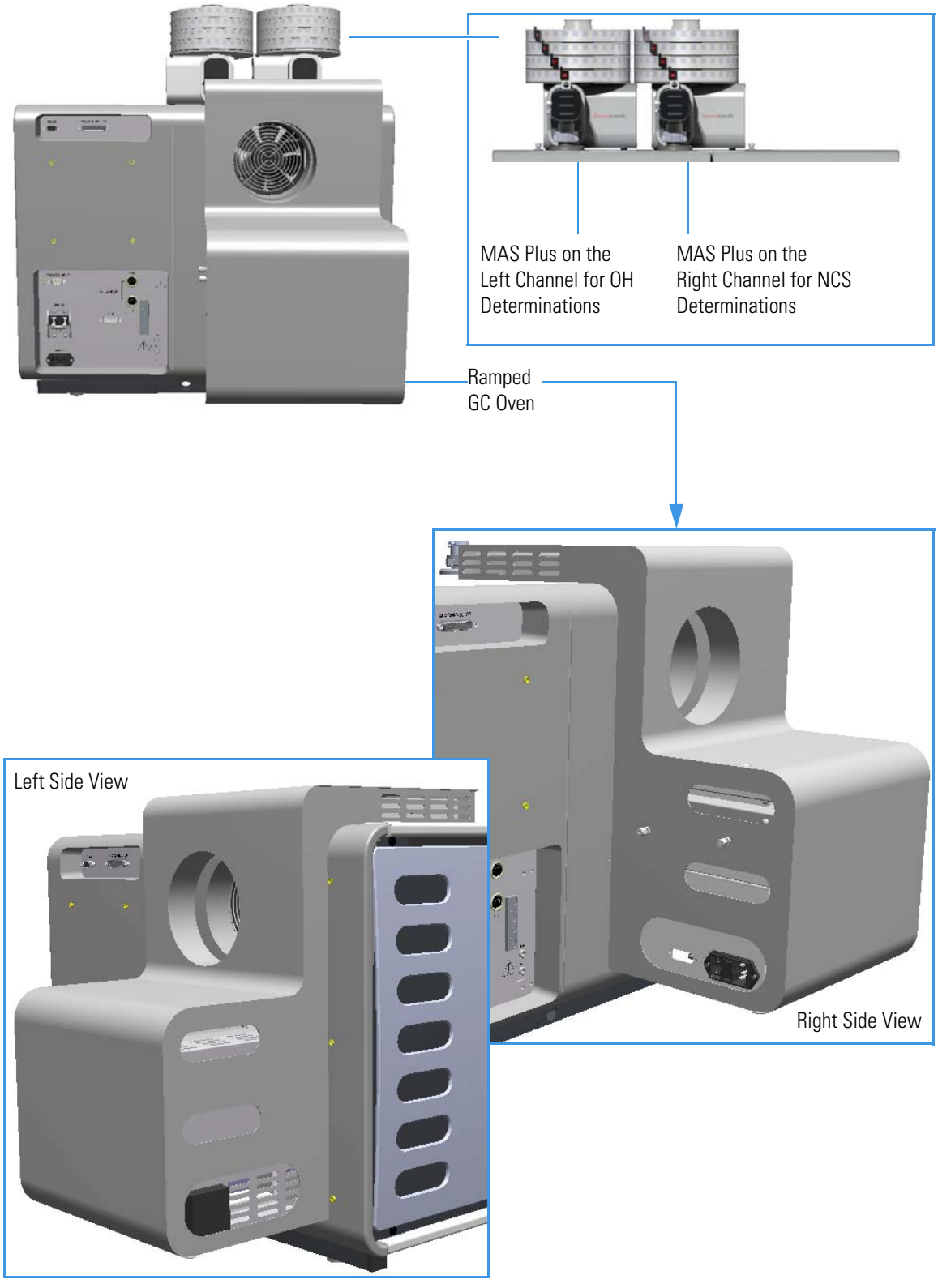
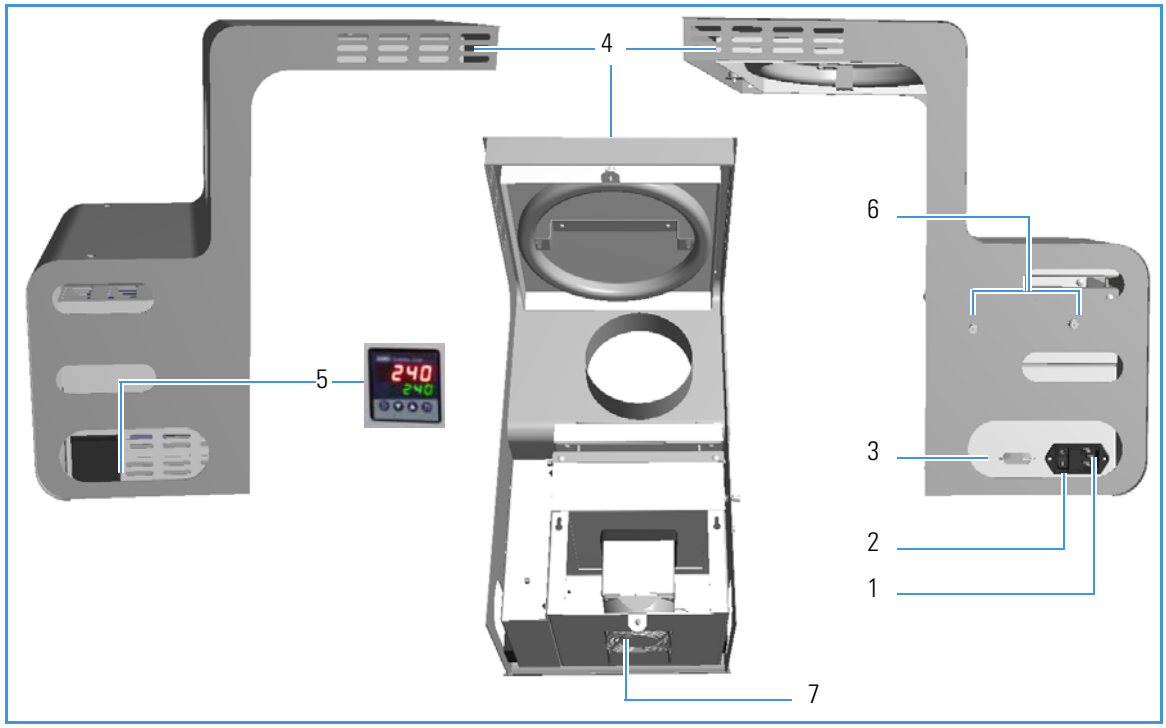


Figure 29 shows the components of the Ramped GC Oven.

Figure 29. Components of the Ramped GC Oven



The Ramped GC Oven includes the following components. See the numbering in the images in Figure 29.

1. AC Input Connector
2. Power Switch to power the Ramped GC Oven On/Off.
   - Position I = instrument On
   - Position O = instrument Off
3. 9-pin connector for the control of the GC.
4. Compartment for the Thermo Scientific™ smartEA™ option.
5. Jumo controller for the manual temperature program setting.

ATTENTION The start and the stop of the temperature program is performed through **Isodat Software Suite** by clicking **GC Start/Stop** function in the method.

6. Gas inlet/outlet ports for the connection of the carrier gas.
7. Cooling fan.

CAUTION For installing, connecting and operating the Ramped GC Oven refer to the *Ramped GC Oven Operating Manual*.

2

Analytical Principles

This chapter describes the analytical techniques with the correlated pneumatic circuits used for the configurations of the EA IsoLink IRMS System for CNSOH.

Contents

About Your System

The Elemental Analyzer is equipped with an automatic switch valve (ASV) between the two modes of **high temperature conversion** (**H** and **O** isotope analysis), and combustion (**N**, **C**, and/or **S** isotope analysis).
The switch also involves the transfer of the electronic autosampler start signal to either side.

The EA IsoLink IRMS System for CNSOH is configured with an optimized reactor and separation column for triple analysis of **N**, **C** and, **S** isotopes, and **Sulfinert**® capillary for minimized water adsorption, and reduced nitrogen background.

The **NCS** separation column is operated in a ramped GC oven attached to the back panel of the Flash IRMS EA. The molecular sieve for high temperature conversion analysis is installed in the isothermal GC oven of the EA.

Two autosamplers on top and full software control automate the analysis of five isotopes with two sample drops.

The additional bottom feed connector for the pyrolysis side extends the sample throughput to more than 400 solid samples without maintenance.

This section describes the new features and software setup of the EA IsoLink IRMS System for CNSOH, and shall be seen in addition to the existing:

- *ConFlo IV Operating Manual Rev. B*

It is recommended to read the above manuals in addition to this guide.

The following analytical setups are possible:

- **H** and **O** isotope analysis in dual or single mode.
  - Using the high temperature conversion technique (also referred to as pyrolysis) with glassy carbon reactor.

    See "High-Temperature Conversion – Analysis of H and O Isotopes" on page 94.

    See Setting Up a HO Method.
- **N**, **C**, and **S** isotope analysis (triple analysis):
  - Using a dedicated single reactor for triple **NCS** analysis, and an optimized separation column and temperature program.

    Please refer to the *Ramped GC Oven Operating Manual.*
- **N** and **C** isotope analysis in dual or single mode.
  - Using a single reactor setup with chemical trapping of $\mathbf{SO_2}$.

    See "Creating an Isodat Method for N+C Measurement (Dual Measurement)" on page 88.
  - Installing an additional $\mathbf{CO_2}$ trap for **N** isotope analysis in single mode reduces the analytical time.
- **S** isotope analysis in single mode.
  - If no separation of $\mathbf{N_2}$ and $\mathbf{CO_2}$ is needed, a dedicated single reactor for triple NCS analysis and an optimized separation column and temperature program can be used. This reduces the analytical time.

    See "Creating an Isodat Method for Single Mode S Measurements" on page 93.

The **EA IsoLink IRMS System for CNSOH** configuration and its features like triple analysis are available from **Isodat Software Suite** version 3.0 on with Service Pack 0.94 or higher.

For recent service packs please contact your local Thermo Fisher Scientific service office.

Pneumatic Circuit

This section describes the pneumatic circuits of the EA IsoLink IRMS System for CNSOH in the combustion NCS configuration and high temperature mode OH configuration. See Figure 30 and Figure 31.

IMPORTANT All the pneumatic diagrams are visualized in the **Pre-analysis** stage.

Figure 30. EA IsoLink IRMS System for CNSOH in Combustion NCS Analysis Pneumatic Diagram

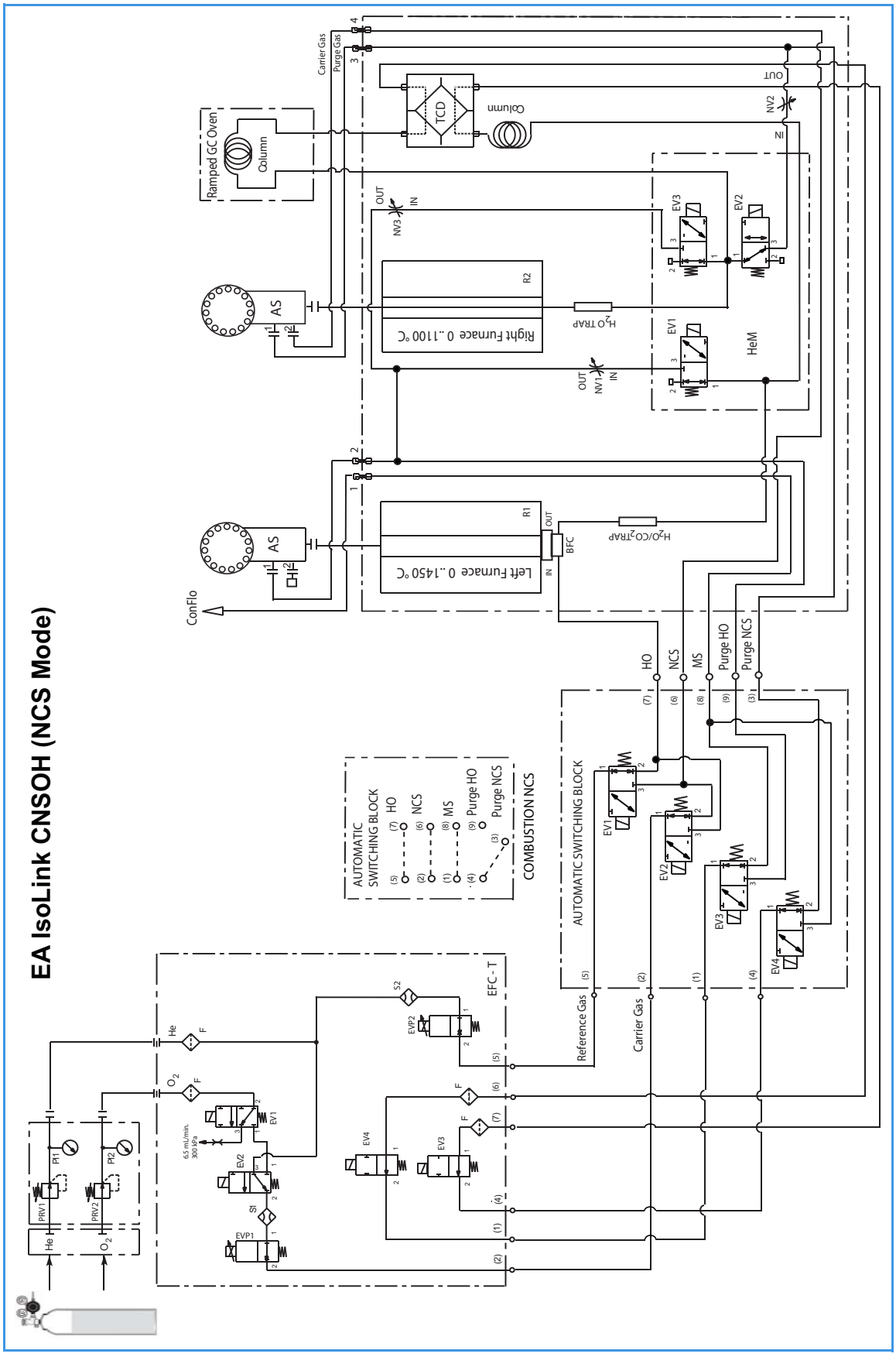
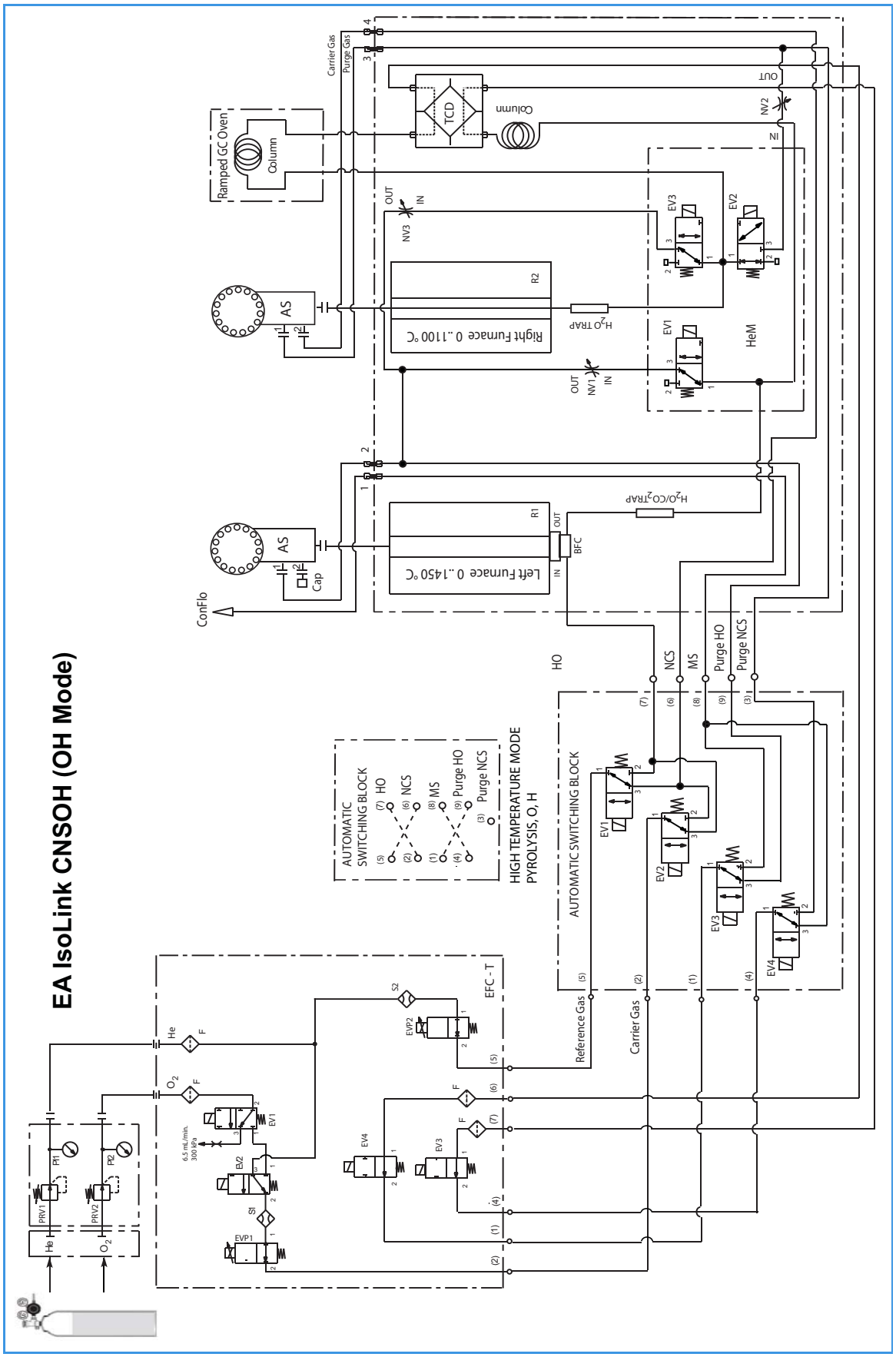
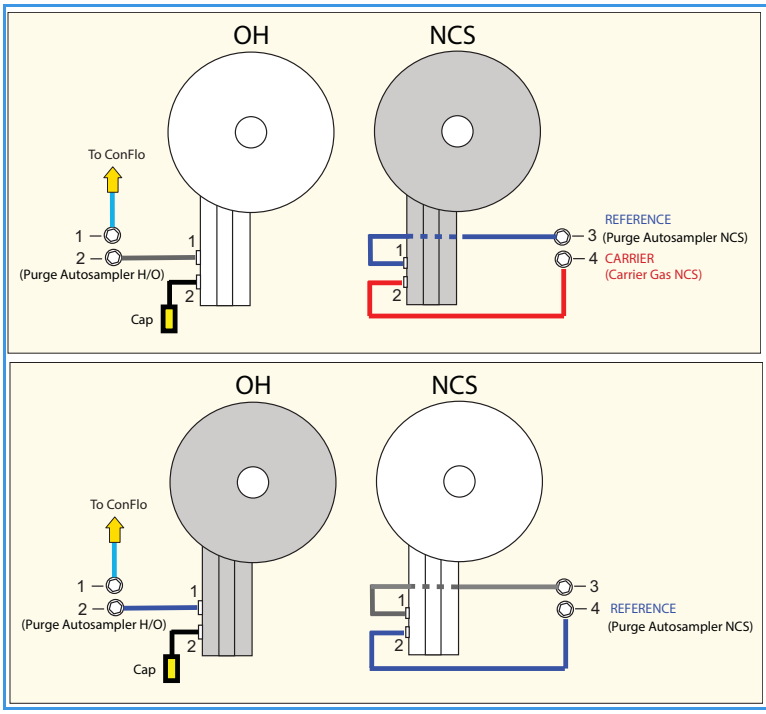


Figure 31. EA IsoLink IRMS System for CNSOH in Pyrolysis OH Analysis Pneumatic Diagram



The external pneumatic connection is shown in Figure 32.

Figure 32. External Pneumatic Connection for NCS and OH Analysis



NCS Configuration uses the **right** channel.

Note: The reference flow enters the pyrolysis reactor from the bottom before it ends as purge in the Autosampler NCS.

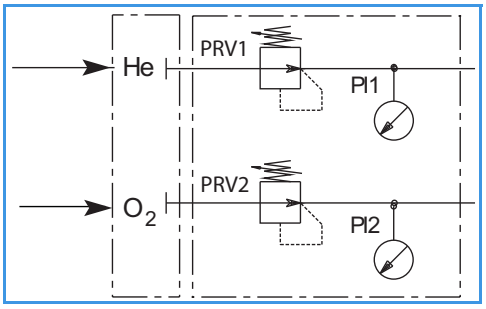
OH Configuration uses the **left** channel.

Note: The carrier gas flow is not shown here. It enters the pyrolysis reactor from the bottom.
The reference flow enters the system at the combustion reactor and ends as purge for the Autosampler H/O.

Pressure Regulators

The pressure regulators allow the manual adjustment of the carrier gas (helium or argon) and oxygen inlet pressures. The regulators are located in the detector compartment and they are schematically shown in Figure 33.

Figure 33. Pressure Regulators



The pressure regulators are common in all the configurations of the EA IsoLink IRMS System for CNSOH. The regulators consist of the following components. See Table 13.

Table 13. Pressure Regulators Pneumatic Diagram (Sheet 1 of 2)

Component	Description and function
He	Helium inlet port
O_2	Oxygen inlet port
PRV1	Helium pressure regulator
PI1	Helium pressure gauge

Table 13. Pressure Regulators Pneumatic Diagram (Sheet 2 of 2)

Component	Description and function
PRV2	Oxygen pressure regulator
PI2	Oxygen pressure gauge

Electronic Flow Controller (EFC-t) Module

The EFC-t module is common in all the configurations of the EA IsoLink IRMS System for CNSOH. The module is schematically shown in Figure 34, and consists of the following components. See Table 14.

Figure 34. Thermo-regulated EFC-t Module Pneumatic Diagram

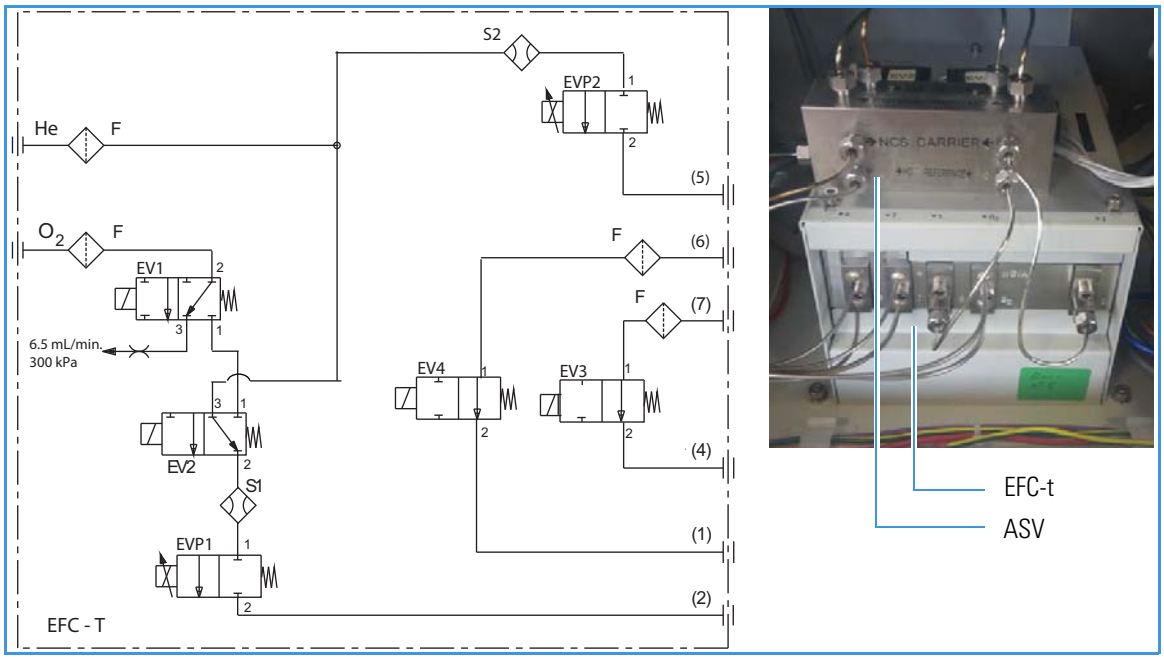


Table 14. Parts of the EFC-t Module (Sheet 1 of 2)

Component	Description and function
He	Helium inlet port
O_2	Oxygen inlet port
F	Stainless steel filter
EV1	Two-way solenoid valve to control oxygen inlet.
EV2	Three-way solenoid valve to control helium inlet and to allow switching between helium and oxygen.
EV3	Two-way solenoid valve, normally open, to control the inlet of helium flowing back from the TCD detector analytical channel. The valve is closed during the leak test.
EV4	Two-way solenoid valve, normally open, to control the inlet of helium flowing back from the TCD detector reference channel. The gas leads to port 1 of the MAS Plus autosampler and then to the ConFlo..
S1	Electronic flow sensor for helium as carrier gas and oxygen during the sampling stage. It cooperates with the EVP1 electronic controller (proportional valve).

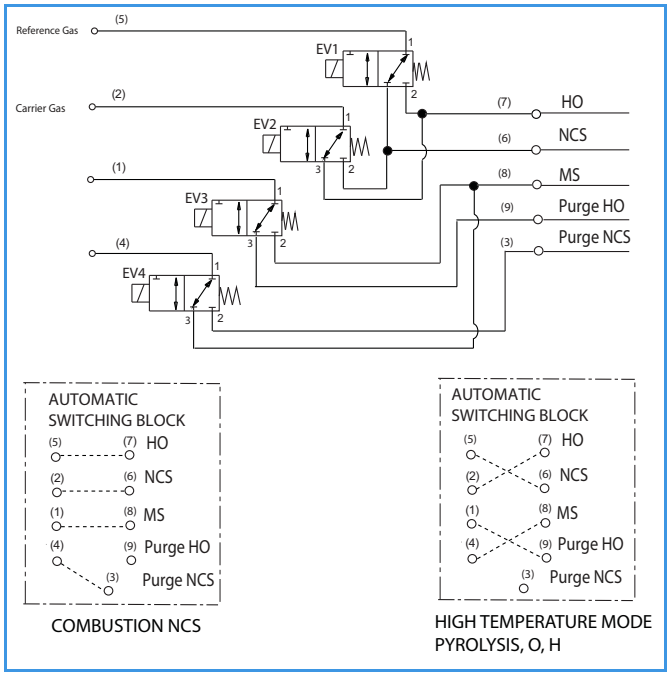
Table 14. Parts of the EFC-t Module (Sheet 2 of 2)

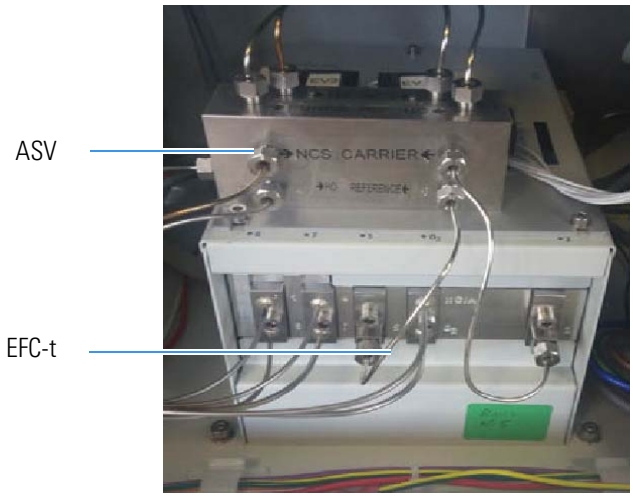
Component	Description and function
S2	Electronic flow sensor for helium as reference gas. It cooperates with the EVP2 electronic controller (proportional valve).
EVP1	Electronic flow controller for helium as carrier gas and oxygen to control the flow rates of gases according to the flow values set.
EVP2	Electronic flow controller for helium as reference gas to control the flow rate according to the required flow value.

Automatic Switching Valve Block (ASV)

Allows the automatic switching from the **combustion** N,C, S configurations to the **pyrolysis** O, H configuration, and vice-versa. The module is schematically shown in Figure 35, and consists of the following components. See Table 15.

Figure 35. Automatic Switching Valve Bock Pneumatic Diagram





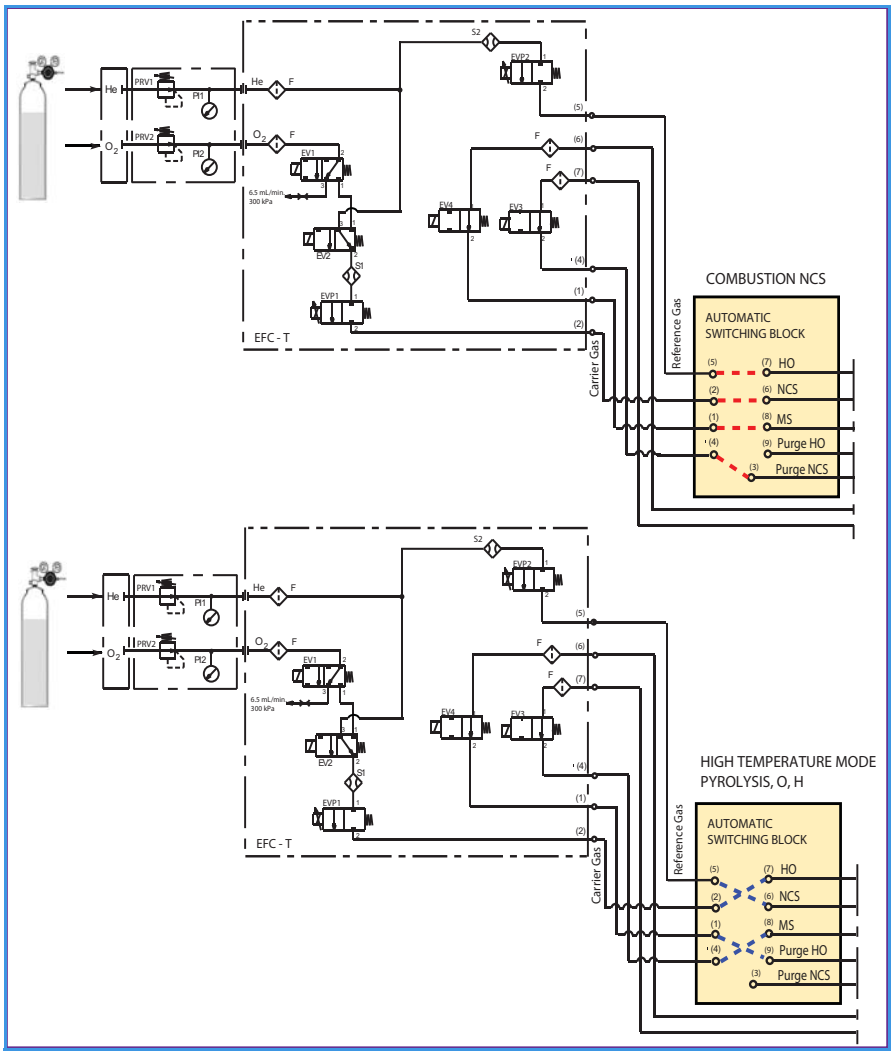


Table 15. Parts of the Automatic Switching Block

Component	Description and function
EV1	In combustion NCS analysis controls the reference gas flow through the right channel. In pyrolysis O,H analysis controls the carrier gas flow through the right channel.
EV2	In combustion NCS analysis controls the carrier gas flow through the right channel. In pyrolysis O,H analysis controls the reference gas flow through the right channel.
EV3	In combustion NCS analysis controls the carrier gas flow to the ConFlow. In pyrolysis O,H analysis controls the reference gas flow to the ConFlow.
EV4	In combustion NCS analysis controls the purge gas flow to the right autosampler. In pyrolysis O,H analysis controls the purge gas flow to the left autosampler.

Helium Management (He^M) Module

This module allows the reduction of helium consumption for each sample. The module is shown in Figure 36, and consists of the following components. See Table 16.

Figure 36. Helium Management Valve Block (Left) and Needle Valve Holder for Split Adjustment (Right)

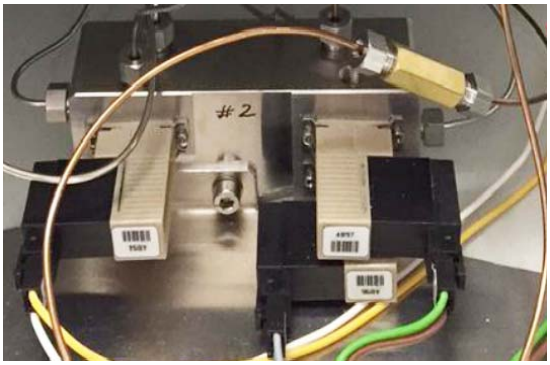


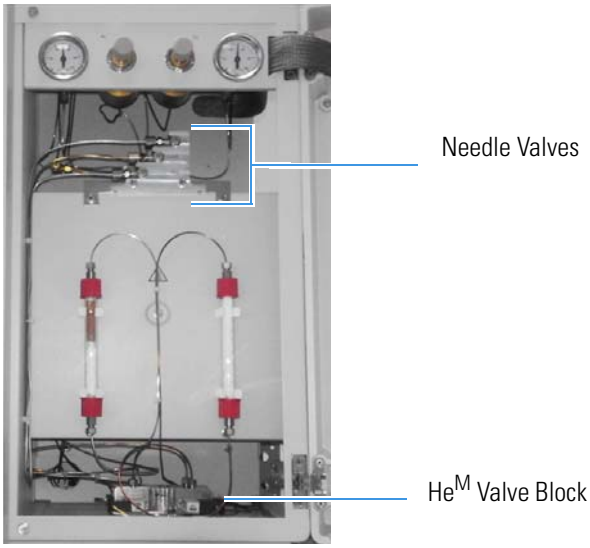
Table 16. Parts of the He^M Module

Component	Description and Function
EV1 - NV1	Pyrolysis — opens and closes the split and controls the split flow
EV2 - NV2	Combustion — opens and closes the split and controls the split flow
EV3 - NV3	Vent — opens and closes the split and controls the split flow for large sample analysis

Helium Management (He^M) Valve Block Description

The Flash IRMS Elemental Analyzer is equipped as standard with the Helium Management (He^M) module. The He^M module reduces the He consumption in an EA significantly, e.g. up to >60% for a CNS triple analysis from a single sample. Large volumes like the reactors require high flows of >80mL/min. Since peak broadening starts in the reactors higher flows are favorable to enhance peak shape. He^M module allows high reactor flows of 180 mL/min. This increase in carrier flow is compensated by recirculating up to 2/3 of the He flow back into the system for purging. He^M module works through a valve block which is installed underneath the isothermal GC oven of the EA). See Figure 37.

Figure 37. HeM Valve Block Installed



The valves are controlled by Isodat Software Suite. The split ratios are set by needle valves which are located above the isothermal GC oven. The needle valves are preset.

Note Any changes may have influence on the system performance and should be done only when necessary and after reading this manual.

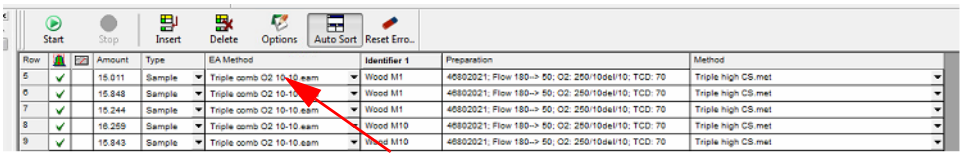
He^M Working Principle

When working with He^M module the analysis is separated into two phases:

1. Analysis mode
2. He save mode

He^M is switched on as default. It can be switched off if no He savings are desired.

- **Combustion** — Before an analysis Isodat uploads an EA method to the EA. This method includes the settings of carrier and reference flow (=purge flow). It is thus obligatory that an EA method is set in the sequence of Isodat to ensure proper functionality of the He^M module. With the upload of the EA method the flows are set.



During initialization of the system (magnet settings, peak center, reference gas flow equilibration in the ConFlo) the reactor flow (carrier gas) is stabilized.

When the sample drops (or is injected by liquids autosampler) the carrier gas flow is maintained for a fixed period until all analyte gases produced upon conversion of the sample in the reactor are certainly on the chromatographic column (analysis mode). Split valve 2 (EV2) is open at that time.

In general, the flow through the chromatographic column is 50 mL/min if the carrier flow is set to 180 ml/min. The split flow is thus 130 mL/min and merges into the reference gas (purge) line to contribute to a sufficient purge flow of the sample in the autosampler (100 – 200 mL/min). The minimum flow for the reference should be 10 mL/min.

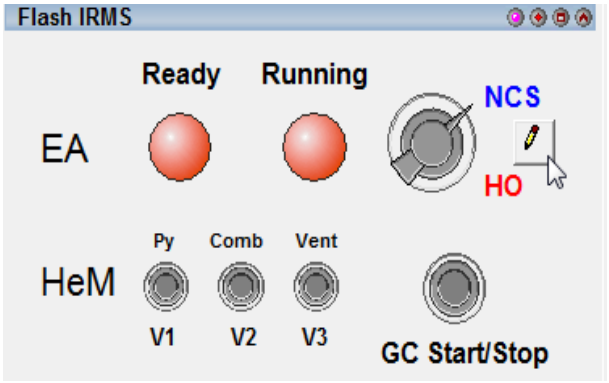
As soon as all analyte gases are on the chromatographic column EV2 is closed and the carrier gas flow is automatically set to 50 mL/min (He save mode). The reference flow is kept at its set value (10 - 70 mL/min) but the contribution from the split is stopped. This status is maintained until the next analysis is started.

- **Pyrolysis** — The carrier gas flow through the pyrolysis reactor is lower than in the combustion mode. It is commonly set to 100 mL/min. The flow through the separation column is the same as in combustion mode: 50 ml/min if the carrier gas flow is 100 mL/min. Unlike in combustion mode the carrier flow is not reduced during the analysis. V1 is open until acquisition is completed. He saving is achieved by using the split flow for purge just as in combustion mode.

  To avoid high pressure on the chromatographic column in the combustion system the reference flow (50 – 150 mL/min) is split using EV3 and reunited after the chromatographic column. Thus V3 is also open all the time.

Setting Different Flows and Switching Times

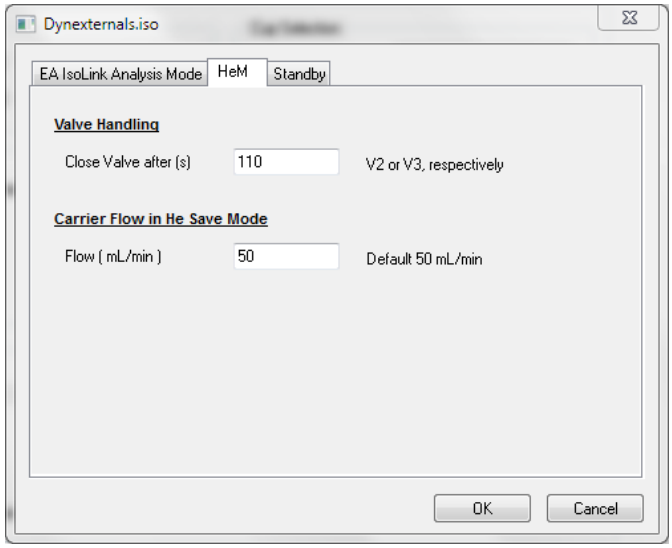
It is possible to change the default settings for switching the He Save Mode. Click on the button and choose the tab "HeM".



Here you can define when the He Save Mode is switched on and how high the carrier flow shall be during the He Save Mode.

Note Changing the values will have influence on your analytical results. All instructions in this manual are based on the default settings (100s, 50 mL/min).

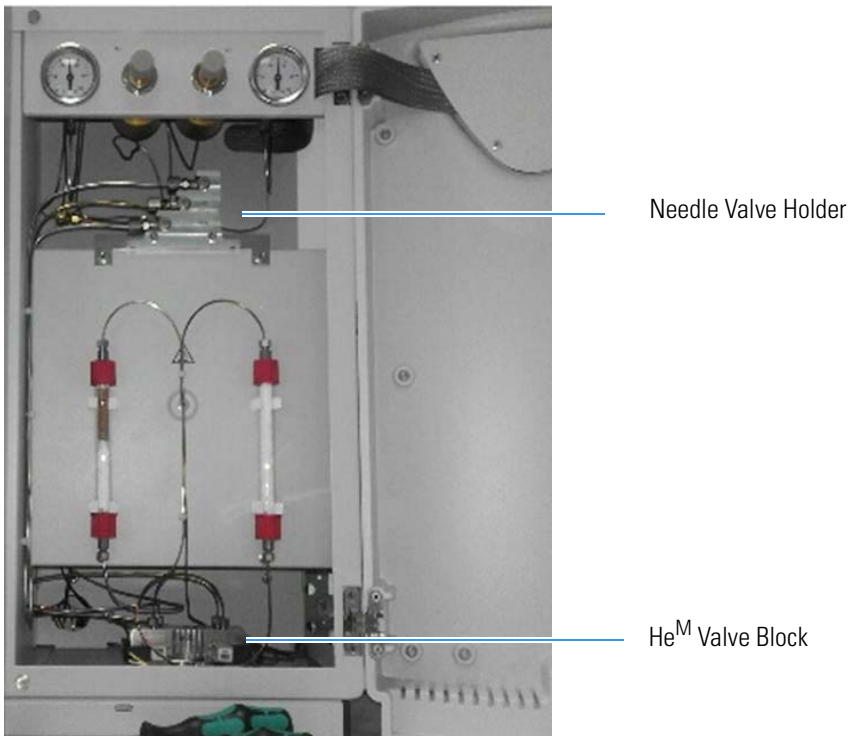
Note The flow during He Save Mode must be identical to the split flow through the GC column during Analysis Mode. This is mandatory to maintain a constant TCD signal.



Adjusting the Split Flow

The split ratio is managed by the valve block and an adjustable restriction, the needle valves on top of the GC oven in the EA. See Figure 38.

Figure 38. Needle Valves



In general, there is no need to adjust the splits. It may become necessary if the restrictions after the valve block (i.e. water trap, GC column) change or if the flow conditions (i.e. carrier gas flows) are modified. This chapter is about the procedure to change the settings.

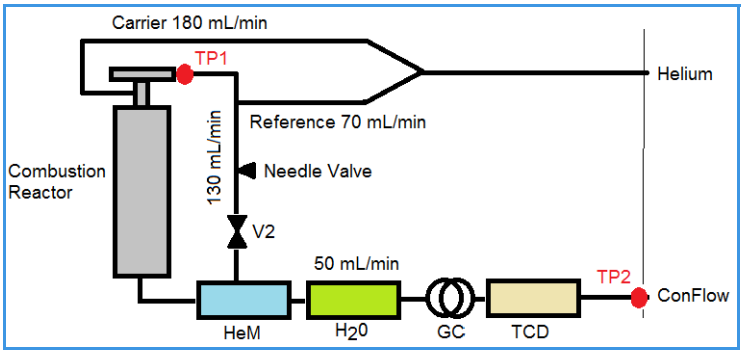
Note Any changes may have influence on the system performance and should be done only when necessary and after reading this manual.

Pneumatic Circuit and Measuring Points

Figure 39 is a simplified pneumatic circuit of the Flash IRMS. It shows two measuring points "**TP**". Here you must disconnect the lines and connect to a flow meter. The split ratio of combustion side should be 130/50 mL/min where 130 mL/min are fed back into the purge line and 50 mL/min go through the GC column.

Note The flow through the GC column should not exceed 60 mL/min in analytical conditions.

Figure 39. Simplified Pneumatic Circuit



1. Make sure you are in combustion mode (arrow points to **NCS**).
2. Make sure **V2 Comb** is on.

3. Set carrier flow to 180 mL/min and the reference flow to 70 mL/min.
4. Disconnect the purge line of your autosampler (see measuring point TP1 in Figure 39) and measure the flow. It should read 200 mL/min (±2 mL/min).
   - If the flow is higher, use a screwdriver to gently close needle valve **V2** turning it counter-clockwise.
   - If the flow is lower, use a screwdriver to open the needle valve turning it clockwise.
5. Now remove the line to the ConFlo (or SmartEA, measuring point TP2 in Figure 39) and measure at the exit of the EA. It should read 50 mL/min (±2 mL/min).

CAUTION The needle valves are very sensitive. It is best if the fixing nut is already fixed before you use the screwdriver. This allows a finer adjustment.

Preparation of Reactors and Adsorption Filter

NC Configuration

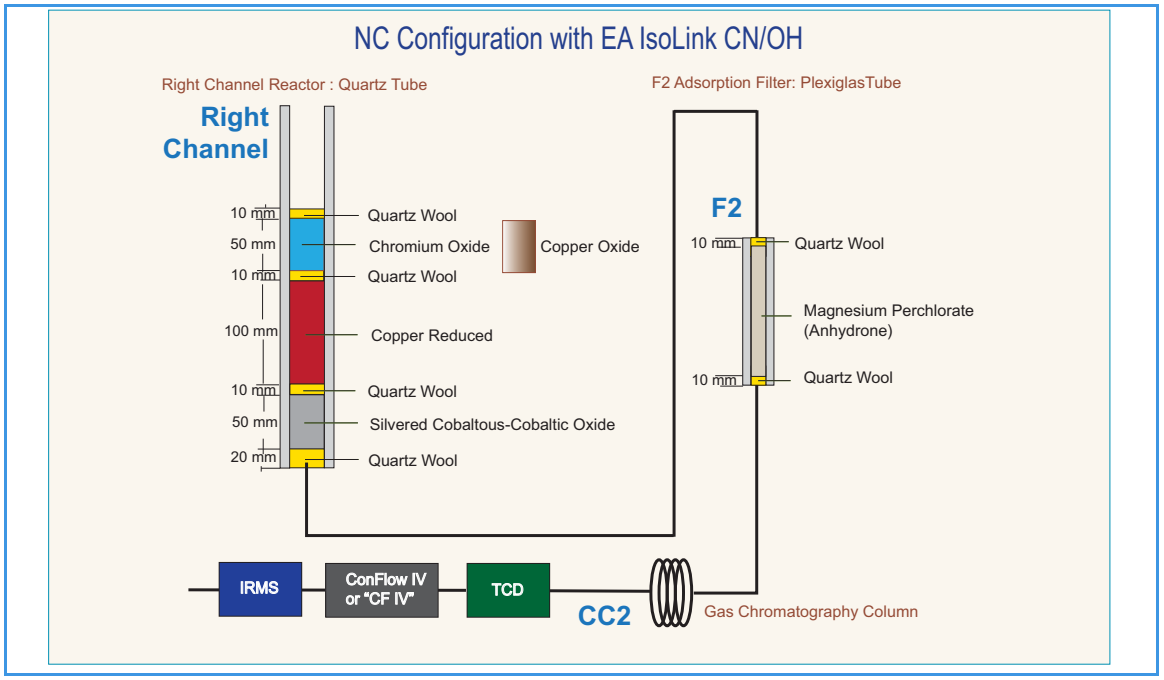
Table 17 report the characteristics of the components required for NC determination, and the type and size of the filling materials to be used for a proper preparation of the reactor.

Table 17. Components required for NC Determination

Reference	Component	Characteristic	Filling Materials
Right Channel	Reactor	*Material:* Quartz	Quartz Wool Chromium Oxide Reduced Copper Silvered Cobaltous-Cobaltic Oxide
F2	Absorption Filter	*Material:* Plexiglas	Quartz Wool Magnesium Perchlorate (Anhydrone)
CC2	Gas chromatographic Column	*Material:* Stainless Steel	---

Figure 40 shows the size of the filling material.

Figure 40. Size of Filling Material



Note In some cases, it can be advantageous to use **copper oxide** instead of **chromium oxide**. Reactors filled with copper oxide must be operated at 900°C - 920°C. Check your calibration with appropriate standards to maintain analytical precision and quality after changing the reactor filling from chromium oxide to copper oxide.

- **Reactors** — The reactor is a quartz tube having a conical bottom end. There are two different sizes available:
  - quartz glass reactor of 18 mm OD

– quartz glass reactor of 25 mm OD (Macro Reactor)

Note The EA IsoLink IRMS System for CN/OH comes standardized for a 18 mm OD quartz glass reactor. For 25 mm reactor tubes the MAS Plus and the top of the EA must be modified. See the section "Upgrading EA for 25 mm OD Macro Reactor" on page 131.

- **Filter** — It is a Plexiglas filter. The filling materials used are according to the NC analytical determination.

❖ To set the temperature

The following procedure is recommended for heating up the furnace of the combustion reactor when operating with chromium oxide (Cr_2O_3) at 1020°C:

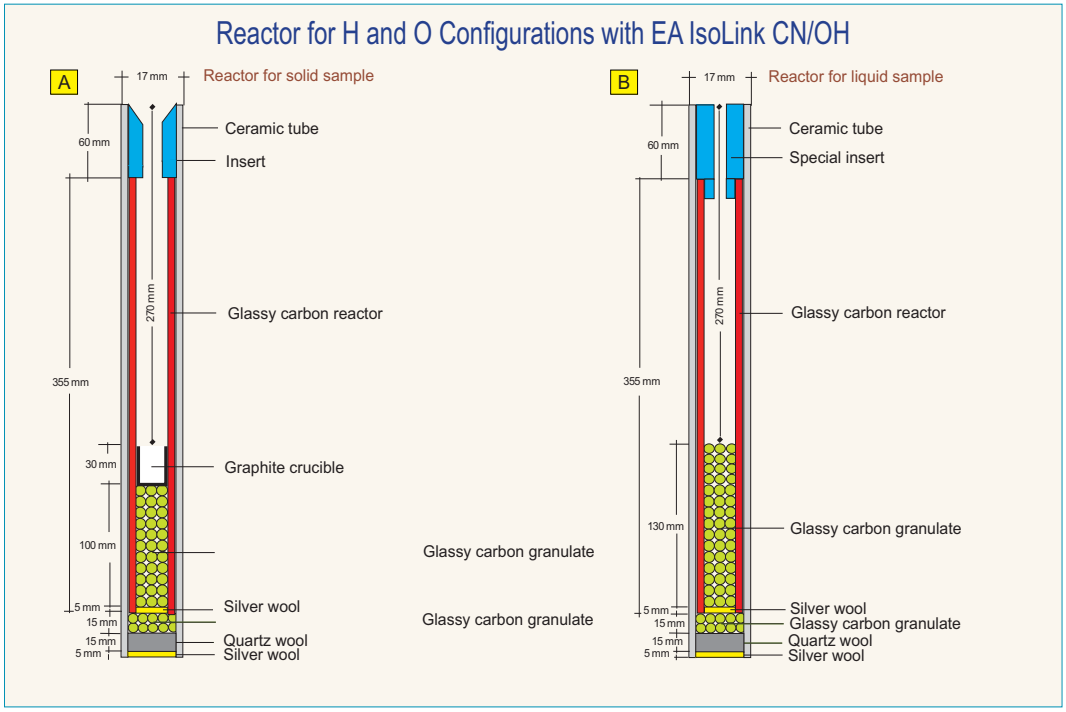
1. Increase the temperature from room temperature to 400°C and check the background signals on the Mass Spectrometer. Check the system for leaks.
2. Increase the temperature from room 400°C to 900°C in steps of 100°C and hold at 900°C for at least 15 min. This avoids melting the copper within the reactor, which will cause poor performance.
3. Increase it to the operating temperature of 1020 °C.

WARNING Immediate temperature increase to 1020 °C can cause copper melting. Cooling down in steps can avoid reactor breaking. Alternatively, you can use the HeatUp and CoolDown script in Isodat. Right-click on the Flash IRMS visualization window in Isodat and choose the appropriate context menu item. See page 82.

H and O Configurations

These configurations require the use of a special ceramic reactor, as shown in Figure 41, and the use of a trap filled with **Carbosorb** (**Ascarite**® P/N 33835236) and **Magnesium Perchlorate** (P/N 338 21900). after the reactor and before the HeM valve block. This additional trap is not included in delivery.

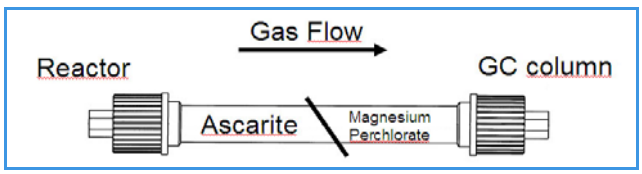
Figure 41. High Temperature Conversion Reactor for H and O Analyses of Solid (A) and Liquid (B) samples



IMPORTANT The filling of the reactor must be performed rigorously respecting the exact proportions of the packing materials. The distance of 270 mm from the head of the ceramic tube to the graphite crucible is of extreme importance, because the crucible must be placed in the hottest zone of the furnace. Additionally avoid that any organic material enters the reactor.

The filling of the trap is given in Figure 42 using quartz wool at top and end and to separate the filling material.

Figure 42. CO_2/H_2O Trap Between High Temperature Conversion Reactor and He^M Valve Block



Preparing the Reactors

According to the instrument configurations, the filling materials are introduced into the reactor in a way to form a series of layers of defined dimensions. For a proper preparation of the filling layers, refer to the filling diagram of the concerned instrument configuration, as described in the section "Preparation of Reactors and Adsorption Filter" on page 44. When using macro reactors (25 mm D) then use the same packing instruction.

Note The conical end of the quartz glass tube must be at the bottom.

WARNING Before using the filling materials required for this operation, please read the hazard warnings and information reported in the Material Safety Data Sheets (MSDS) provided, referring to the relevant CAS (Chemical Abstract Service) number.

The filling of reactors requires the use of quartz wool. Before handling quartz wool, we recommend to wear gloves and face protection.

Always use original Thermo Fisher Scientific materials and products. The use of materials not meeting the technical specifications of our products does not ensure a good operation of the instrument and may even damage it.

The filling procedure should be carried out on a wide and clean workbench. See the following procedures:

- "To fill the quartz reactor" on page 47
- "To fill the adsorption filter" on page 49

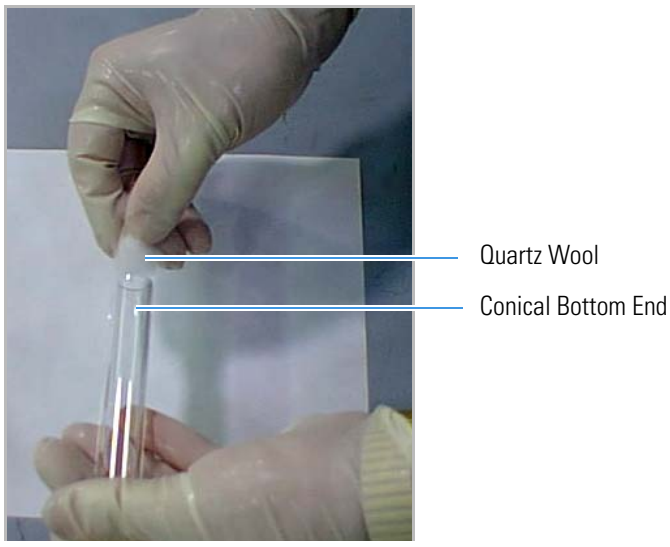
❖ To fill the quartz reactor

The following procedure provides instructions for filling a quartz reactor.

Material Required
Quartz reactor
Compression rod
Filling material

1. Starting from the reactor conical bottom end, introduce a sufficient amount of quartz wool to form the required layer, as shown in Figure 43.

   Figure 43. Introduction of Quartz Wool into the Conical End of the Reactor

2. Plug with your finger the mouth of the reactor conical end. Gently press the quartz wool using the rod provided, as shown in Figure 44.

Figure 44. Compression of Quartz Wool into the Quartz Reactor

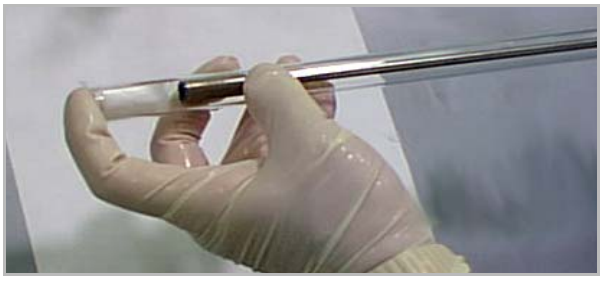
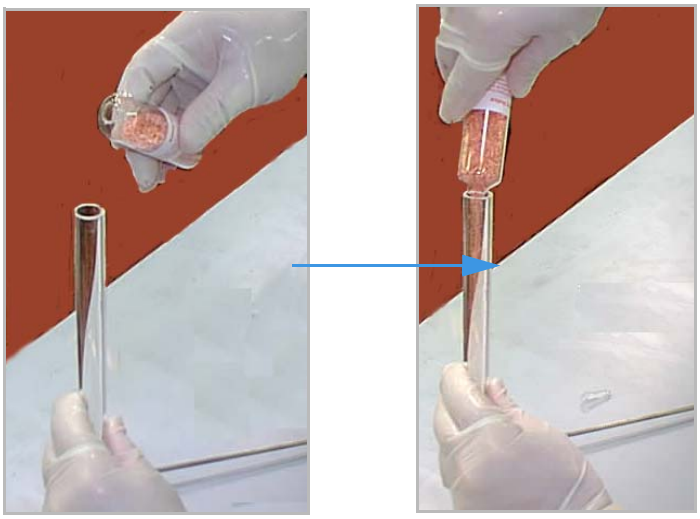
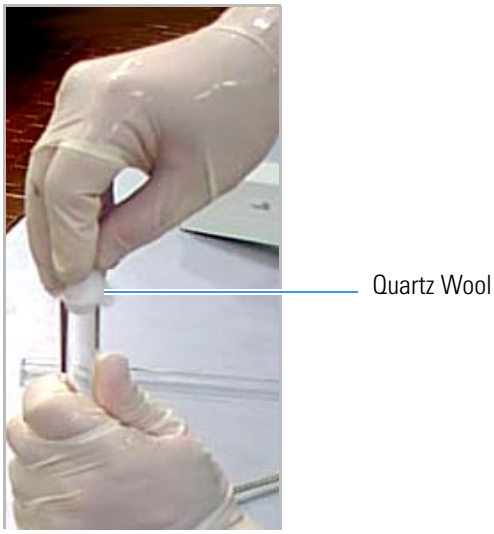


Figure 45. Filling of the Quartz Reactor



3. Turn the reactor conical end downward and rest it delicately onto the workbench.
4. Pour sequentially the required filling materials into the reactor, as shown in Figure 45, ensuring that each layer has the indicated size. At each step gently press the quartz wool using the rod provided.
5. The last step of the sequence consists in introducing a sufficient quantity of quartz wool to form the last required layer, as shown in Figure 46.

Figure 46. Introduction of Quartz Wool as Last Layer of the Sequence



6. Delicately press the quartz wool using the rod provided.

Preparing the Adsorption Filter

The filling materials are introduced into the empty filter to form a series of layers of defined dimensions. For a proper preparation of the layers, refer to the filling diagram of the concerned instrument configuration, as described in the section "Preparation of Reactors and Adsorption Filter" on page 44.

WARNING Before using the filling materials required for this operation, please read the hazard warnings and information reported in the Material Safety Data Sheets (MSDS) provided, referring to the relevant CAS (Chemical Abstract Service) number.

The filling of reactors requires the use of quartz wool. Before handling quartz wool, we recommend to wear gloves and face protection.

Always use original Thermo Fisher Scientific materials and products. The use of materials not meeting the technical specifications of our products does not ensure a good operation of the instrument and may even damage it.

The filling procedure should be carried out on a wide and clean workbench. See "To fill the adsorption filter" on page 49.

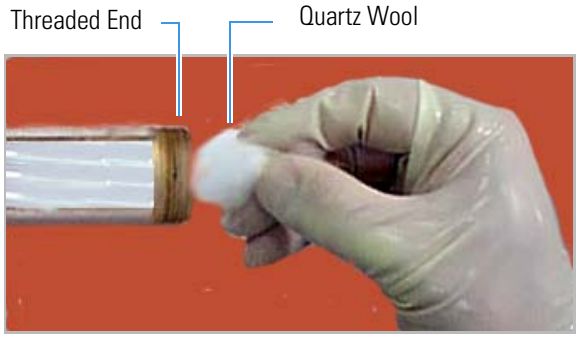
❖ To fill the adsorption filter

The following procedure provides instructions for filling an adsorption filter.

Material required
Plexiglas filter
Compression rod
Filling materials

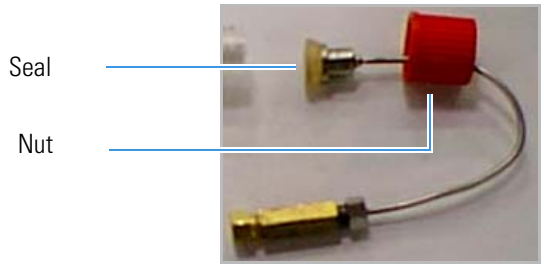
1. Introduce into either of the tube ends a sufficient amount of quartz wool to form the required layer as shown in Figure 47.

Figure 47. Introduction of Quartz Wool into the Tube



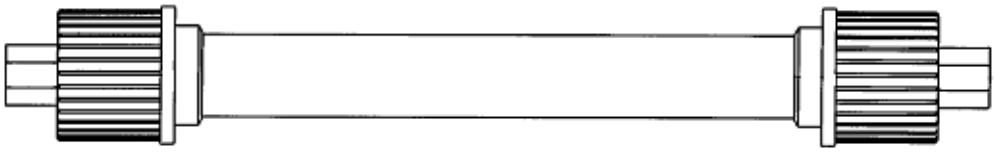
2. While plugging the tube mouth with your hand, press gently the quartz wool using the rod provided.
3. Screw the nut complete with its seal onto the threaded mouth, as shown in Figure 48.

Figure 48. Nuts and Seals for Adsorption Filter



4. Pour sequentially the required filling materials into the adsorption filter, ensuring that each layer has the indicated size. At each step gently press the quartz wool using the rod provided.
5. Do the last layer using a sufficient quantity of quartz wool to form the required layer.
6. Complete the procedure by screwing on the second nut complete with its seal, as shown in Figure 49.

Figure 49. Preparation of the Adsorption Filter



The Ramped GC Oven

The Ramped GC Oven is controlled by a JUMO controller. The controller is pre-programmed and locked. It allows to switch between two setpoints, **SP1** and **SP2**.

- **SP1** is the start temperature of 70 °C
- **SP2** is the end temperature of 240 °C

When receiving the GC start signal (GC On) from Isodat, the Ramped GC Oven increases automatically the temperature from SP1 to SP2. When stopping the GC (GC Off) the oven cools down to SP1. The starting temperature of 70 °C is defined to separate the N_2 and CO_2 peaks. After the CO_2 peak a rapid increase of the temperature accelerates the elution of the SO_2. Detailed information can be found in the *Ramped GC Oven Operating Manual*.

3

Installing Flash IRMS Elemental Analyzer

This chapter provides the instruction for installing the Flash IRMS.

Contents

Preliminary Information

This chapter contains the preliminary information for installing the EA IsoLink IRMS System for CNSOH, and for the electrical requirements.

Who Performs the Installation

Your EA IsoLink IRMS System for CNSOH will be installed by an authorized Thermo Fisher Scientific engineer (FSE), who will verify the instrument operation. If, for any reason, your system is not installed by a Thermo Fisher Scientific FSE, you should ensure that the following operations are performed.

Standard Outfit

Use the standard outfit checklist accompanying the instrument to verify that all items have been received.

Verify Site Preparation

Before installing the EA IsoLink IRMS System for CNSOH, your laboratory must be in compliance with the guidelines and requirements described in the *EA IsoLink IRMS System for CNSOH Preinstallation Requirements Guide*.

Unpacking the Instrument

ATTENTION This operation must be carried out by a Thermo Scientific Field Service Engineer.

After inspecting the exterior of the shipping container for damage carefully unpack the instrument and do the following:

1. Check the contents of each box against the packing list to verify the shipment is complete.
2. Inspect each item for damage.
   a. If equipment is damaged, keep boxes and their equipment in their existing condition and immediately notify the carrier.
   b. Submit a damage claim directly to the carrier, and send a copy (including any shortage claim) to your authorized Thermo Fisher Scientific sales representative.
   c. Do not return any equipment to the dealer or the factory without prior Thermo Fisher Scientific authorization.

Placing the Instrument

Place the EA IsoLink IRMS System for CNSOH on the workbench, allowing free access to electrical connections and gas lines.

LIFTING HAZARD The Flash IRMS Elemental Analyzer weighs approximately 65 kg (145 lb) when unpacked. Pay attention when lifting the instrument onto the workbench. At least TWO people should perform this operation, each standing on left/right side of the instrument and putting their hands near its supporting feet.

You should already have prepared your laboratory according to the space requirements specified in the *Preinstallation Requirements Guide*. The gas and power supplies should have been made accessible. Optional equipment should be placed near the analyzer to be easily connected.

Making the Gas Supply Plumbing Connections

This section provides instructions for making the gas supply plumbing connections.

Building the Gas Lines

Building the gas supply lines from the supply cylinders to the elemental analyzer includes connecting the gas lines to the supply tanks and installing any traps or filters on the line.

To properly connect the gas lines to the gas tanks, you will need the following materials:

- 1/8-in. in diameter (gas lines longer than 3 m [10 ft]
- 1/8-in. stainless steel tubing, properly cleaned
- a 1/16-in. stainless steel tubing, properly cleaned (for the last couple of meters to the instrument)
- a tubing cutter
- connecting nuts and relevant ferrules, reducing pieces 1/8-in. to 1/16-in.
- two wrenches

WARNING Secure gas cylinders to an immovable structure or wall. Handle all gases according to local safety regulations.

Use the following procedure to connect regulators and tubing to the gas supply tanks:

❖ **To regulate and connect tubing**

1. Make sure the initial supply valves are turned off.
2. Connect the regulator to the gas supply tank. Use an open-ended wrench or adjustable wrench to tighten the regulator connection.
3. Determine the length of tubing you need. Use only enough tubing to connect the instrument to the gas cylinders, but allow enough slack in case the instrument should be moved at least 40 cm (16 in.) from other equipment. This allows enough room to perform system maintenance.
4. Use a tubing cutter to cut the tubing.

Purging Gas Lines

We recommend to purge the lines any time you make a cut in the tubing during the gas line assembly process. This will clear them of any debris from the cut. You should also purge the completely assembled gas lines before you connect the gas supply to the EA IsoLink IRMS System for CNSOH. Use the following procedure to purge the gas lines:

❖ **To purge the gas lines**

1. Turn the gas supply on, and set the pressure to 35 kPa (0.35 bar).
2. Allow the line to purge for 10 minutes.
3. Turn off the gas supply.

Connecting the Gas Lines

The gas supply lines must be connected to the instrument back panel using the proper inlets and fittings.

CAUTION The maximum pressures of the gases to supply the Flash IRMS Elemental Analyzer is 700 kPa (7 bar).

❖ **To connect gas lines**

1. Connect the Helium gas line to the inlet marked **He** on the instrument rear panel.
   Gas inlet pressure must be set **400-500 kPa** (4-5 bar, 58-73 psig).
2. Connect the Oxygen gas line to the inlet marked $\mathbf{O_2}$ on the instrument rear panel.
   Gas inlet pressure must be set **400-500 kPa** (4-5 bar, 58-73 psig) according to the instrument configuration.
3. By using the pressure regulators and the pressure gauges located in the detector compartment of the instrument, set the pressure of the gases as follows:
   - **250-300 kPa** (2.5-3 bar) for **helium** (**He**)
   - **250-300 kPa** (2.5-3 bar) for **oxygen** ($\mathbf{O_2}$)

Electrical Connections

This paragraph explains the electrical connections of the EA IsoLink IRMS System for CNSOH, and helps you install and configure peripheral devices and Eager*Smart* Data Handling Software.

CAUTION The power line and the connections among the instruments must maintain good electrical grounding. Poor grounding represents a danger for the operator and may seriously affect the instrument performance.

Do not connect the EA IsoLink IRMS System for CNSOH to lines feeding devices of a heavy duty nature, such as motors, UV lamps, refrigerators and other devices that can generate disturbances. If other instruments, such as computer, balance, printer, ans so forth, have to be connected to the same electrical line as the EA IsoLink IRMS System for CNSOH, ensure that such electrical line is capable of withstanding such electrical consumptions by calculating the total absorption.

Mounting Peripheral Devices

Unpack them and follow instructions included with them. Follow the instructions in the paragraphs below to connect your peripheral devices to the EA IsoLink IRMS System for CNSOH.

Power Connections

WARNING This instrument is electrically powered, and therefore all electrical connections must be provided with good grounding.
Poor grounding can represent a danger to the operator and adversely affect the instrument efficiency.

Performing Electrical Connections

❖ To connect autosampler cable

1. Connect the signal cable of the MAS Plus autosampler to the connector marked **Autosampler**, on the back panel of the Elemental Analyzer.

❖ To connect RS232 cable

Perform this operation if the RS 232 serial line connection is required.

2. Connect the RS 232 cable supplied in the standard outfit between the **COM1** or **COM2** ports of your computer and the 9-pin connector marked **RS 232** on the instrument connection panel.
If your computer is equipped with USB ports, a Serial-to-USB adapter is required for properly connecting the cable.
3. Plug in the instrument and computer power cables.

Installing the Reactors into the Furnaces

Note In the following figures, the connections of the tubings are not visible for convenience.

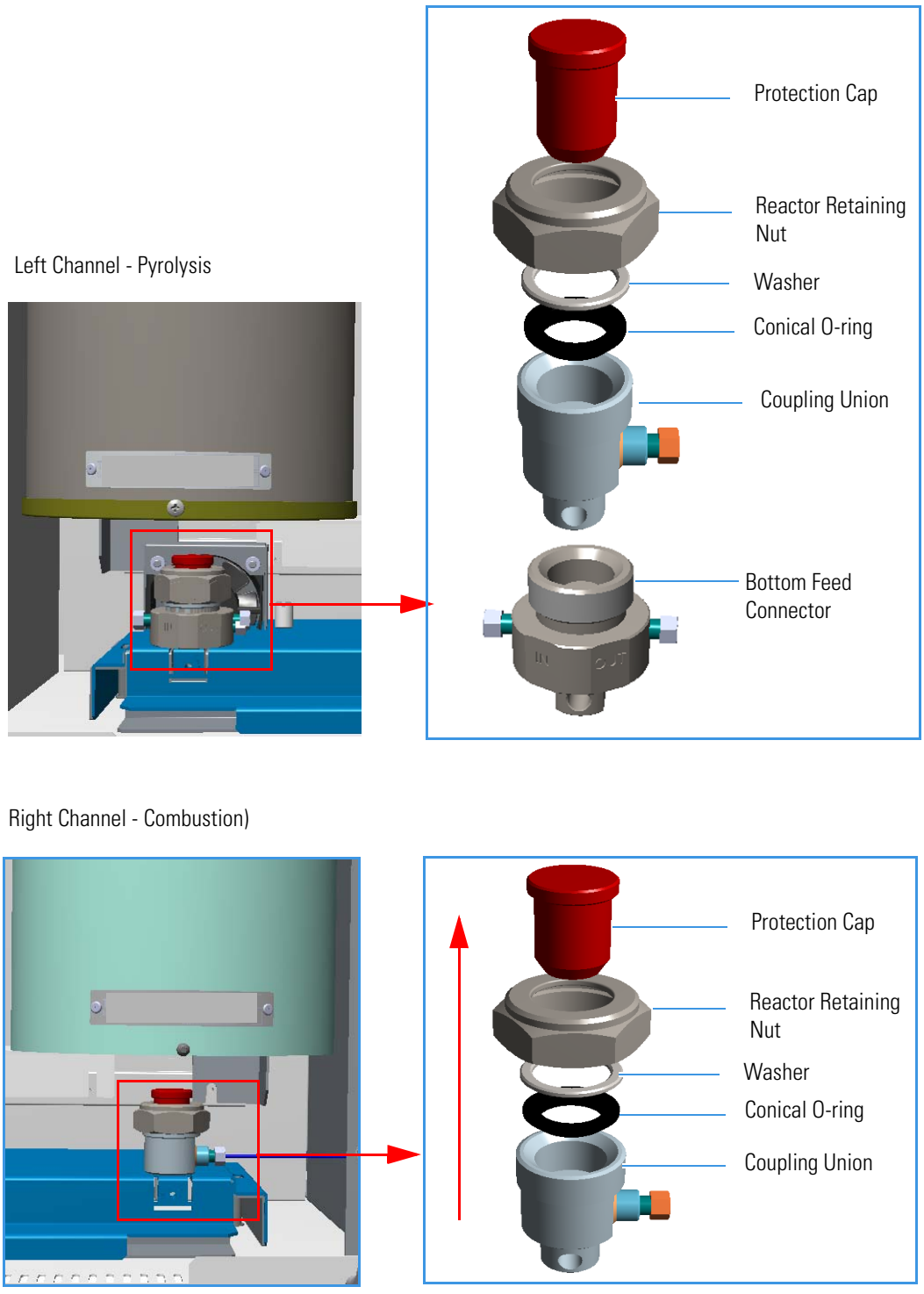
Before installing the reactors for pyrolysis and combustion the following preliminary operation must be carried out.

1. Make sure that the furnaces are at room temperature.
2. Open the furnaces compartment by lifting the cover and removing the protection plate. See Figure 50.

Figure 50. Access to the Furnace Compartment

3. If present, remove the protection cap from the coupling union of each channel.
   a. By using a 32-mm wrench, unscrew the reactor retaining nut. Remove the protection cap, the washer, and the conical O-ring as shown in Figure 51.

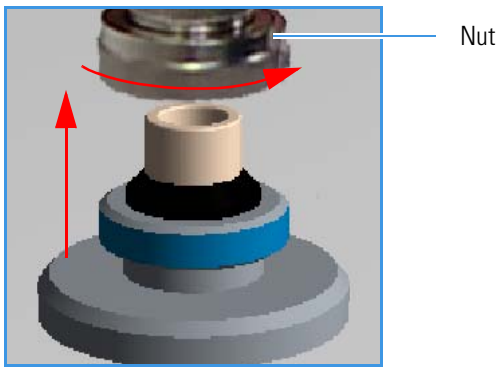
Figure 51. Protection Cap Removal



ATTENTION Do not dispose of the protection cap but keep it in your lab for future use. The protection cap should be reinstalled on the coupling union in case the instrument is not used **for a long time**. The washer and the conical O-ring removed are used for the installation of the reactor.

4. Remove the MAS Plus autosampler, if installed, by manually undoing its fixing nut. See Figure 52.

Figure 52. Removing MAS Plus Autosampler



5. Install the reactors following the instruction reported in the To insert the reactor with a bottom Feed Connector (BFC) and To Install the combustion reactor into the right furnace operating sequences.

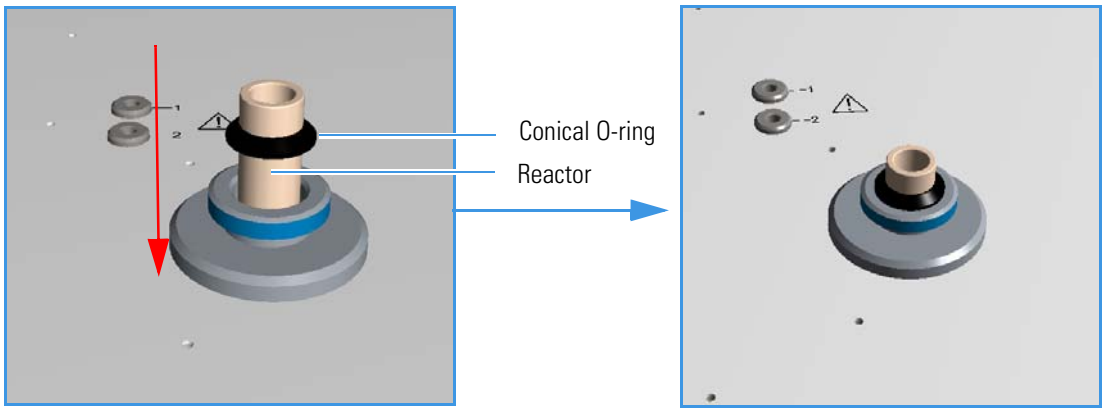
CAUTION The reactors must be installed with the furnace at room temperature.

❖ **To insert the reactor with a bottom Feed Connector (BFC)**

Material required
Reactor retaining nut
Washer
Conical O-ring
Coupling union fixing tool
32-mm wrench

1. Loosely screw the nut with washer and o-ring on the BFC. Insert the ceramic tube from top. Fix it in this position using the upper o-ring. See Figure 53.

Figure 53. Introduction of the Reactor with a Bottom Feed Connector (1)



2. Insert the bottle brush in the glassy carbon tube and introduce it in the ceramic tube from top. Use your other hand to carefully slip it over the o-rings of the BFC while keeping the ceramic tube up. Hold it tight while removing the bottle brush. See Figure and Figure 55.

Figure 54. Introduction of the Reactor with a Bottom Feed Connector (2)

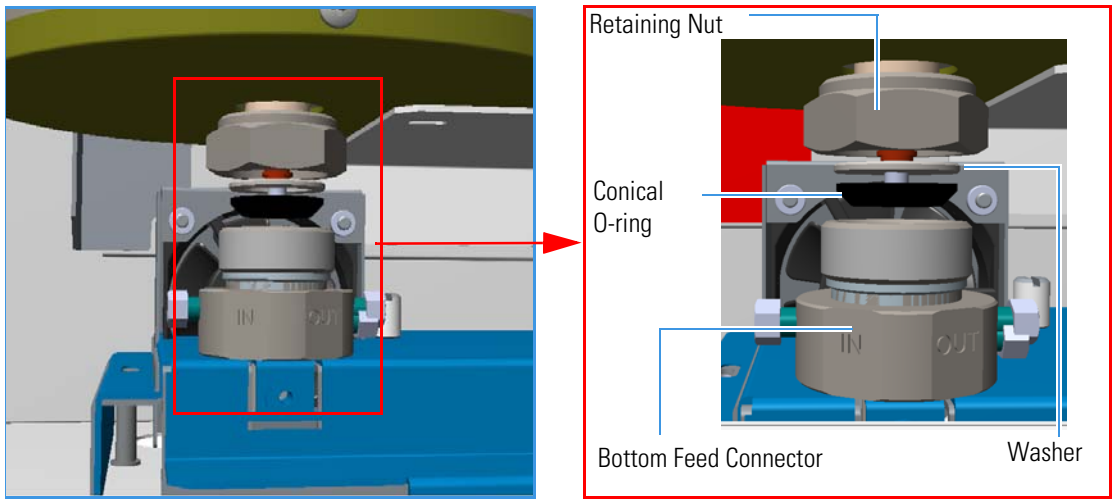
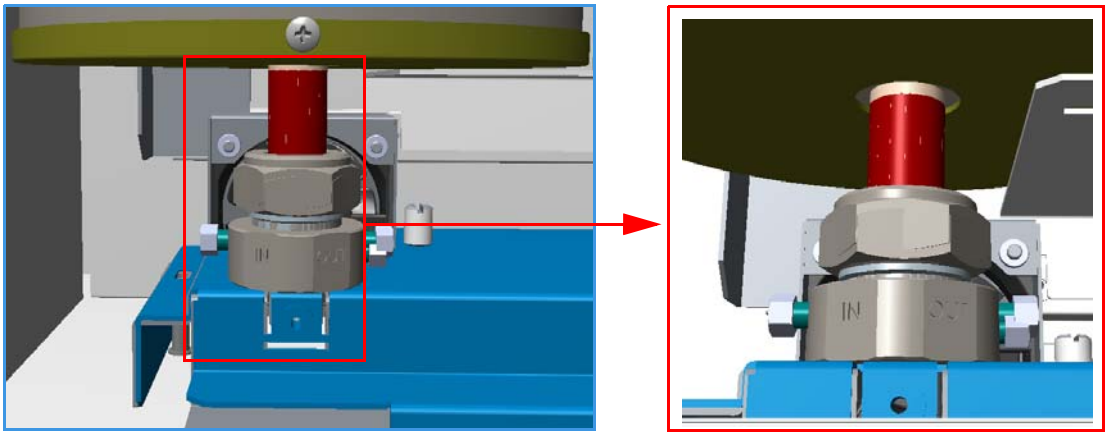
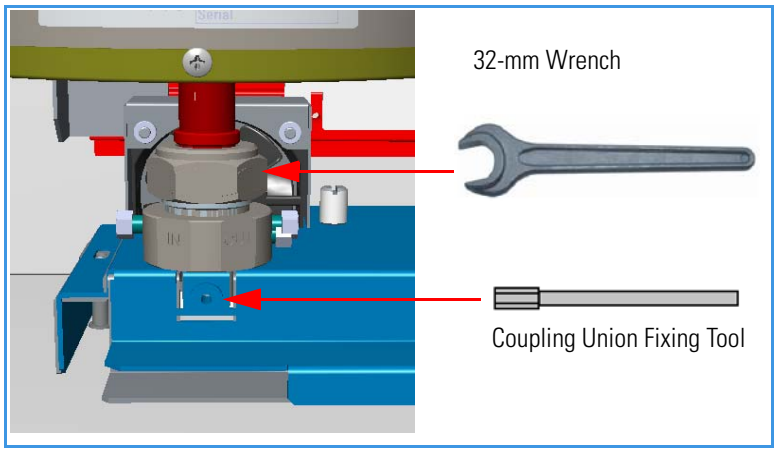


Figure 55. Introduction of the Reactor with a Bottom Feed Connector (3)



3. Now unscrew the nut and slip it with washer and o-ring over the ceramic tube at the bottom. Fix it using the steel rod and a 32 mm wrench. See Figure 56.

Figure 56. Introduction of the Reactor with a Bottom Feed Connector (4)

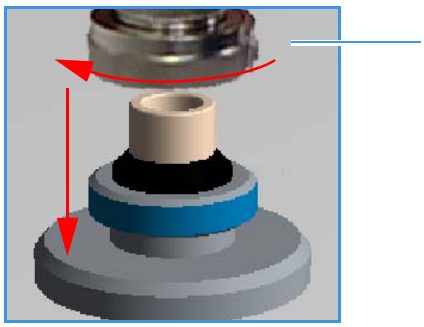


4. Slowly insert the granules into the glassy carbon tube from top. Make sure no granule blocks the tube. Check the height with the mark on the graphite crucible remover tool by placing the crucible

on the crucible remover and inserting it into the glassy carbon tube. The distance from the top of the crucible to the rim of the ceramic must be 270 mm. Here is the hottest zone of the furnace.

5. Drop the crucible into the reactor and introduce the insert on top of the glassy carbon tube.
6. To complete the operation manually screw the fixing nut of the autosampler. See Figure 63.

Figure 57. Reinstall MAS Plus Autosampler



7. Close the furnace compartment with the protecting plate and the front cover.

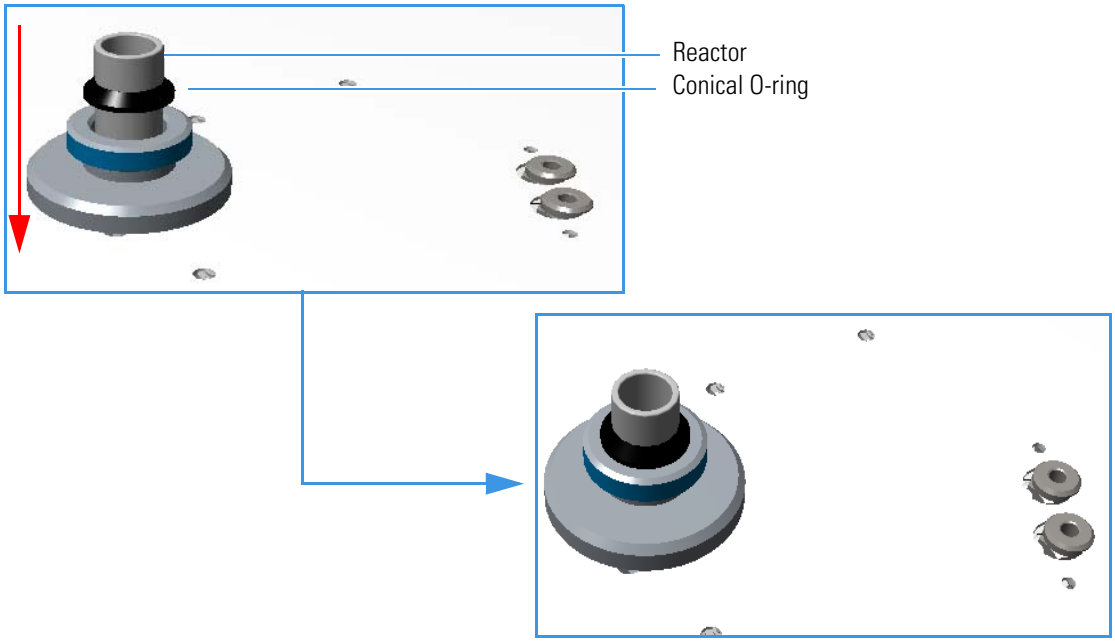
❖ **To Install the combustion reactor into the right furnace**

Note The figures in this operating sequence show the installation of a reactor into the right furnace.

Material required
Reactor retaining nut
Washer
Conical O-ring
Coupling union fixing tool
32-mm wrench

1. Carefully introduce and guide the reactor into the furnace until the conical O-ring on the top of the reactor touches the furnace fitting. See Figure 58.

Figure 58. Introduction of the Combustion Reactor Into the Right Furnace (1)



2. Onto the bottom end of the reactor, which protrudes from the bottom of the furnaces, slide first the reactor retaining nut, then the washer, and finally the conical O-ring, paying attention that the conical section of the O-ring must be turned downwards. See Figure 59 and Figure 60.

Figure 59. Conical O-ring

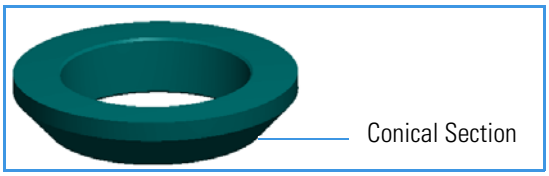
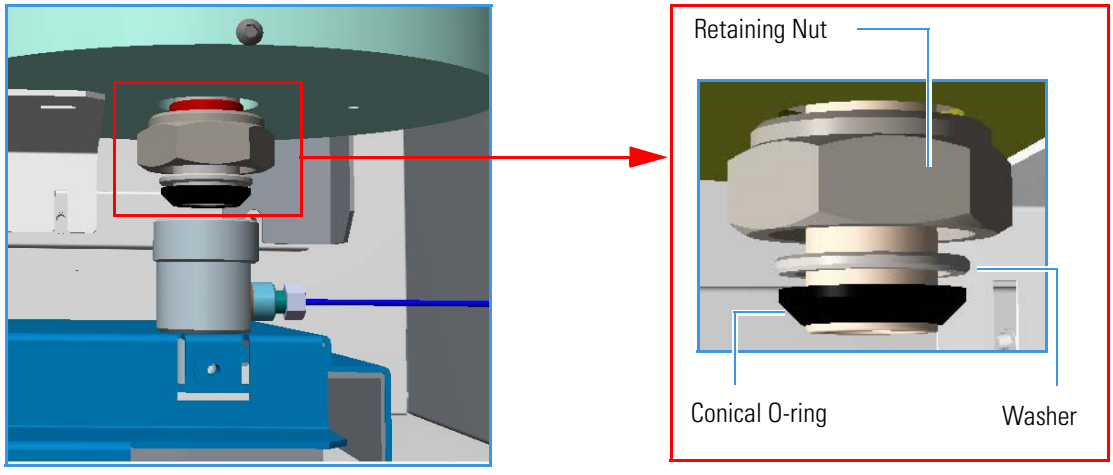
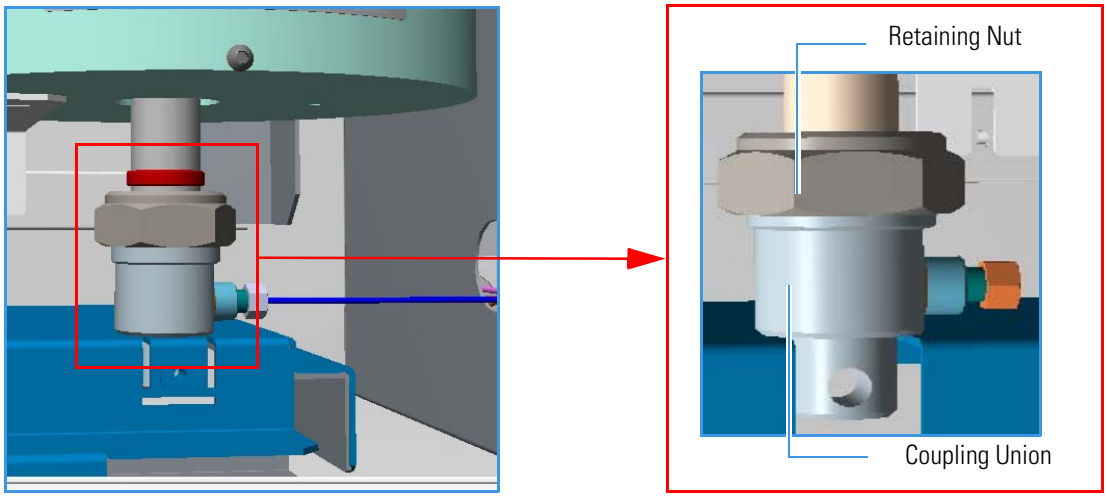


Figure 60. Introduction of the Combustion Reactor Into the Right Furnace (2)



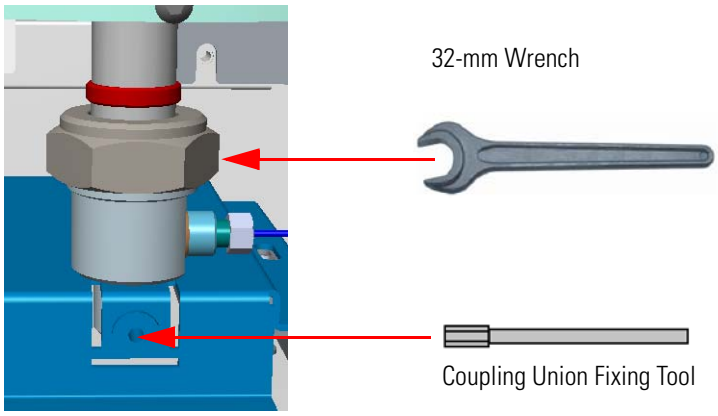
3. Insert the reactor end into the coupling union located on the base of the furnace compartment. See Figure 61.

Figure 61. Introduction of the Combustion Reactor into the Right Furnace (3)



4. Finger-tighten the reactor retaining nut until it starts to grip the coupling union.
5. Use the coupling union fixing tool and the 32-mm wrench to tighten the retaining nut. See Figure 62.

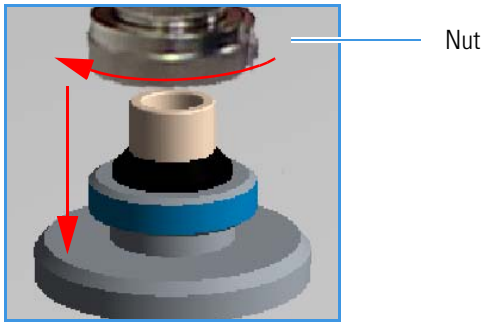
Figure 62. Introduction of the Combustion Reactor Into the Right Furnace (4)



CAUTION Use appropriate pressure to obtain a good sealing (1/4 to 1/2 turn). Do not overtighten the retaining nut to avoid reactor damaging.

6. To complete the operation manually screw the fixing nut of the autosampler. See Figure 63.

Figure 63. Reinstall MAS Plus Autosampler



7. Close the furnace compartment with the protecting plate and the front cover.

Setting Pressure and Flow Parameters

The following tables report the pressure and flow parameters to set if the helium and oxygen flows are:

- Preset at the helium, and oxygen pressure regulators.
- Adjusted and controlled through the software by means of the EFC-t control.

NCS Determination

Table 18 details the pressure and flow parameters for NCS determinations.

Table 18. NCS Determination Pressure and Flow Parameters Setting

Parameters	Flow adjustment/control through software by EFC-t control
Flow to set through Isodat Software Suite/Eager*Smart* Data Handling Software	
Helium as carrier gas	180 mL/min (for pressure 280-300 kPa)
Helium as reference gas	70 mL/min (for pressure 280-300 kPa)
Oxygen	250 mL/min (for pressure 250 kPa)

H-O Determinations

See Table 19 details the pressure and flow parameters for H-O determinations.

Table 19. O-H Determination Pressure and Flow Parameters Setting

Parameters	Flow adjustment/control through software by EFC-t control
Flow to set through Eager*Smart* Data Handling Software/sodat Software Suite	
Helium as carrier gas	100 mL/min (for pressure 280-300 kPa)
Helium as reference gas	100 mL/min (for pressure 280-300 kPa)
Oxygen	0 mL/min (for pressure 250 kPa)

IMPORTANT For H-O Analysis only: From Isodat Software Suite open the **Elemental Analyzer Status** page. Select **Special Function** then check **Disable Oxygen Injection** to ensure no oxygen introduction into the carrier circuit. This is usually taken care of by Isodat Software Suite automatically.
The color of the button turns **red**. Always remember to maintain a flow of minimum **10 mL/min** through the **NCS Separation Column** during **H-O** determination in order to avoid damage to the separation column in heated condition. This is the case in particular for liquid injections when no purge of the autosampler is required.

Installing Autosampler

As standard you must install the two MAS Plus autosamplers for solid samples furnished with the instrument. See Chapter 4, "Installing MAS Plus Autosampler."
Alternatively, if you need to analyze liquid samples, you must install the AI/AS 1310 autosampler. See Chapter 5, "Installing AI 1310/AS 1310 Autosampler."

Isodat Software Suite

The instrument comes with installed Isodat Software Suite and appropriate configuration.
The software includes all necessary tools to operate the Flash IRMS and the mass spectrometer simultaneously. If for any reason Isodat Software Suite is not installed, install Isodat Software Suite as described in the Isodat Software Suite manual.

IMPORTANT A Flash IRMS can also be run as a stand-alone instrument for elemental analysis. In this case the instrument must be modified and the full Eager*Smart* Data Handling Software version must be installed. Please see the section Chapter 9, "Running the Flash IRMS as Stand-alone Instrument," and read the Technical Note.

Installing MAS Plus Autosampler

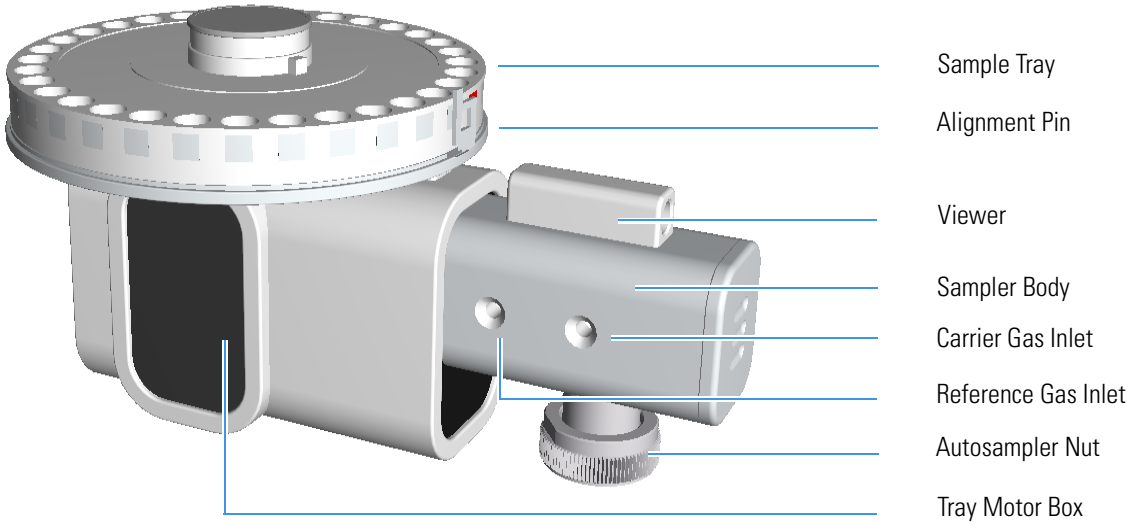
This chapter provides the instruction for installing the MAS Plus autosampler on the Flash IRMS.

Contents

MAS Plus Autosampler Overview

The MAS Plus autosampler for solid samples is provided with the Elemental Analyzer. See Figure 64.

Figure 64. MAS Plus Autosampler

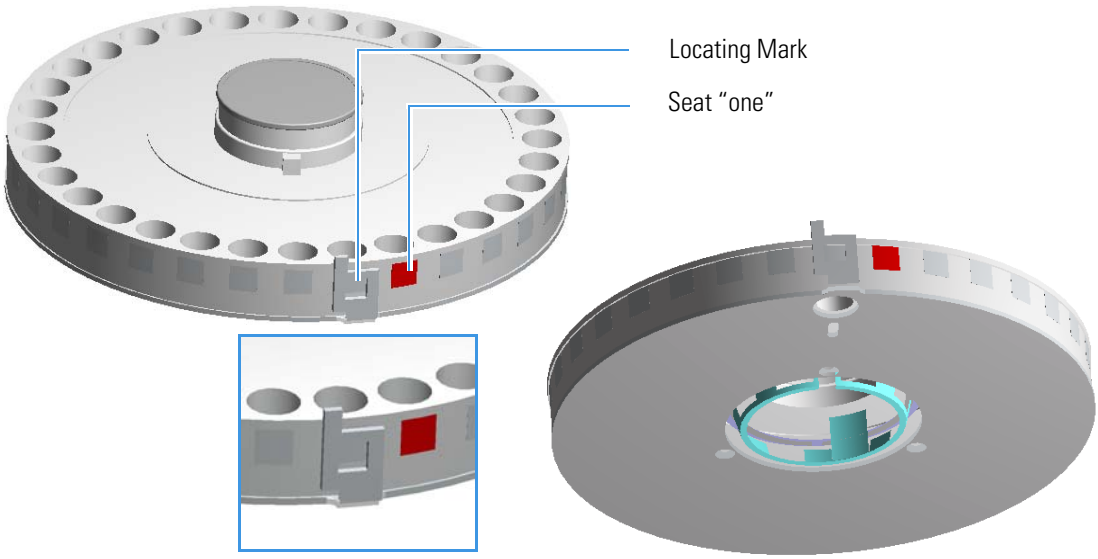


It consists of:

- An anodized aluminum block (sampler body) provided on the left side with fittings for carrier gas and reference gas lines connection.
- A 32-position sample-holding tray numbered 1 to 32. It is provided with a reference pin to be introduced into the seat marked 1(one) which has a locating mark. See Figure 65.

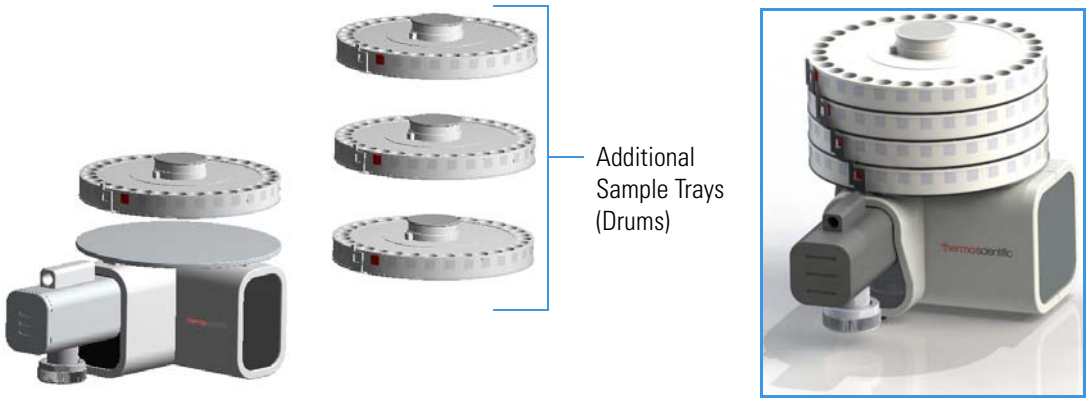
Purge/reference gas inlet

Figure 65. Sample Tray



The modular design of the MAS Plus allows up to three additional 32-position sample trays can be added to reach a capacity of 125 samples. Each sample tray is installed in a specific position defined by the numbering, and therefore they are not interchangeable. See Figure 66

Figure 66. Additional Sample Trays



The sample numbering is detailed in Table 20.

Table 20. Sample Tray Numbering

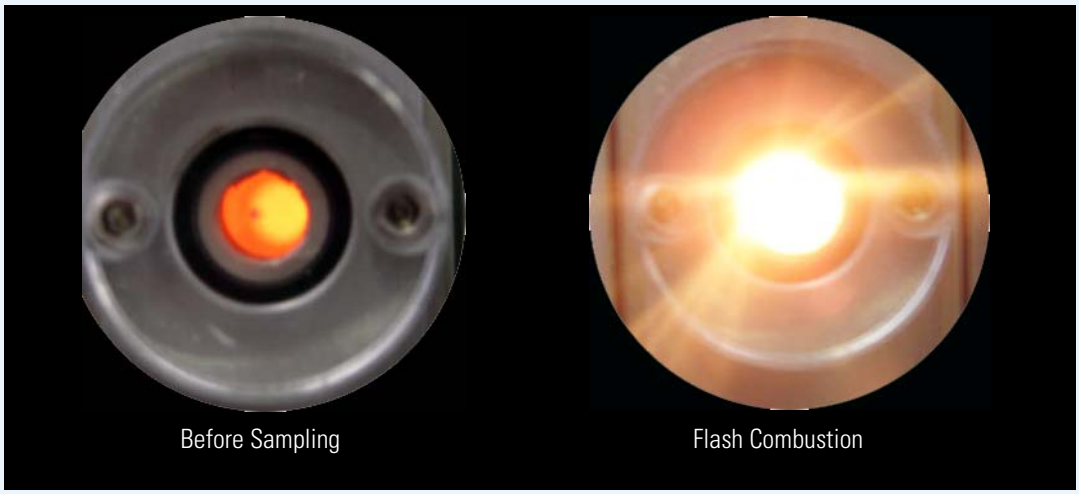
Sample Tray	Locating Mark	Numbering
#1	Seat marked 1 (one)	from 1 to 32
#2	Seat marked 0 (zero)	from 33 to 63
#3	Seat marked 0 (zero)	from 64 to 94
#4	Seat marked 0 (zero)	from 95 to 125

Any sample has to place in the locating mark position. The correct alignment of the locating mark is important for the installation of the sample tray on the MAS Plus autosampler. See Figure 67.

Figure 67. Sample Trays

- A motor for the tray.
- A viewer on the sampler body.

Tip The viewer on the sampler body allows observation of the Flash combustion.



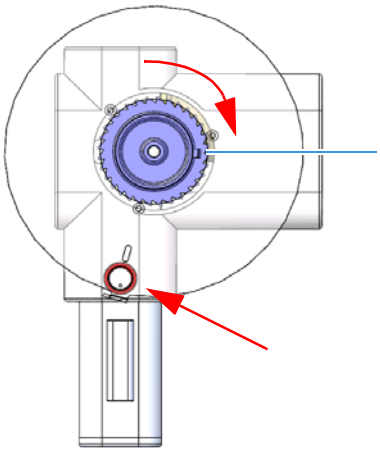
MAS Plus Autosampler Installation

WARNING Before starting, make sure that the EA IsoLink IRMS System for CNSOH is powered off and that the reactors required for your analyses are installed in their corresponding furnaces.

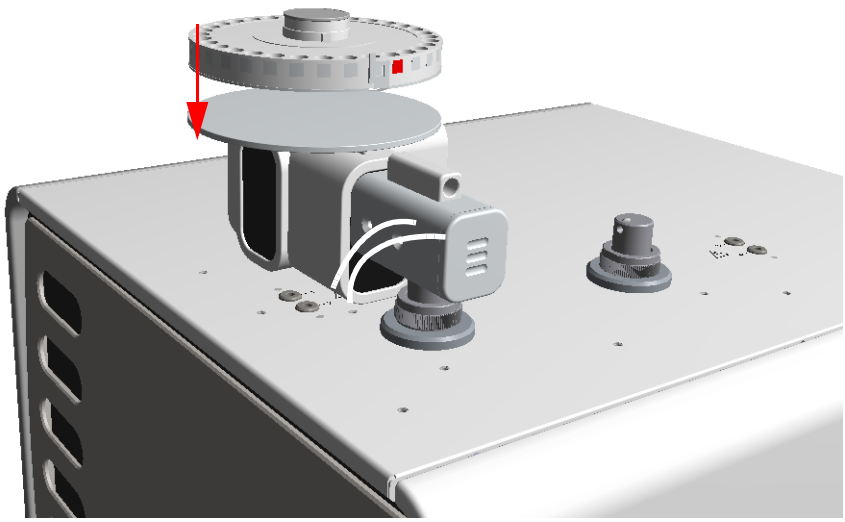
❖ **To install a MAS Plus autosampler**

Material Required
8 mm wrench

1. Place the autosampler on the connecting nut of the concerned channel.
2. Manually screw the autosampler nut on the concerned channel.
3. Connect the tubings coming from the gas connections, located on the analyzer, to the relevant connections of the autosampler.
4. Connect the signal cable of the MAS Plus autosampler to the 2-pin connector, marked **Autosampler**, on the back panel of the analyzer.
   Take care that the autosampler on top of the left furnace is connected to the connector marked **HO** and the autosampler on top of the combustion furnace is connected to the connector marked **NCS**. See Figure 20 on page 21.
5. Install the samples tray.
   a. Manually rotate the toothed wheel clockwise until the guide located on its rim is perfectly aligned with the metal pin of the autosampler body.

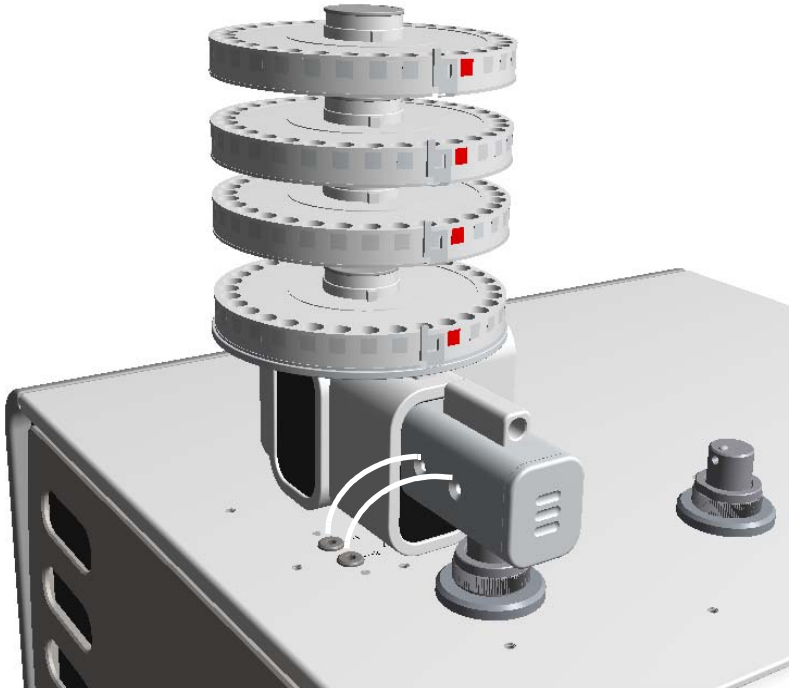


   b. Check that the sample tray (drum) reference pin is in correspondence with the seat marked "1".
   c. Place the sample tray, with the reference pin in correspondence with the "1" seat, onto the toothed wheel, paying attention to have the base match with the guides.



   d. Place the protection cover over the sample tray with the surface marked "**Side up**" turned towards you.

6. If additional sample trays are required, install them in the correct order one over the other, paying attention that the relevant locating marks are in correspondence with the relevant seats marked **0** (zero), and placing the samples properly.



IMPORTANT Before installing an additional sample tray, make sure that the samples to analyze are placed in all the seats of the previous tray installed.

CAUTION Before starting samples analyses, make sure that the protection cover is positioned over the sample tray. A complete deaeration of the area where samples are housed is only possible if the cover is in place.
Pay attention not to invert the cover; the surface marked "*Side-up*" must be turned towards you.

Installing AI 1310/AS 1310 Autosampler

This chapter provides the instruction for installing the AI 1310/AS 1310 autosampler for liquid samples on the Flash IRMS Elemental Analyzer.

Contents

Preliminary Information

This section contains the preliminary information for the installation and the connection of the AI 1310/AS 1310 sampling system to the Flash IRMS Elemental Analyzer.

Who Performs the Installation

The AI 1310/AS 1310 is installed by authorized Thermo Fisher Scientific technical engineers, who will check its correct operation. For more details, please contact Thermo Fisher Scientific local representatives. Should the instrument not be installed by Thermo Fisher Scientific personnel, strictly adhere to the instructions reported in this section.

Electrical Requirement

The instrument has the following power supply rating:

- 24 Vdc through a portable external power supply, level VI efficiency
  - input 100-240 Vac; 50-60 Hz — output 24 Vdc; 3 A - 3.75 A

WARNING YOU MUST ONLY USE THE PORTABLE EXTERNAL POWER SUPPLY FURNISHED WITH THE INSTRUMENT BY THERMO FISHER SCIENTIFIC.

CAUTION The power line and the connections between the instruments must maintain good electrical grounding. Poor grounding represents a danger for you and might seriously affect the instrument performance. Do not connect the AI 1310/AS 1310 sampling system to lines feeding devices of a heavy duty nature, such as motors, UV lamps, refrigerators, and other devices that can generate disturbances.

Lift and Carry the Sampling Unit

Lift and carry the sampling unit by hand. See Figure 68.

Figure 68. How to Lift and Carry the Sampling Unit

Installing the Direct Injection Device for Flash IRMS Elemental Analyzer

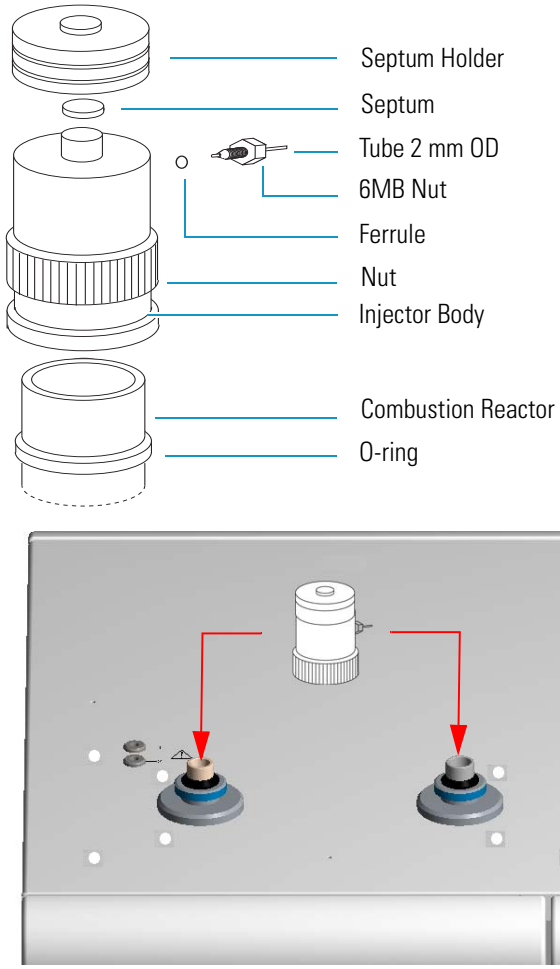
This device is installed in replacement of the MAS Plus autosampler, when present.
See Figure 69.

Figure 69. Direct Injection Device

❖ **To install the direct injection device**

1. If present, disconnect the MAS Plus autosampler from the reactor, and place the autosampler nut on the stainless steel plate. If the MAS Plus autosampler is not present, remove the reactor fitting unscrewing the relevant fixing nut.
2. Disconnect the gas connection.
3. Install the direct injection device over the reactor. See Figure 70.

Figure 70. Direct Injection Device Installation



Note When installing the injection device on the pyrolysis side the insert in the reactor must be replaced by the stainless steel insert which is provided in the water injection kit.
For detailed information please refer to the corresponding manual.

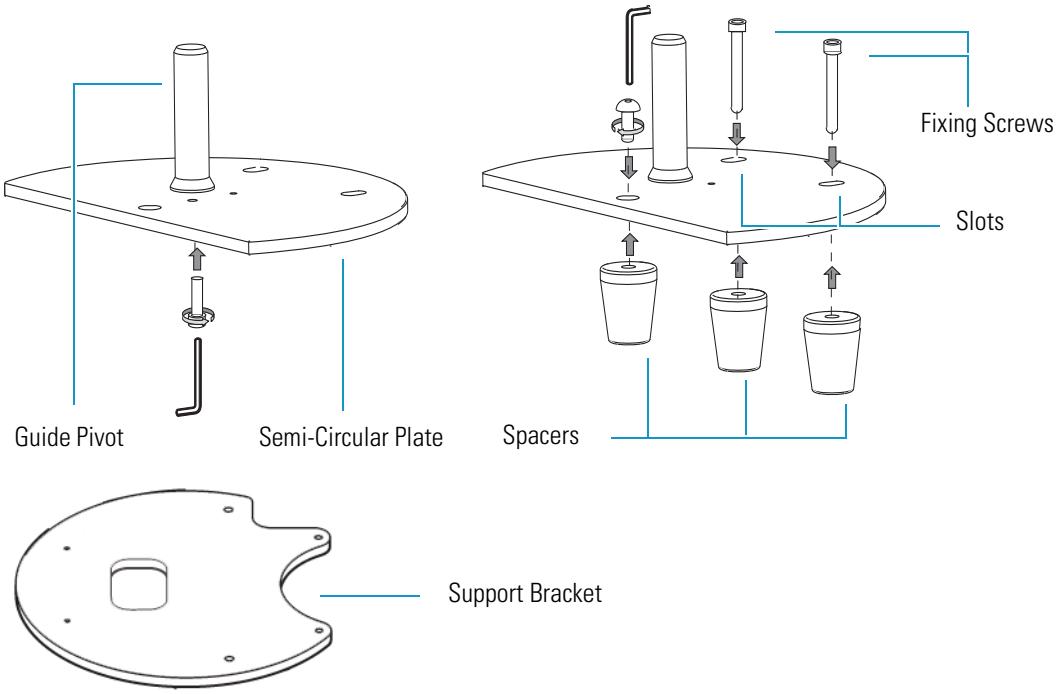
a. Mount the septum by using the septum holder provided in the standard outfit.

b. Connect the gas line to the direct injection device.

Installing the Sampler Support on the Flash IRMS Elemental Analyzer

The AI 1310/AS 1310 sampling system is installed on the Flash IRMS Elemental Analyzer by using the appropriate support provided. See Figure 71.

Figure 71. Sampler Support



The support consists of a semi-circular plate resting on three spacers non adjustable in height. The top surface of the plate is provided with two slots for the introduction of the corresponding fixing screws. Use the guide pivot for the accommodation and the centering of the sampling unit.
Before mounting the support, you must place and fix the support bracket on the top panel of the Flash IRMS Elemental Analyzer.

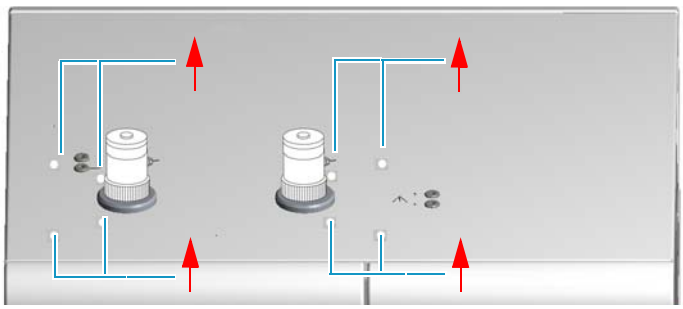
❖ To install the sampler support on the Flash IRMS Elemental Analyzer

1. Mount the support bracket.

   Note The support bracket can be installed on the left side as well as the right side of the Flash IRMS Elemental Analyzer. Install the support bracket on the side of interest according to the instrument configuration.

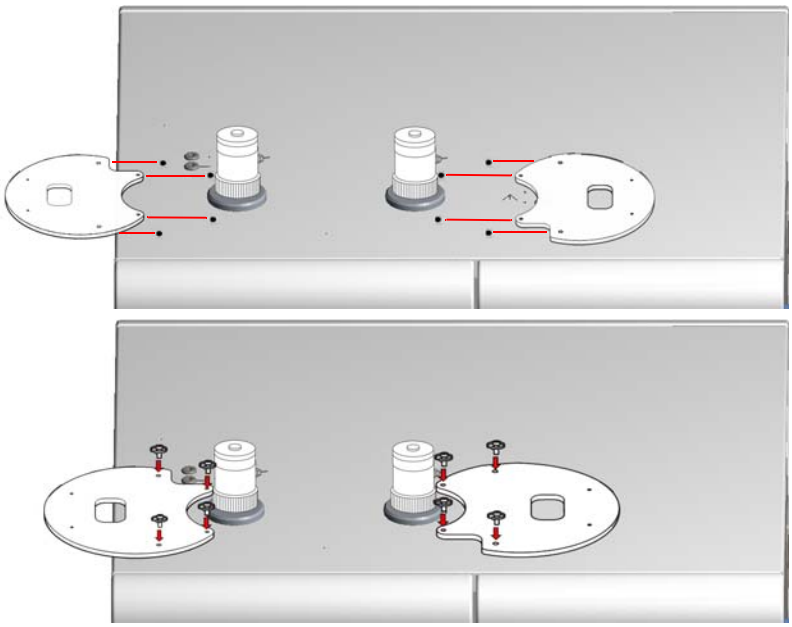
   a. From the top panel of the Flash IRMS Elemental Analyzer remove the four plastic caps covering the corresponding fixing holes. See Figure 72.

   Figure 72. Plastic Caps Removal

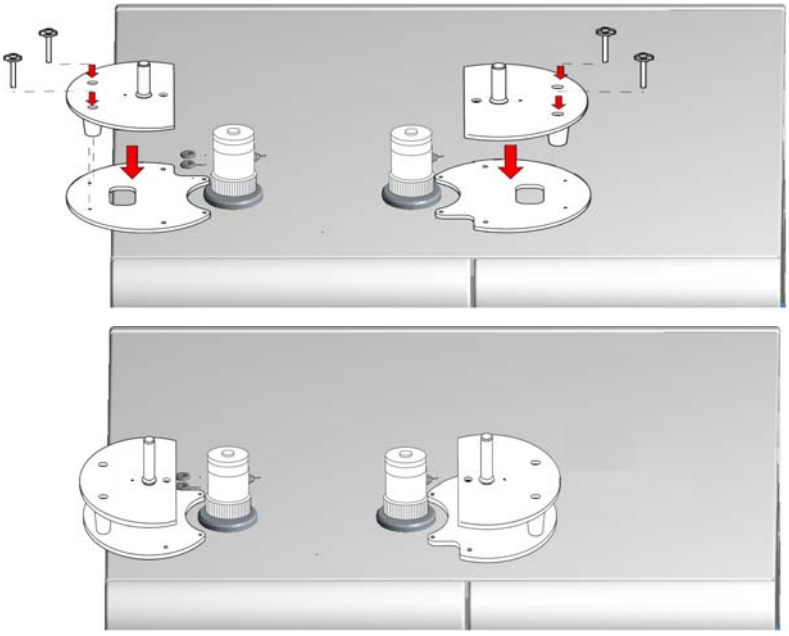
   b. Mount and fix the support bracket on the top panel of the Flash IRMS Elemental Analyzer by using the provided fixing screws. See Figure 73.

Figure 73. Mounting Support Bracket



2. Mount the sampler support. See Figure 74.

Figure 74. Mounting Sampler Support



a. Insert the provided fixing screw into each slot present on the support.

b. Insert each screw into the relevant spacer paying attention to keep its largest surface turned toward the support base.

c. Hold the spacers in position with their flat side toward the inside, then place the sampler support on the support bracket.

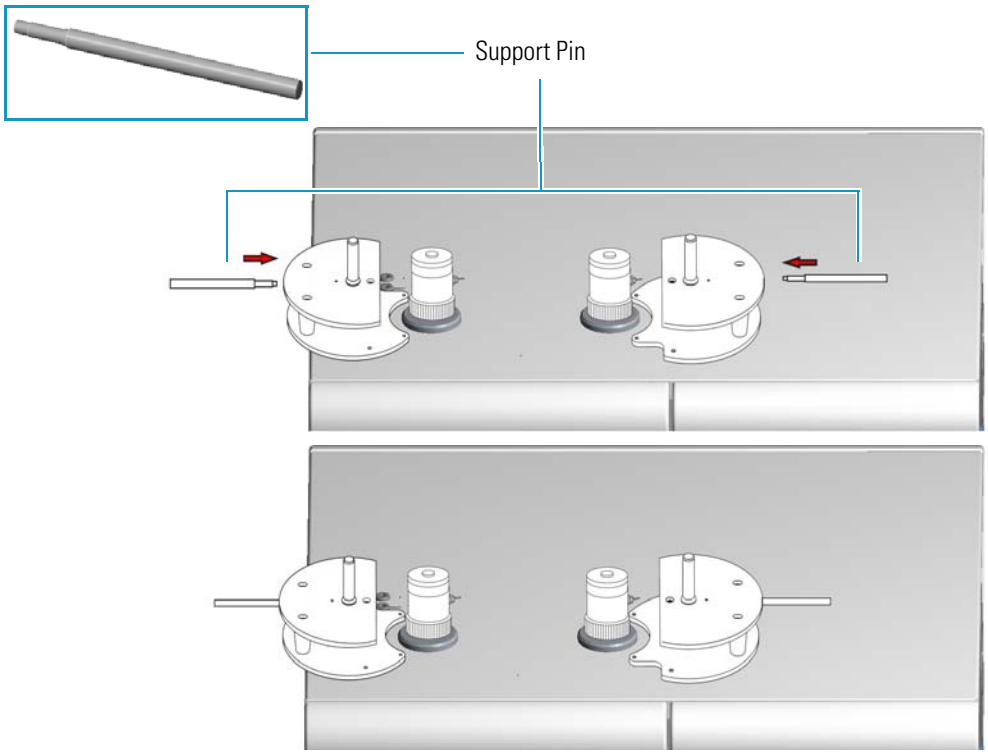
d. Guide the two fixing screws located on the external spacers into the corresponding fixing holes.

e. Loosely tighten the screws.

f. If you must install an **AS 1310**, screw the support pin into the dedicated hole on the top plate. See Figure 75.

Note The support pin is NOT required if you must install an AI 1310.

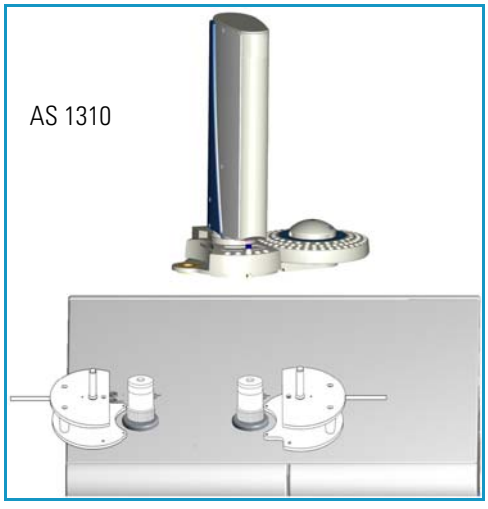
Figure 75. Mounting Support Pin



Installing the AI 1310/AS 1310 on the Flash IRMS Elemental Analyzer

This section provides the instruction for installing the AI 1310/AS 1310 on the Flash IRMS Elemental Analyzer. See Figure 76.

Figure 76. AI 1310/AS 1310 Installation



The installation procedure includes the following steps:

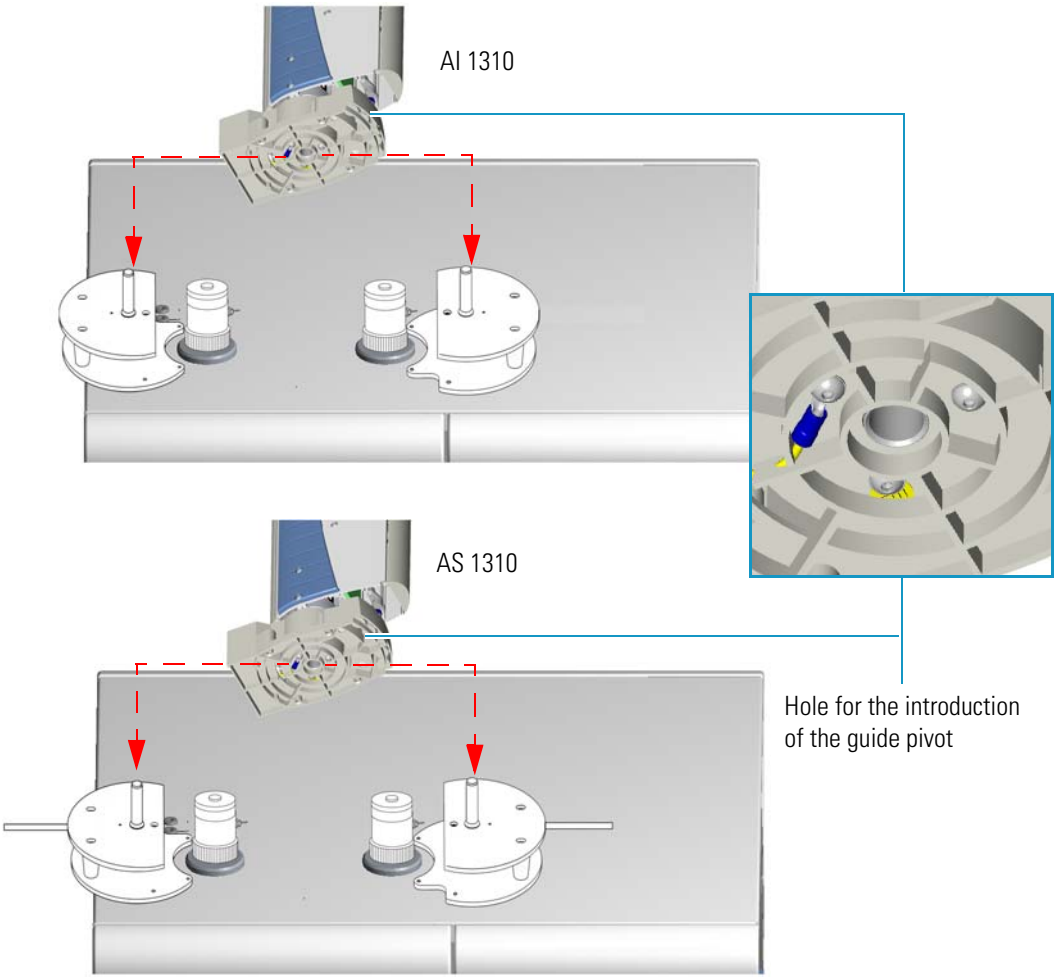
- Installation of the Sampling Unit
- Installing the Syringe
- Electrical Connections
- Starting-up

Installation of the Sampling Unit

❖ **To install the sampling unit**

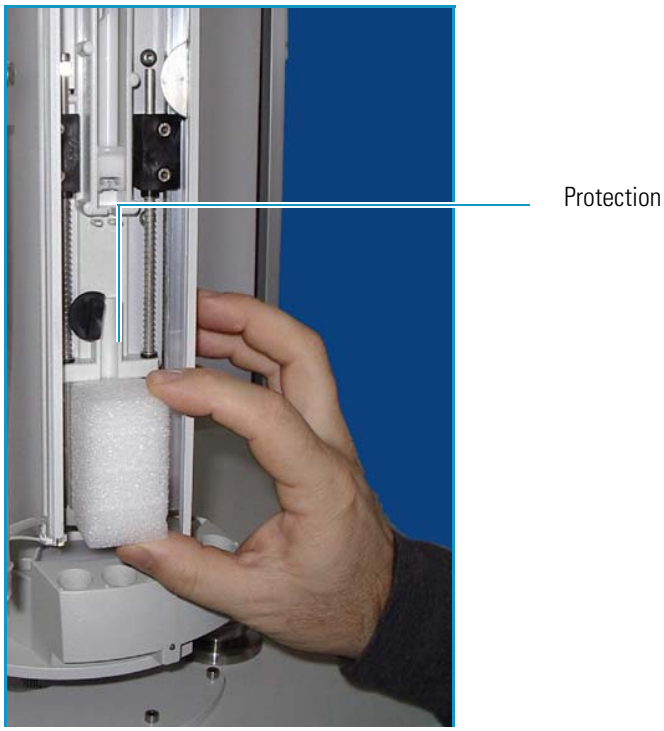
1. Lift the sampling unit and insert it into the guide pivot located on the sampling system support. Introduce the guide pivot into the hole provided on the bottom of the base. See Figure 77.

Figure 77. Installation of the Sampling Unit



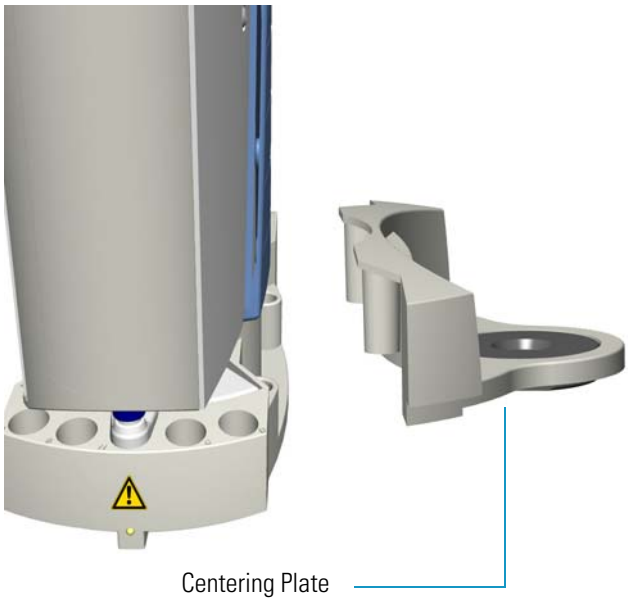
2. Open the safety door and remove the protection of the injection assembly. See Figure 78.

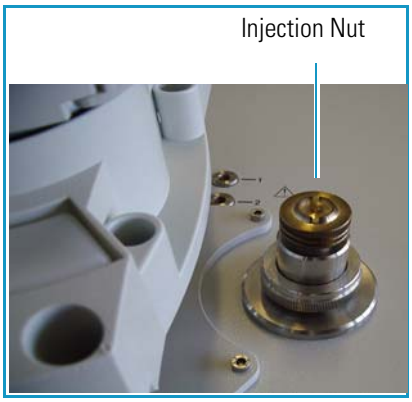
Figure 78. Remove Protection

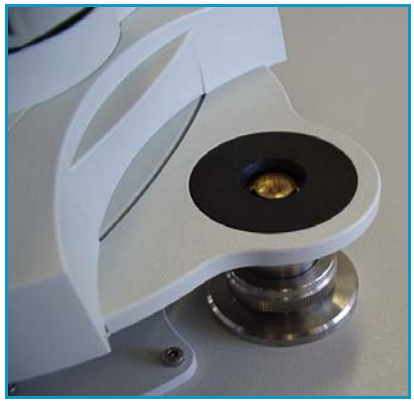


3. Insert the centering plate into its seat located in the **right** side section of the sampling unit base paying attention that the guide hole, present on the arm of the centering plate, correctly fits the injector nut. See Figure 79.

Figure 79. Centering Plate

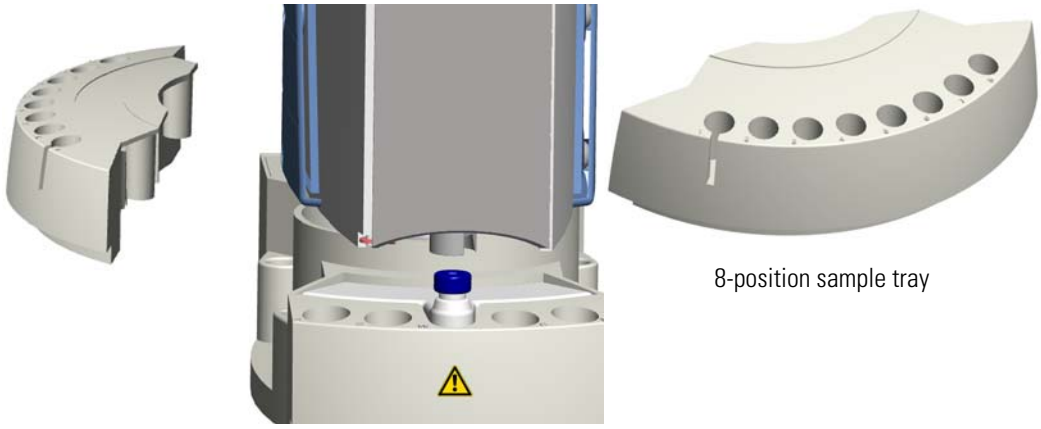






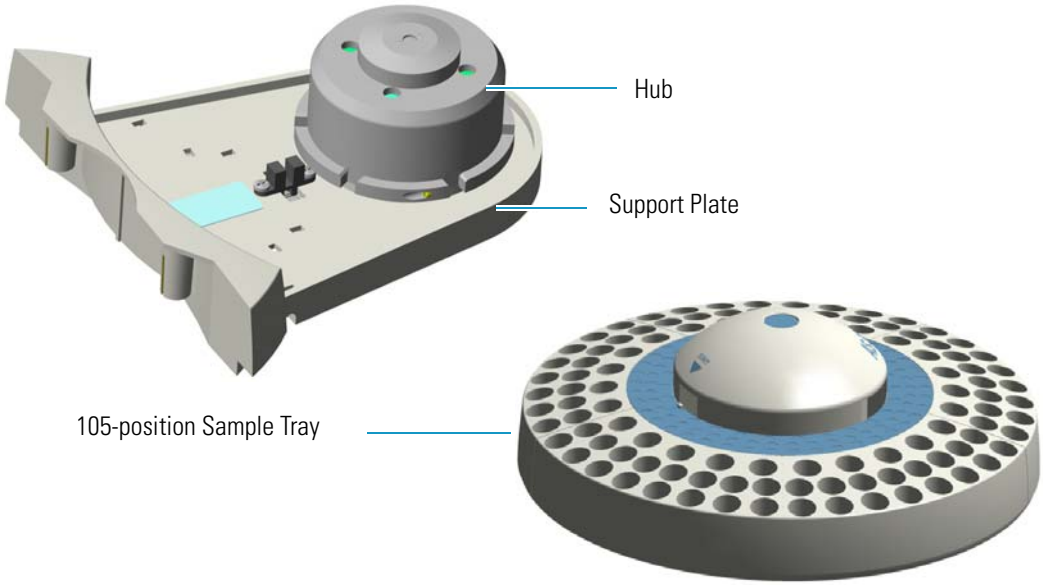
4. Check the correct alignment of the sampling system support, then fix it by tightening the proper fixing screws.
5. Insert the sample tray into the sampling unit base.
   - **AI 1310** — Insert the 8-position sample tray into the appropriate housing of the sampling unit base. See Figure 80.

     Figure 80. 8-position Sample Tray

   - **AS 1310** — Insert the dedicated support plate into its appropriate housing of the sampling unit base. Place the 105-position sample tray on the hub located on the support. The system will automatically recognize the sample tray at the instrument power on. See Figure 81.

     Figure 81. 105-position Sample Tray

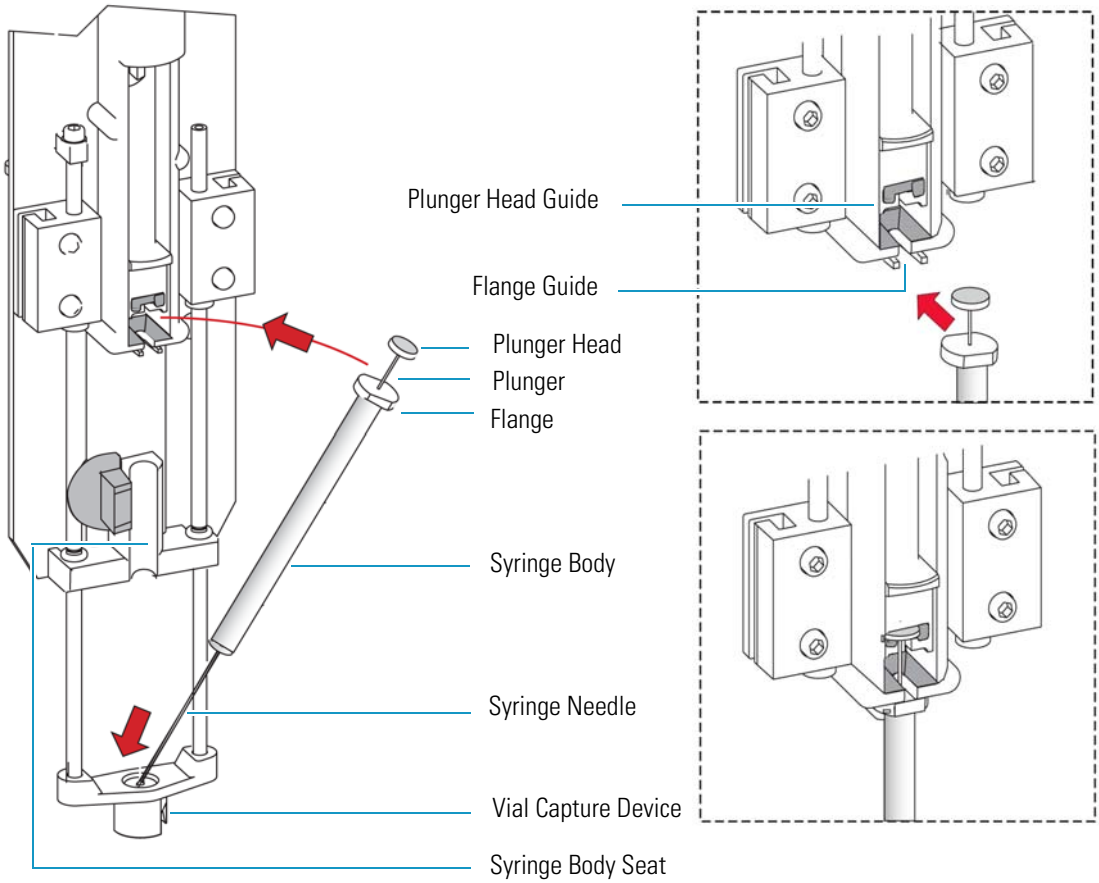
Installing the Syringe

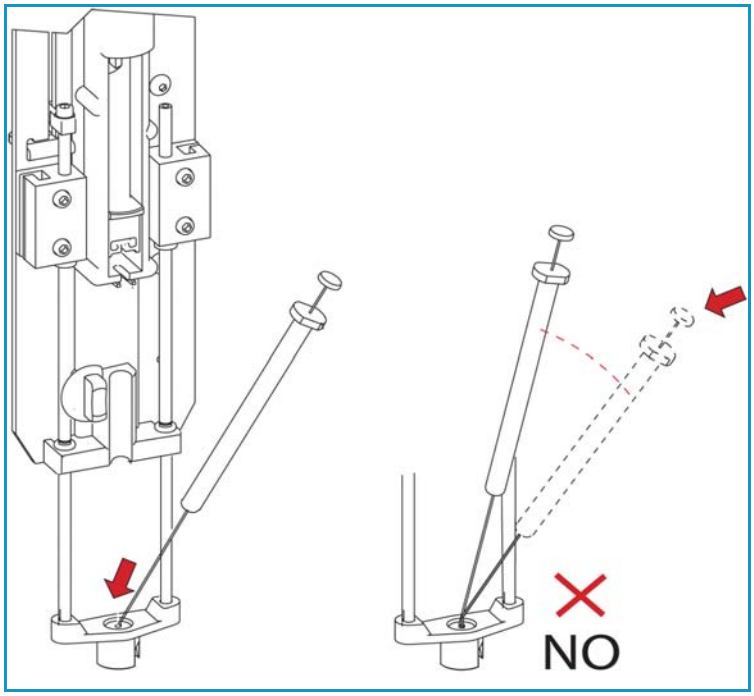
The installation of the syringe is a simple operation. However, it must be performed with caution to avoid damages to the syringe needle and ensure an optimal performance of the injection device. The standard syringes have 10 µL and 250 µL capacity with a 50 mm needle. It is also possible to install 50 µL and 100 µL syringes with needles of 50 mm.

❖ To install the syringe

1. Open the safety door of the turret.
2. Insert the syringe needle into the vial capture device. See Figure 82.

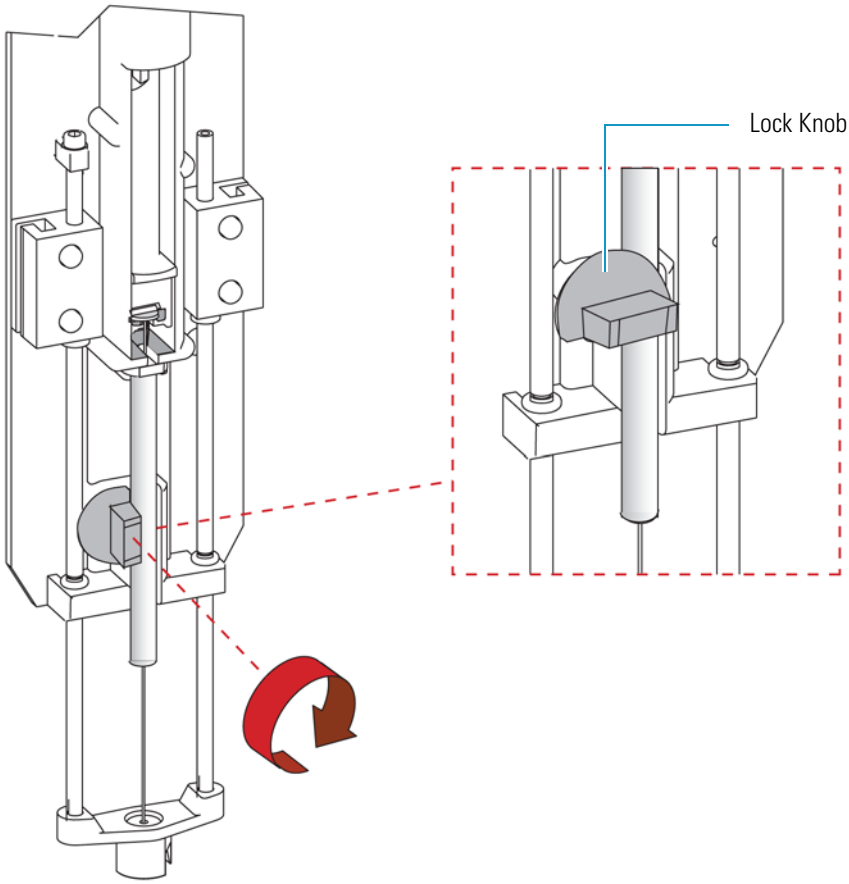
Figure 82. Syringe Installation (1)





3. Accommodate the syringe body into its seat paying attention to insert the flange and the head of the syringe plunger simultaneously into their relevant guides.
4. Turn the lock knob by approximately 180° clockwise to lock the syringe. See Figure 83.

Figure 83. Syringe Installation (2)



5. Close the safety door of the rotating turret.

Electrical Connections

❖ **To perform electrical connections**

1. By using the cable provided, connect the 9-pin female connector marked **RS232** located on the sampling unit back side to a 9-pin serial port connector (COM) of the PC.
2. Plug in the tampered connector provided into the 6-pin female connector marked **GC** located on the sampling unit back side.
3. **Only for AS 1310**, connect the 15-pin female connector of the cable, coming from the support plate of the 105-position sample tray, to the connector marked **TRAY** located on the back side of the sampling unit.

Starting-up

❖ **To start-up your AI 1310/AS 1310 sampling system**

1. Plug in the Vdc power cable of the external portable power supply level VI efficiency into the jack marked **24 Vdc** located on the sampling unit back side.
2. Connect the power cord of the external power supply to the mains outlet.

   The AI 1310/AS 1310 sampling system will automatically run the self-testing routine during which the following automatic checks and settings are carried out:

   - Alignment between AI 1310/AS 1310 sampling system and injector
   - Check of the turret travel
   - Acknowledgment of the installed sample tray
   - Calculation of the syringe zero

   Note The self-test routine is automatically carried out every time the safety door of the turret is closed.

6

Using EA IsoLink IRMS System for CNSOH

This chapter provides instruction for using the EA IsoLink IRMS System for CNSOH.

Introduction

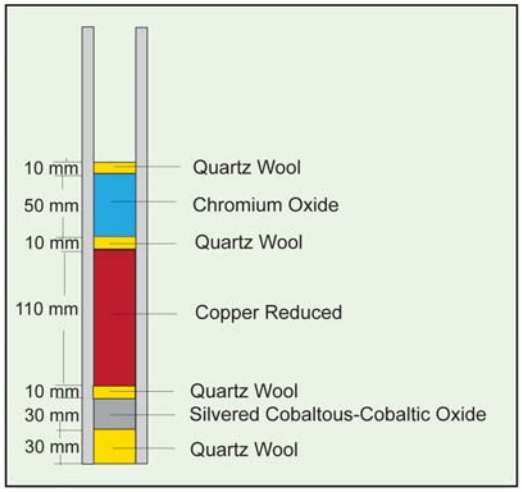
The EA IsoLink IRMS System for CNSOH is an enhanced elemental analyzer for O and H as well as N, C, and S isotope analyses and element analysis. It is equipped with an automatic switching valve (ASV) that allows fast switching from NCS analysis (combustion) to the OH analysis (high temperature conversion/pyrolysis). Isodat Software Suite is able to detect the required analysis mode by the gas configuration defined in the method, and automatically switches the carrier gas from one mode to the other. The system can be set up to run elements in single, dual or even triple mode, i.e. one element per sample, two elements per sample or three elements per sample. The ASV allows to switch unattended between pyrolysis and combustion so that five elements of a sample can be analyzed from two sample drops. This chapter covers the setup of dual measurements. Single measurement can be derived from it. Triple measurements are only recommended with the ramped GC Oven. These measurements are covered in the *Ramped GC Oven Operating Manual*.

Contents

Dual Measurement

Analyses of **N** and **C** in dual or single mode have to be performed with a reactor, which traps **SO_2**. The packing instructions for such a reactor are given in Figure 84.

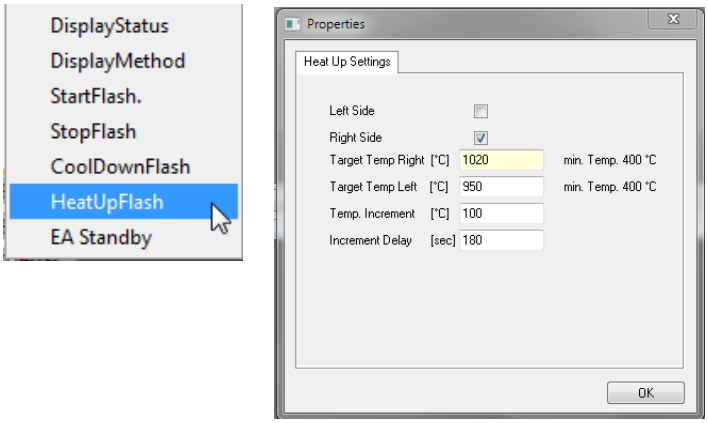
Figure 84. Reactor Setup NC



After installation of a new NC reactor heat up the furnace in the following stepwise manner: **room temperature > 900 °C > 1020 °C**. Upon reaching **900 °C** step, allow at least 15 minutes of equilibration time before commencing the next heating step.

A stepwise heating up of the reactor avoids temperature overshooting, and hence melting of the reduced copper contained in the reactor. Alternatively, use **HeatUpFlash** from the Context Menu with a right-click on the Flash IRMS peripheral visualization. See

Figure 85. HeatUpFlash



It is possible to perform dual measurements of hydrogen and oxygen as well as carbon and nitrogen from a single sample with the system. This analysis mode is suitable for both solid and liquid samples. If no S analysis is required, it is recommended to use a reactor setup that traps SO_2. If S analysis is required, it is recommended to use a triple analysis.

Procedure

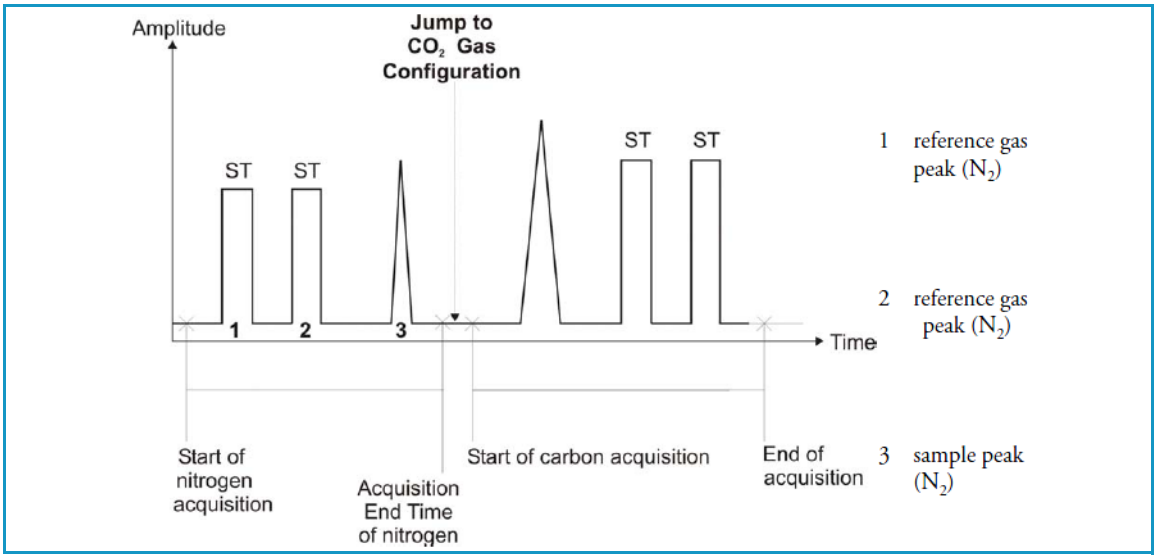
For the analysis of two isotopic species (hydrogen and oxygen, or nitrogen and carbon) from a single sample, an Isodat Method, which comprises both of them has to be defined.

Due to the separation of the gas chromatographic column the acquisition is divided in two parts, each dedicated to the isotopic species (i.e. gas configuration).

As soon as the first species has been identified (i.e. **H_2** or **N_2**), Isodat Software Suite automatically changes the magnetic field so that the mass traces of the second isotopic species (i.e. **CO** or **CO_2**) can be recorded (see: **Switch Gas** column in the **Time Events** list).

If no hydrogen or nitrogen peak can be found, Isodat waits a defined time, for example 25 s, before switching to the second gas configuration.

Figure 86. Dual Measurement for NC - Schematic Chromatogram



Performing a Jump Calibration

In order to determine the isotope ratios of different elements during the same run, switching to another Gas Configuration is necessary. In contrast to a single element measurement, in which the magnetic field runs the gamut from high to low and after that to the pre-calculated magnetic field, there is not sufficient time in dual measurement to perform this procedure for the next gas configuration. For this reason, a so-called **Jump Calibration** from the first gas configuration to the next gas configuration is necessary. After the **Jump Calibration** has been performed, the computer finds exactly the peak center even without performing any peak center procedure. A Jump Calibration should be performed daily to stabilize the performance of the magnet jump.

❖ How to perform a jump calibration

The following procedure describes how to perform a jump calibration for **H_2 --> CO**.
For an **N_2 --> CO_2** jump calibration proceed similarly.

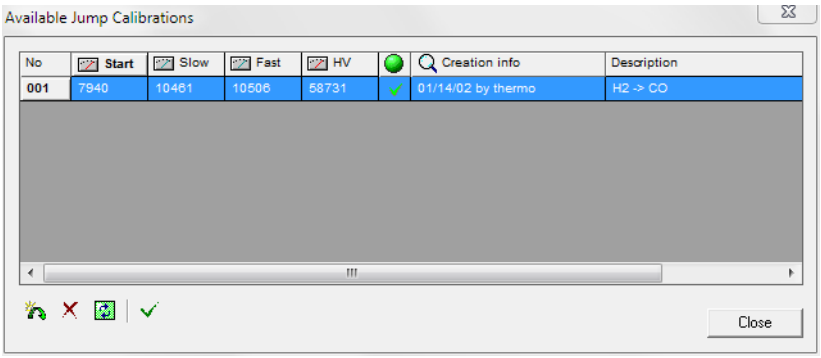
1. Open **Instrument Control**.
2. Choose your **Configuration** containing ConFlo IV and EA IsoLink IRMS System for CNSOH.
3. Allow **CO** reference gas to enter the source. In case of an **N_2 --> CO_2** jump calibration open CO_2 reference gas.
4. Select **Scan | Jump Calibration**.

a. In the list of available Jump Calibrations, mark the one for **H_2 --> CO** by clicking its No. (for example 001).

   – **Slow**: compensate hysteresis by Max/Min settings of the magnet.

   – **Fast**: magnet setting of Jump Calibration.

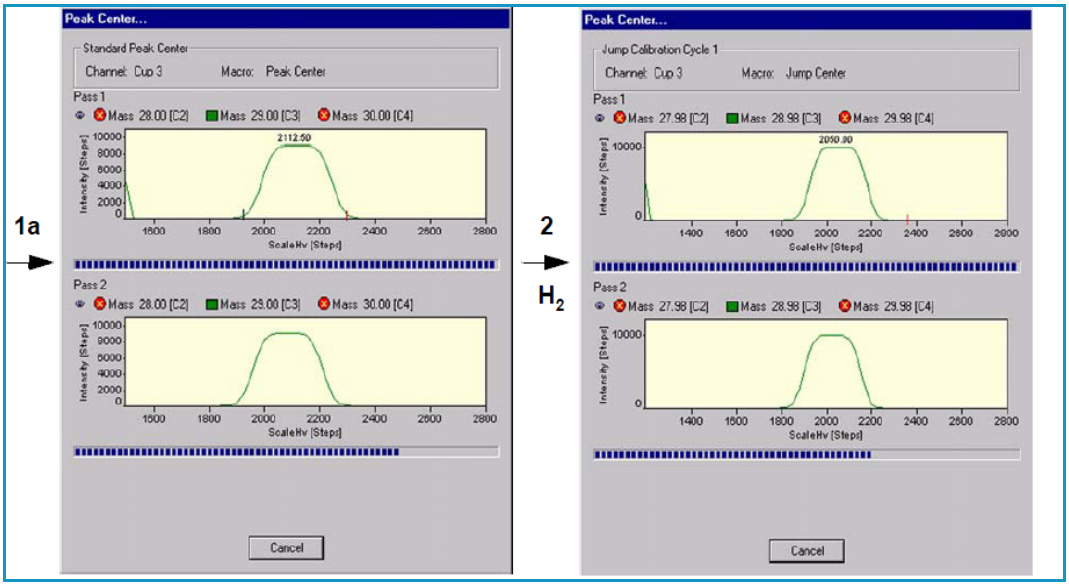
   – **HV**: high voltage setting of Jump Calibration.



b. Press **Recalibrate** Icon  .

> **Note** If no Jump Calibration is available in the list, create a new one as shown below:
>
> **How to Create a new Jump Calibration:**
>
> - Click **New**.
> - From **Gasconfiguration**, select **H_2.**
> - To **Gasconfiguration**, choose **CO**.
> - Accept the default values.
> - Click **OK**.
>
> **Note** Magnet jumps are always from a low mass to a high mass (e.g. 2 to 28 at H_2 -> CO, or 28 to 44 at N_2 ->CO_2).

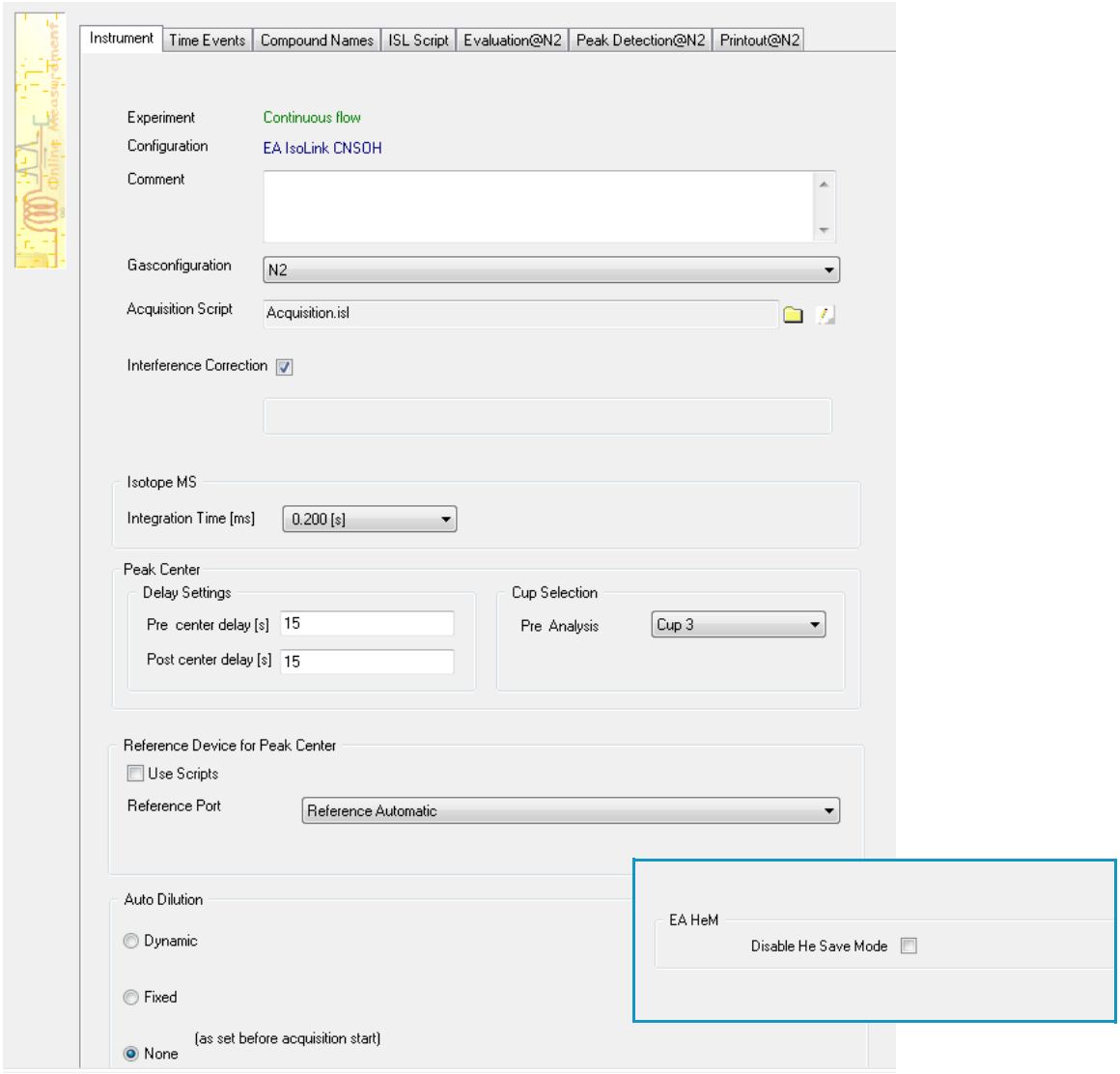
5. Confirm with Yes, because you have already opened the Reference Port.
6. Start Jump Calibration Procedure.
   a. Jump to **CO** (along hysteresis curve).
   b. Perform a peak center for **CO** in order to get the signal height.
   c. Jump to **H_2** (along hysteresis curve; H is origin).
   d. Jump to CO (not along hysteresis curve).
   e. Perform a peak center for **CO** in order to catch the peak.
   f. Repeat until magnet field setting is within the **magnet window** defined.


1a
Peak Center...
Standard Peak Center
Channel: Cup 3
Macro: Peak Center
Pass 1
Mass 28.00 [C2]
Mass 29.00 [C3]
Mass 30.00 [C4]
2112.50
Intensity [Steps]
ScaleHv [Steps]
Pass 2
Cancel
2
H2
Peak Center...
Jump Calibration Cycle 1
Channel: Cup 3
Macro: Jump Center
Pass 1
Mass 27.98 [C2]
Mass 28.98 [C3]
Mass 29.98 [C4]
2050.00
Pass 2
Cancel

Creating an Isodat Method for N+C Measurement (Dual Measurement)

For setting up an Isodat method for **N+C** analysis only, please refer to the figures from Figure 87 to Figure 96.

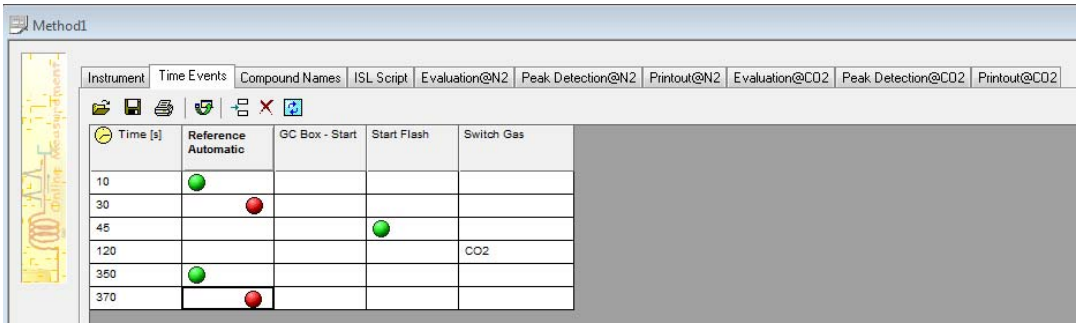
Figure 87. NC Method: Instrument Tab



Note

- The gas configuration denotes the starting gas configuration for an analysis. When analyzing only one element, e.g. CO_2, the starting gas configuration must be changed accordingly.
- The dilution for the second gas configuration appears only after defining a **Switch Gas** in the Time Event List (see below). The settings are examples and must be adjusted to your sample type. Dynamic dilution is only available with the smartEA Option.
- He^M settings — Ticking the box **Disable He Save Mode** will maintain the starting flow over the complete acquisition time (Analysis Mode). This mode does not switch into **He Save Mode**.

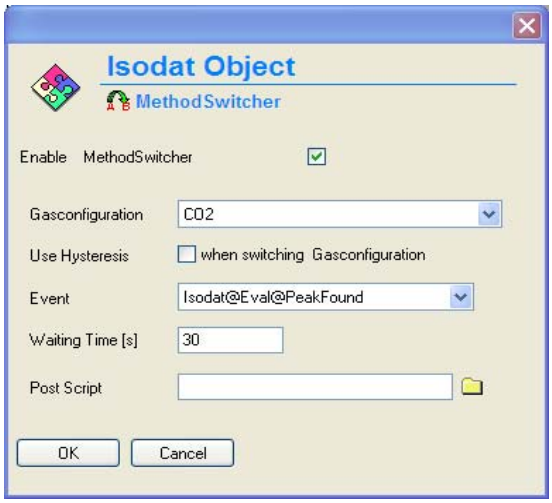
Figure 88. NC Method: Time Events List



Note By double-clicking on the relevant gas name in the time events list in column **Switch Gas**, you access to **Isodat Object Method Switcher** window. See Figure 90.

Figure 89. NC Method: Acquisition and Time

Figure 90. NC Method: Method Switcher



Note **Waiting Time** specifies the maximum period of time, for how long the system/software will wait for the previous peak (for example N_2 in a dual N-C measurement), to reach background again, and delay the subsequent peak jump.
For example: if you enter a waiting time of 10 seconds, the software will delay the peak jump as specified in the time event list by a maximum of 10 seconds.

- If the N_2 peak has already reached background until the time specified in the time events list, then the peak jump will occur straight away at that specified time.
- If the N_2 peak hasn't reached background until the specified time in the time events list and the entry for waiting time is for example 10 seconds (you can also enter a different waiting time or 0 seconds here), the peak jump will be delayed by a maximum of that number of seconds, so that the N_2 peak still has that number of seconds to reach background.
- If the N_2 peak reaches background within these seconds of waiting time, the peak jump will then occur. If the N_2 peak does not reach background within the waiting time, the peak jump will definitely occur after the waiting time period.

Figure 91. NC Method: Evaluation@N2

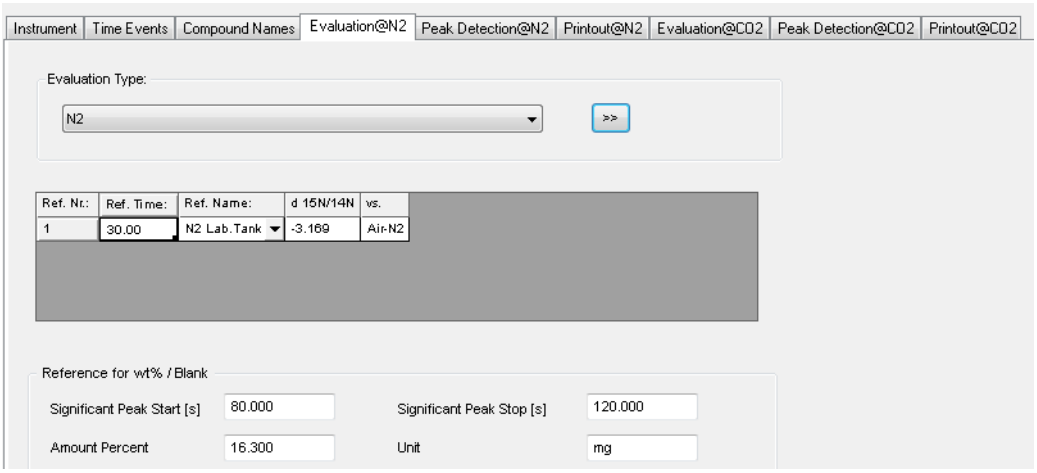


Figure 92. NC Method: Peak Detection@N2

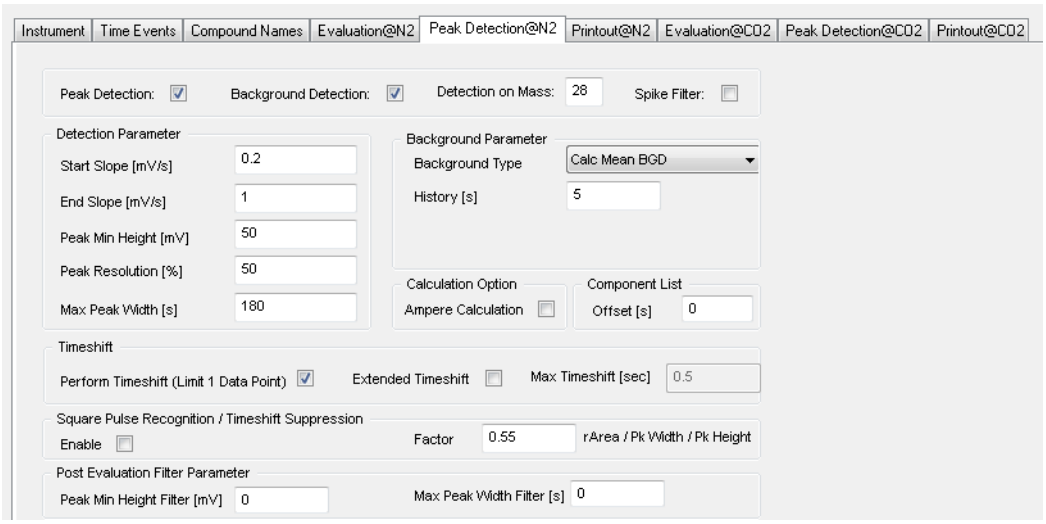


Figure 93. NC Method: Printout@N2Tab

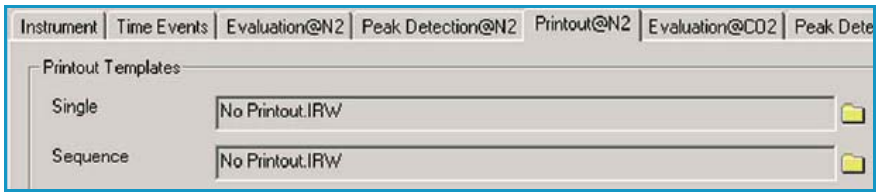


Figure 94. NC Method: Evaluation@CO2 Tab

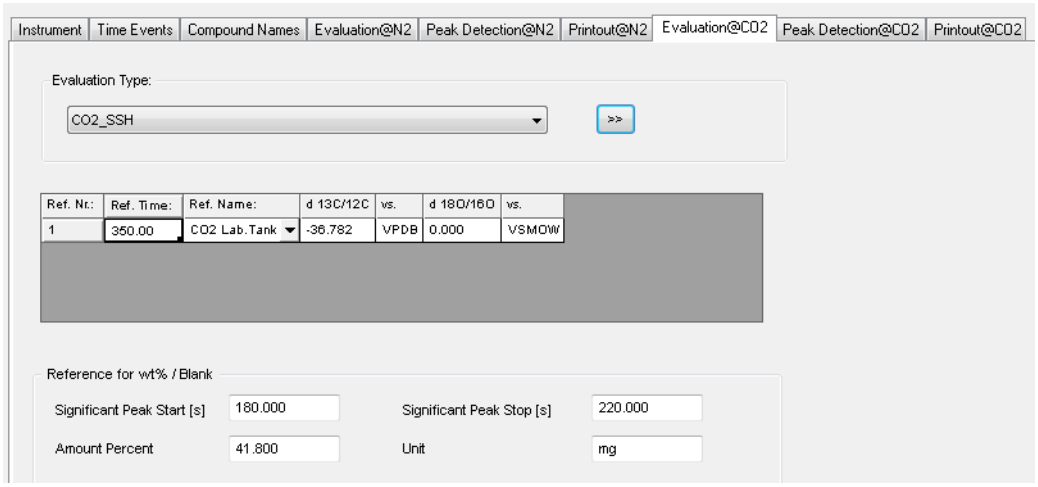


Figure 95. NC Method: Peak Detection@CO2 Tab

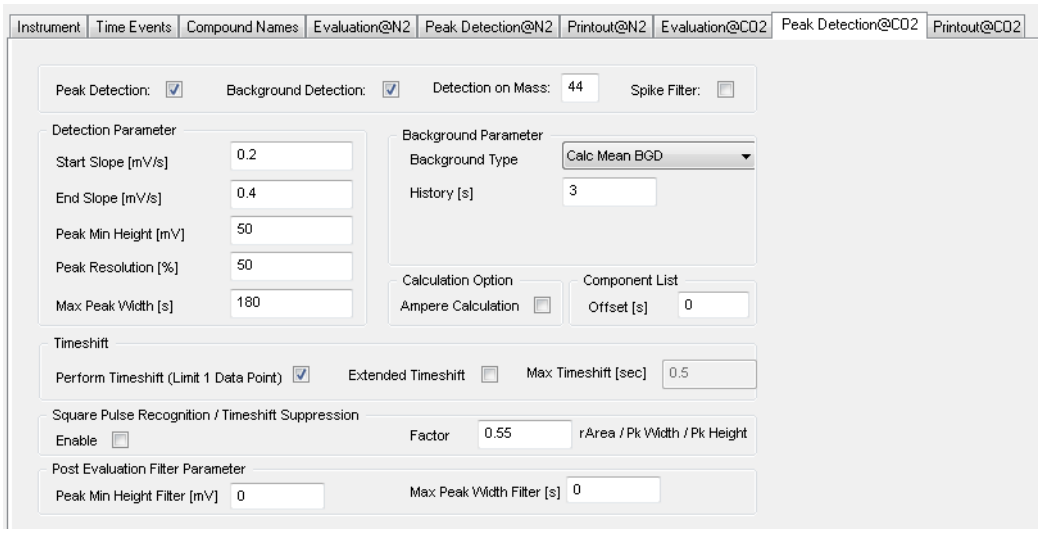
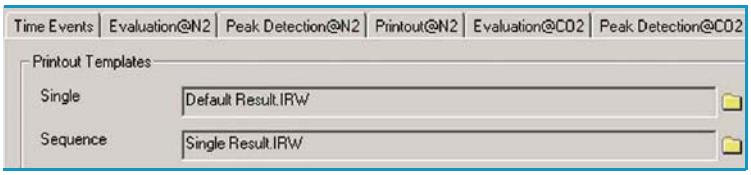


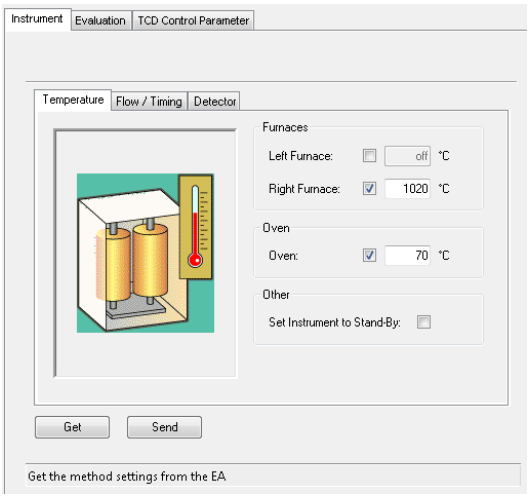
Figure 96. NC Method: Printout@CO2 Tab



Creating an EA Method for N+C Measurement

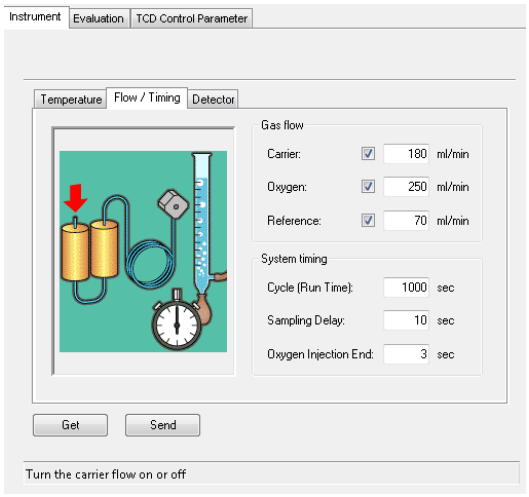
The following figures (Figure 97 to Figure 101) show how an EA-method for a dual analysis **N+C** may be set up.

Figure 97. NC EA Method: Instrument Temperature



Note The temperature of the high-temperature conversion furnace (left furnace) may be different depending on the intended use of it.

Figure 98. NC EA Method: Instrument Flow-Timing



Note Sampling delay and oxygen injection end vary depending on the sample nature. O_2 injection can be crucial in a single reactor if it exceeds the required amount.
The reference flow is used for the autosampler purge. This flow can be set to values between **20 mL/min** and **70 mL/min** (**150 - 200 mL/min in total when using He^M**), with the goal to minimize or eliminate the blank contribution of the autosampler.

For single mode N analyses, a second trap can be installed filled with **Carbosorb®** or **Ascarite®** to remove **CO_2**. The analysis time has to be reduced even further. In this case the entry Switch Gas **CO_2** can be deleted in the **Time Event List** of the **Isodat Method**.

Figure 99. TCD Settings

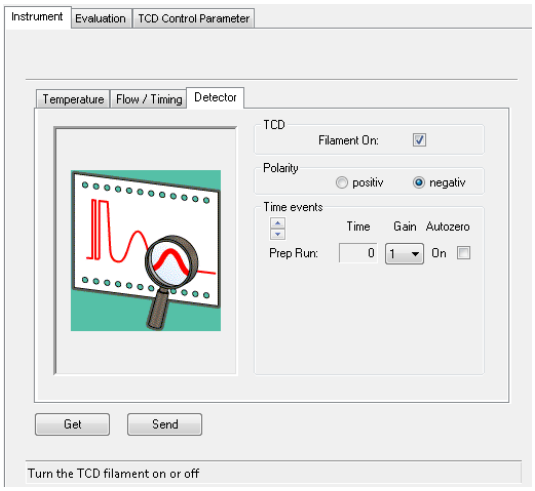


Figure 100. Peak Integration and Evaluation Parameters

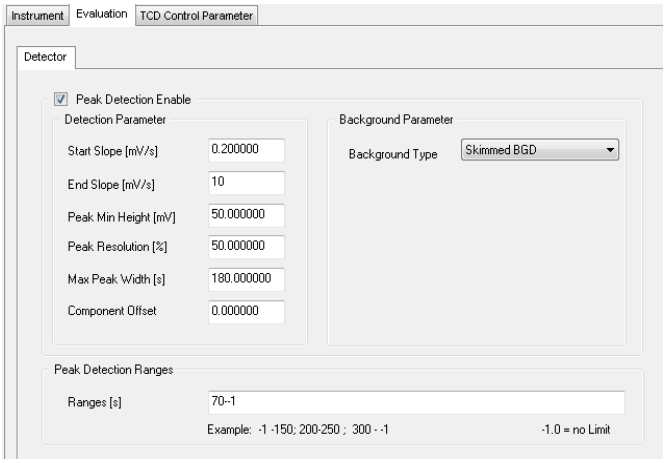
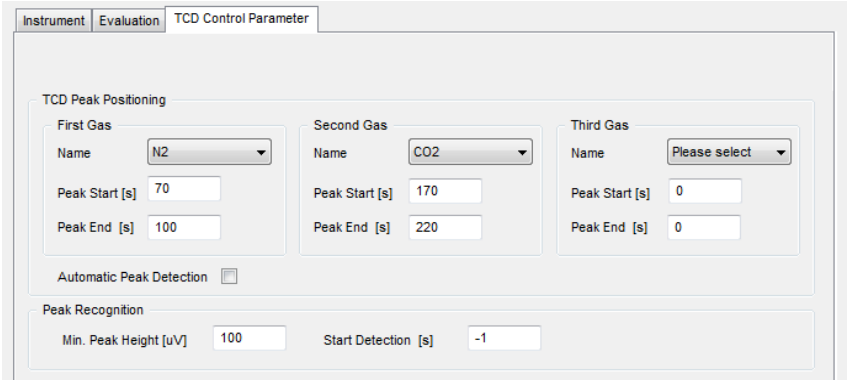


Figure 101. TCD Control Parameters



Creating an Isodat Method for Single Mode S Measurements

This Isodat method must be used with reactor packing as for the triple **NCS** measurements. The elution time of the SO_2 peak can be decreased if the GC oven temperature is ramped earlier. For setting up an Isodat method for single mode **S** analysis only please refer to the *Ramped GC Oven Operating Manual*.

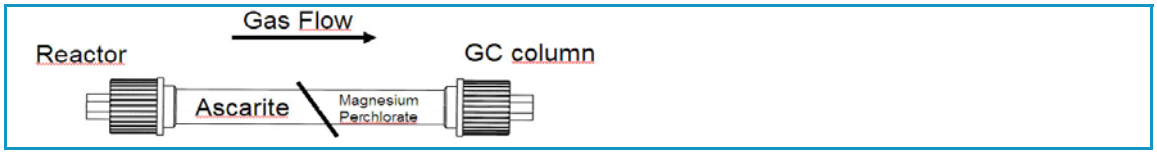
High-Temperature Conversion – Analysis of H and O Isotopes

The separation column is installed in the GC Oven of the EA together with the TCD. During analysis the temperature should be kept in a range between 70 - 90 °C. Nitrogen containing samples may be run at low temperature using dilution at the elution of the N_2 prior to the CO

Note Bake-out at maximum temperature for at least 24 hours (plus carrier flow for both analytical sides) must be considered when separation of the peaks is not sufficient any more or if $\delta^{18}O$ precision, accuracy, or both are deteriorating.

To achieve longer maintenance periods, the pyrolysis side has a trap filled with **Carbosorb**® (e.g. **Ascarite**® P/N 338 35236) and **Magnesium Perchlorate** (P/N 338 21900). These chemicals remove CO_2 and water from the carrier gas at standby temperatures of the reactor minimizing the bake-out intervals. The filling of the trap is given in Figure 102 using quartz wool at top and end, and to separate the filling material.

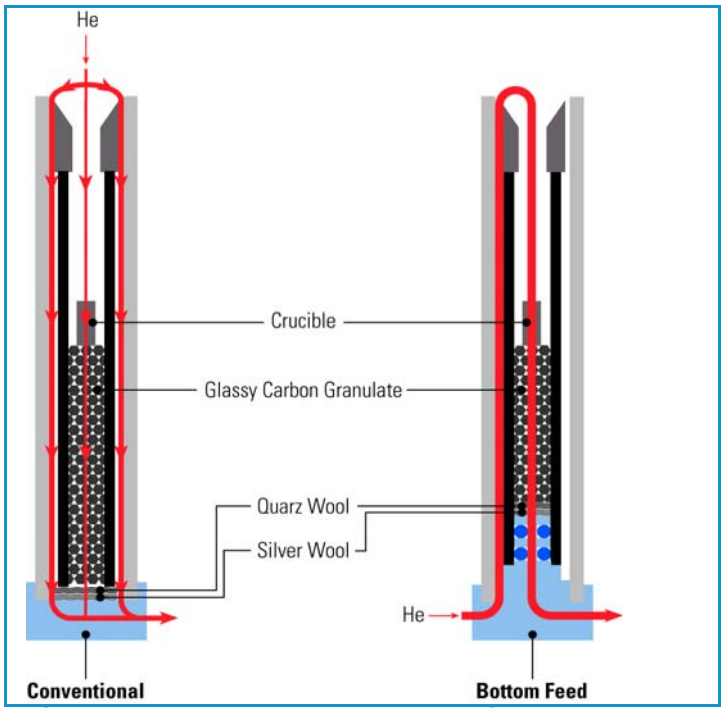
Figure 102. Trap Pyrolysis



Bottom Field Connector

The EA IsoLink IRMS System for CNSOH is equipped with a bottom feed connector for high temperature conversion analysis, also referred to as pyrolysis. It allows high sample throughput. This connector supplies the carrier gas helium from the bottom into the reactor. The helium flows up and is redirected downwards through the glassy carbon tube where it exits at the bottom connector again. See Figure 103.

Figure 103. Flow Path with Conventional and Bottom Feed Connector



Inserting the Reactor with a Bottom Feed Connector (BFC)

❖ To insert the reactor with a bottom feed connector

1. Loosely screw the nut with washer and o-ring on the BFC. Insert the ceramic tube from top. Fix it in this position using the upper o-ring.
2. Insert the bottle brush in the glassy carbon tube and introduce it in the ceramic tube from top. Use your other hand to carefully slip it over the o-rings of the BFC while keeping the ceramic tube up. Hold it tight while removing the bottle brush.
3. Now unscrew the nut and slip it with washer and o-ring over the ceramic tube at the bottom. Fix it using the steel rod and a 32 mm wrench.
4. Slowly insert the granules into the glassy carbon tube from top. Make sure no granule blocks the tube. Check the height with the mark on the graphite crucible remover tool by placing the crucible on the crucible remover and inserting it into the glassy carbon tube. The distance from the top of the crucible to the rim of the ceramic must be **270 mm**. Here is the hottest zone of the furnace.
5. Drop the crucible into the reactor and introduce the insert on top of the glassy carbon tube.

Removing the Reactor from the Bottom Feed Connector (BFC)

❖ To remove the reactor from the bottom feed connector

1. Wait to cool down.
2. Put in sample dilution in the ConFlo, or close the needle valve at the mass spectrometer. Stop the carrier gas flow through the pyrolysis system. Wait for the system to de-pressurize (3-5 minutes).
3. Remove the autosampler for liquids.
4. Unscrew the BFC using the steel rod and a 32 mm wrench.
5. Put on lint free gloves. Remove upper graphite tube insert from inside the ceramic tube.
6. Insert the bottle brush into the glassy carbon reactor.
7. Lift the ceramic tube with one hand and get a finger underneath the glassy carbon tube keeping the ceramic tube up with the same hand.
8. Slip the glassy carbon tube from the o-rings of the BFC while carefully pulling the tube with the bottle brush from top.
9. Remove o-ring, washer and nut at the bottom of the ceramic tube and take it out.

Setting Up a HO Method

This section describes the dual measurement to determine hydrogen and oxygen from a single sample.

It contains the following topics:

Procedure Overview

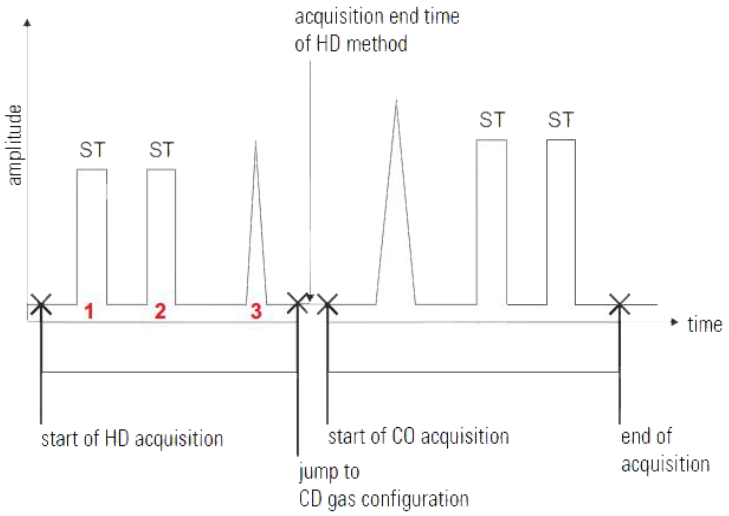
It is possible to perform dual measurements of hydrogen and oxygen from a single sample with the system (Thermo Chemical Elemental Analyzer, ConFloIV and IRMS) in less than 7min offering fast sample throughput and high productivity. The technique is suitable for both solid and liquid samples. Different autosamplers are used to measure different sample types, see Table 21.

Table 21. Dual Measurement and Sample Type

Sample Type	Autosampler
Solids and viscous liquids (for example, honey, serum, olive oil)	MAS 200, MAS Plus or others
Liquids, for example, water, alcohol, urine	AS3000, AS 1310, GC Pal, TriPlus 100 LS.

For the analysis of two isotopic species (hydrogen and oxygen) from a single sample, a method that comprises both must be defined. As soon as the hydrogen peak (**3** in Figure 104) has been identified, Isodat Software Suite stops the HD acquisition. The magnet jumps to the CO configuration (defined in the Switch Gas column in the time events list, see Figure 106 on page 98).

Figure 104. Schematic Chromatogram of a HO Dual Measurement



If no hydrogen peak can be found, Isodat Software Suite waits, for example, 25s. Data acquisition follows immediately after injection. See Figure 104.

Dual Measurement

Thermo Fisher Scientific assumes that the user already has working experience with the ConFlo IV interface and the IRMS. Thermo Fisher Scientific recommends to perform a simple check in order to test the analytical condition of the complete system before measuring any samples. Before the dual measurement can be started, some preparation are necessary.
Before running a dual measurement, the Peak Jump from H_2 to CO must be created. See "Performing a Jump Calibration" on page 85. If a jump is already defined, make sure the jump is re-calibrated regularly. It is recommended to perform a re-calibration on a daily basis.

Preparing the Hardware

❖ **To prepare the hardware components**

1. Set the reactor temperature to 1450 °C.
2. Set the GC column temperature to 90 °C. Set it to 70 °C if you are running N-containing samples.
3. Set the helium flow to >100mL/min.
4. Set the reference flow to 100 mL/min.
5. Make sure that the standard gas (H_2 and CO) on ConFloIV are available.

Determining the H3-Factor

The H3-factor is determined as described in the section "H3-Factor Determination" on page 107.

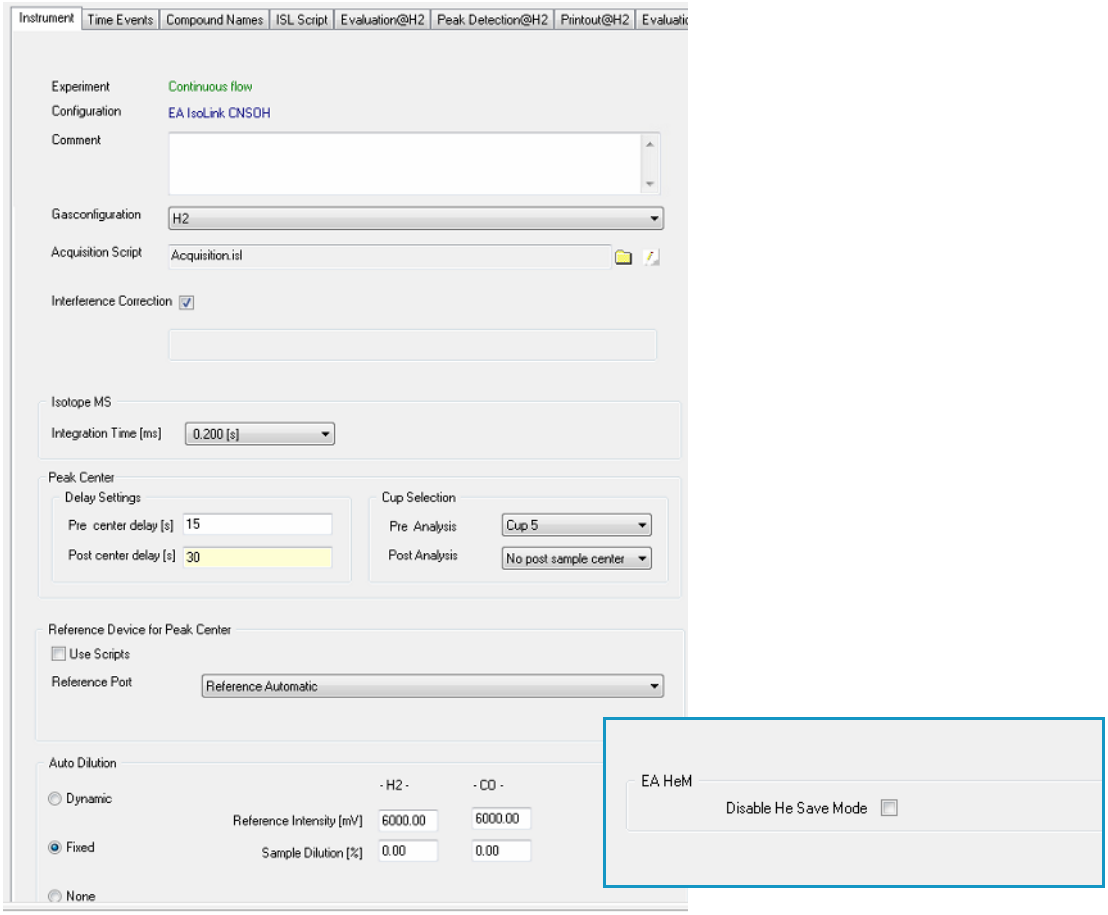
Creating a New Method for Dual Measurement

As a guideline for creating a new dual measurement method, the method HD_CO.met should be used.

❖ **To create a new method for an dual measurement**

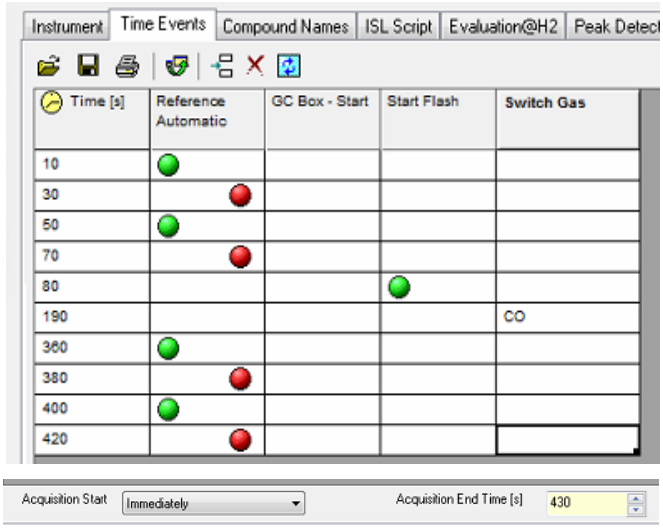
1. Open **Acquisition**.
2. Click **New** to create a new method.
3. In the **File New** dialog box, double-click **Method**.
4. Click the tab **Instrument** of the new dual measurement method, see Figure 105.

Figure 105. Instrument Page of Dual Measurement Method



5. Set the **Instrument** parameters for your new dual measurement method as shown in Figure 105.
6. Click the tab **Time Events**, see Figure 106.

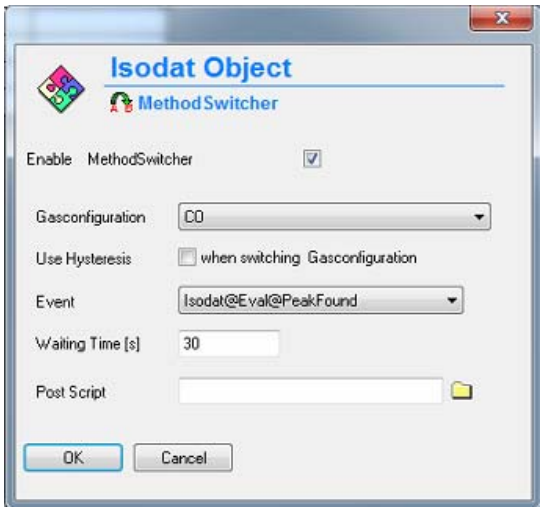
Figure 106. Time Events Page of the Dual Measurement Method.



7. Set the **Time Events** parameters for your new dual measurement method as shown in Figure 106.
8. Double-click on **CO** in the column **Switch Gas**. The Event Time is active since for example, 130s.

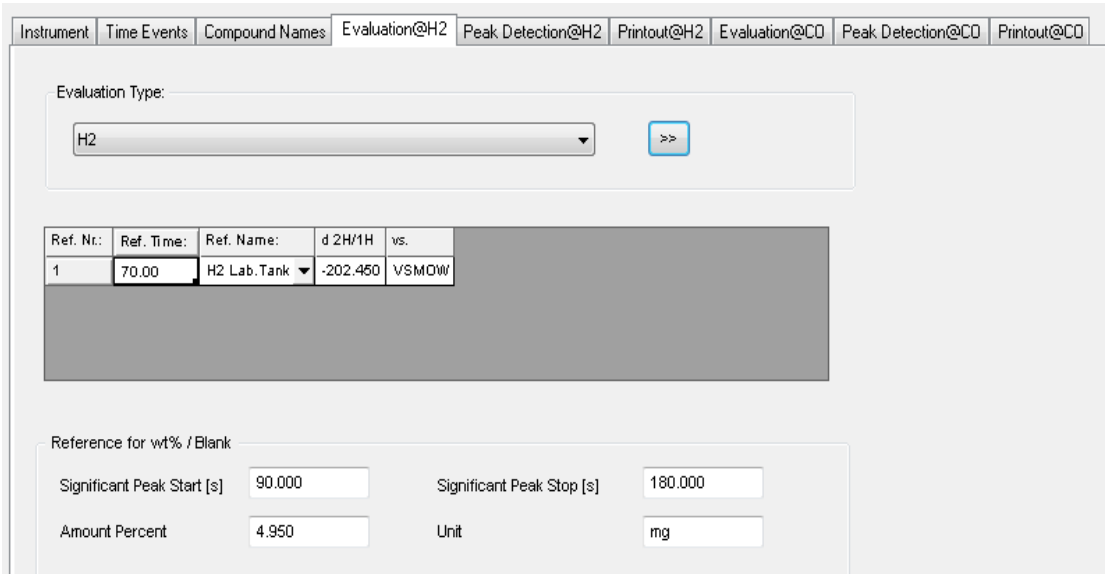
The **MethodSwicher** window opens, see Figure 107.

Figure 107. Switching Between Methods



9. For **Gasconfiguration**, select the gas configuration to be switched to, for example, **CO**.
10. Select the **Event** when the switch is to be performed, for example, **Isodat@Eval@PeakFound**.The switch is performed when the peak is found.
11. For **Waiting Time**, enter for example, after 30 s.
    If a peak is found, the jump takes place. If no peak is found, the waiting time elapses before the jump takes place immediately.
12. Click **OK**.
13. Click the tab **Evaluation@H2**. See Figure 108.

Figure 108. Evaluation@H2 Page of the Dual Measurement Method



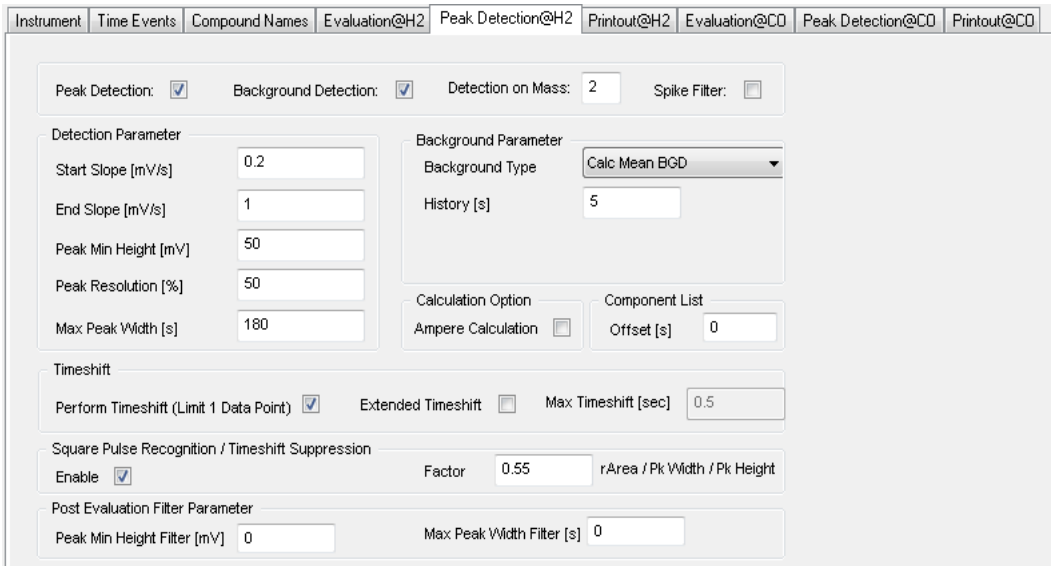
Note There are Evaluation pages, Peak Detection pages and Printout pages are for each, H_2 and CO, for example, Evaluation@H2 and Evaluation@CO2.

Note The values for reference gas and Amount% are examples and must be adjusted according to the isotope value of the reference gas and amount% of H in your sample. Please refer also to the *ConFlo IV manual, Chapter 4*, where the settings for reference/blank are described.

14. Set the **Evaluation@H2** parameters for your new dual measurement method as shown in Figure 108.

15. Click the tab **Peak Detection@H2**, see Figure 109.

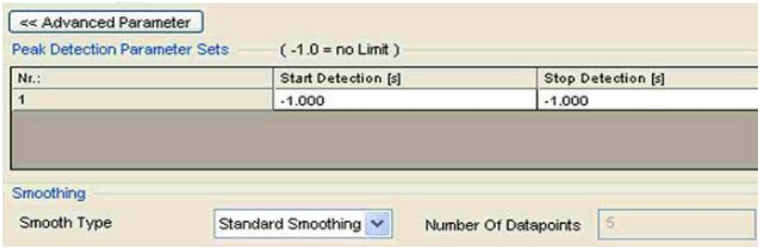
Figure 109. Peak Detection@H2 Page of the Dual Measurement Method



16. Set the **Peak Detection@H2** parameters for your new dual measurement method as shown in Figure 109.

17. Click **Advanced Parameter** to open the advanced parameter settings, see Figure 110.

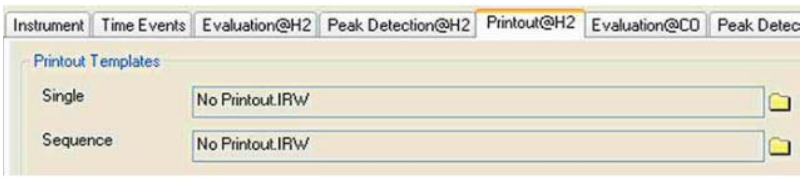
Figure 110. Peak Detection@H2 Page – Advanced Parameters of the Dual Measurement Method



18. Set the **Advanced Parameters** parameters for your dual measurement method as shown in Figure 110.

19. Select the **Printout@H2 tab** of your new dual measurement method, see Figure 111.

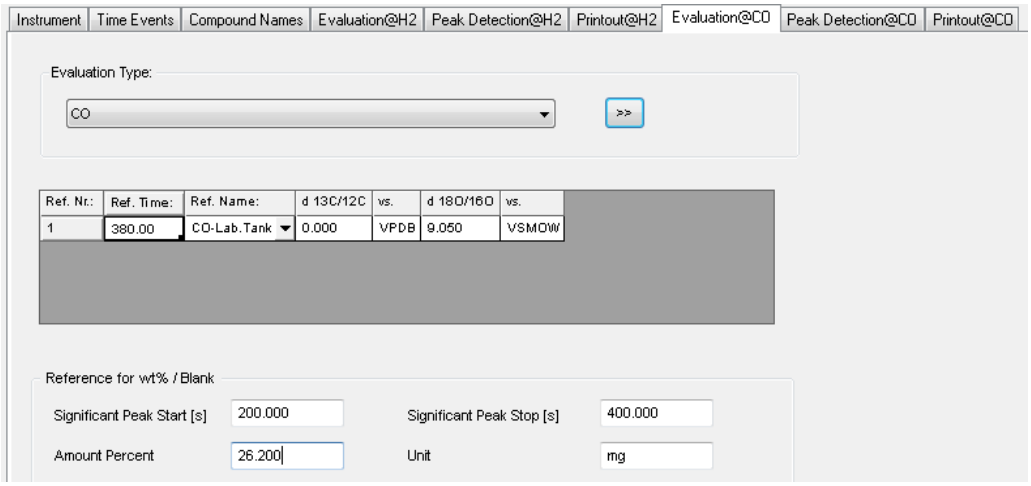
Figure 111. Printout@H2 of the Dual Measurement Method



Note Usually, the printout of results is not performed until the complete measurement has been finished. Therefore, it is recommended to choose **No Printout.irw** on the Printout@H2 page. Printout options are best defined at the end of the Printouts page.

20. Click the tab **Evaluation@CO**. See Figure 112.

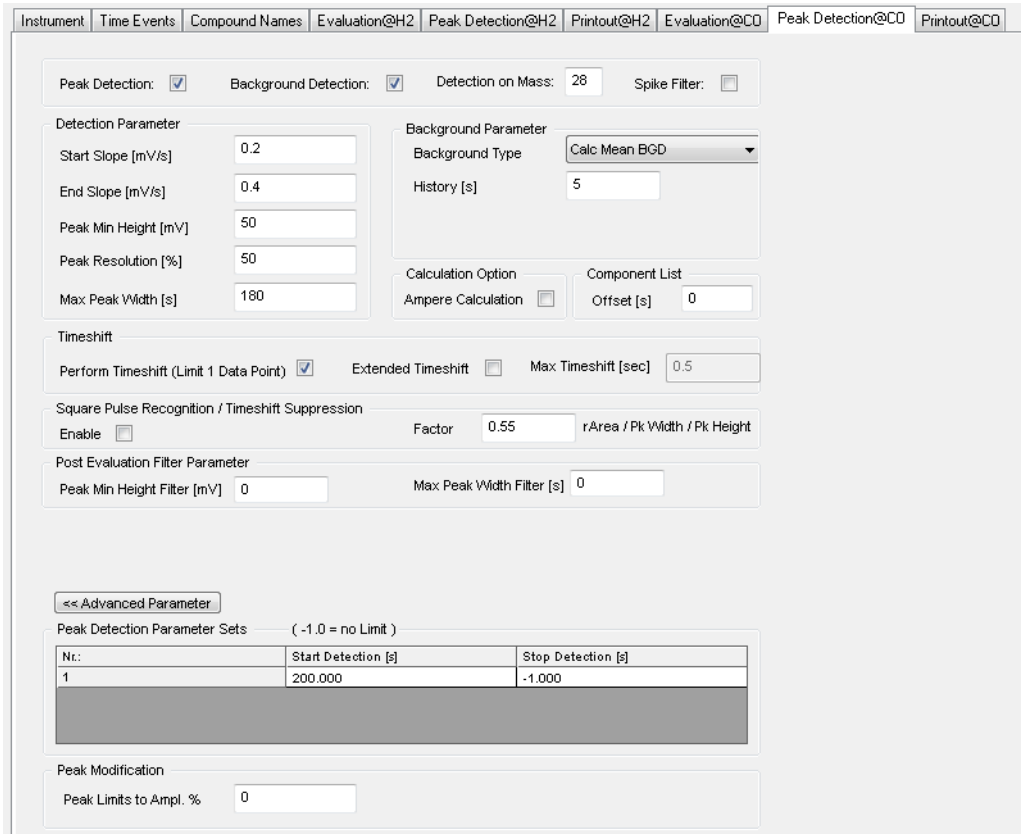
Figure 112. Evaluation@CO Page of the Dual Measurement Method



21. Set the **Evaluation@CO** parameters for your new dual measurement method as shown in Figure 112.

22. 3.Click the tab **Peak Detection@CO**, see Figure 113.

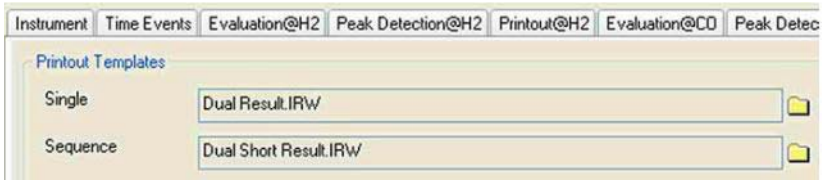
Figure 113. Peak Detection@CO Page of the Dual Measurement Method



Note The **Start Detection** time must be adjusted so that the spikes on the mass traces from the magnet jump are excluded. This may vary depending on the **Time Event List** setup.

23. Set the **Peak Detection@CO** parameters for your new dual measurement method as shown in Figure 113.
24. Select the **Printout@CO** tab of your new dual measurement method, see Figure 114.

Figure 114. Printout@CO Page

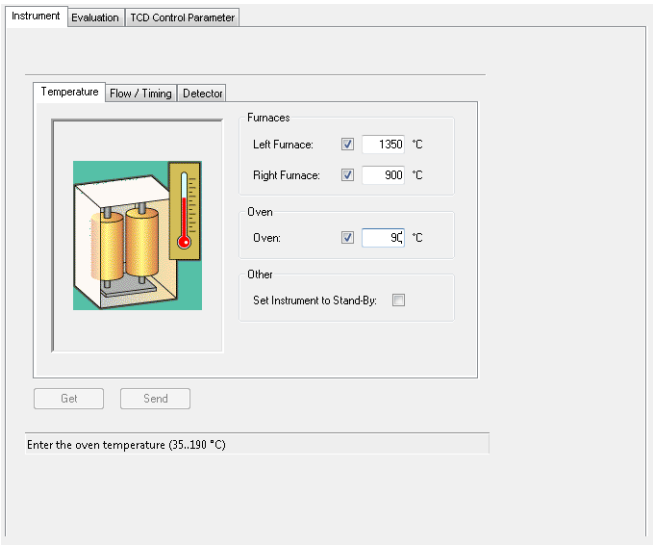


25. Select your Printout templates.
26. Click **Save | Save as** to save this method with the file ending *.met.30.
27. Enter a name for your method and click **Save**.

Creating an EA Method for H+O Measurement

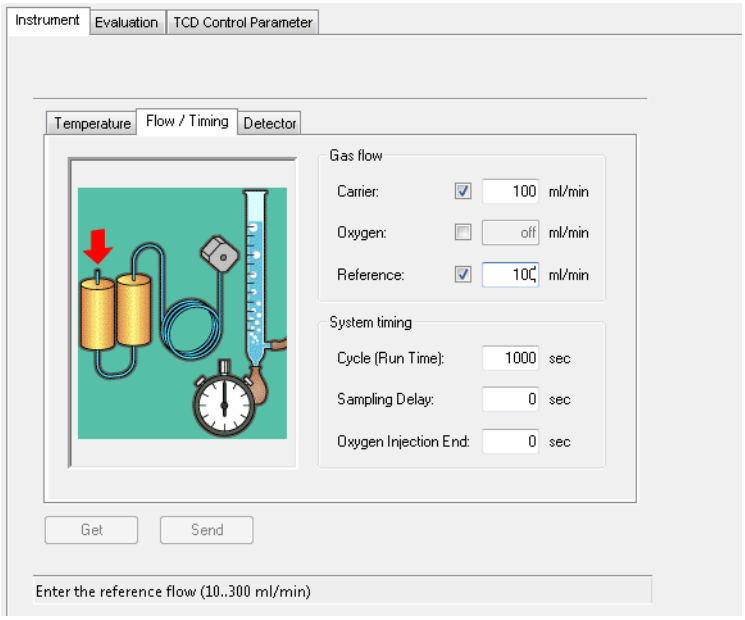
The following figures show how an EA-method for a dual analysis **H+O** may be set up.

Figure 115. HO EA Method: Instrument Temperature



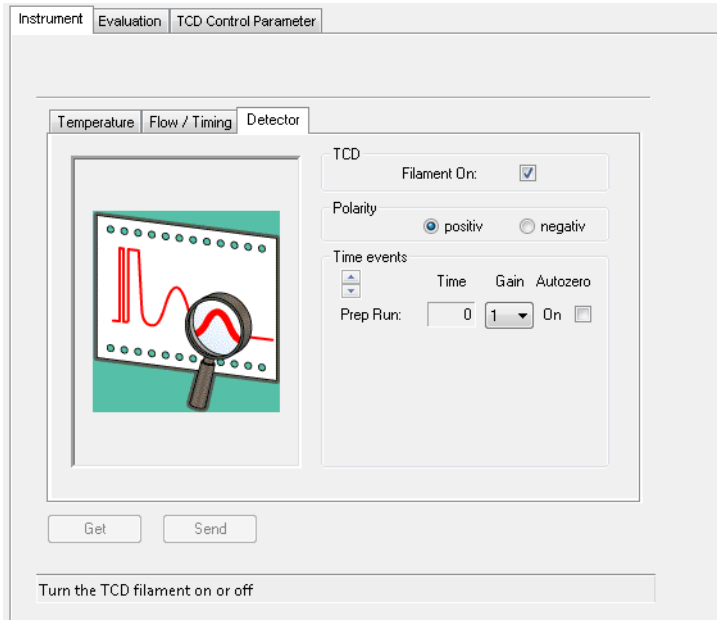
Note The temperature of the high-temperature conversion furnace (left furnace) may be different depending on the intended use of it.

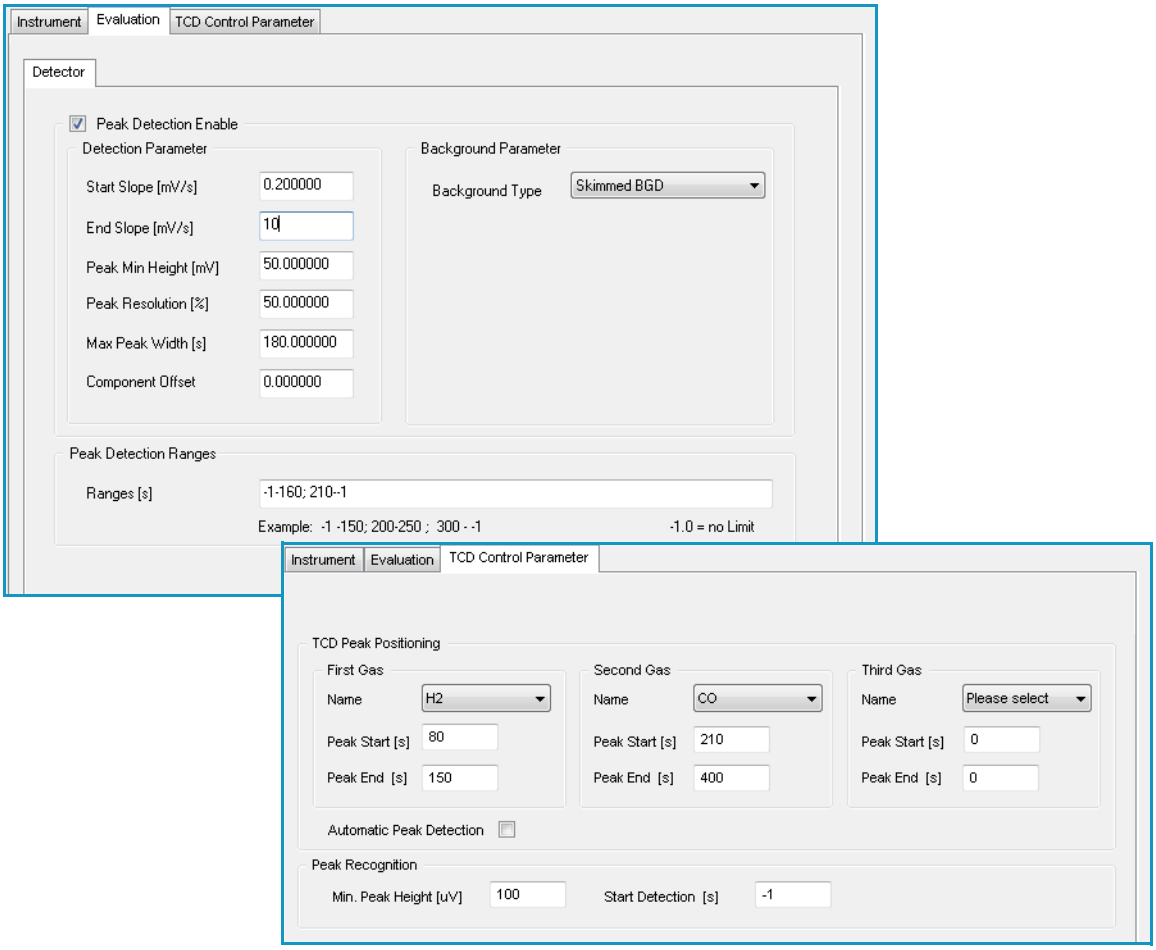
Figure 116. NC EA Method: Instrument Flow-Timing



Note Sampling delay and oxygen injection end vary depending on the sample nature. O_2 injection can be crucial in a single reactor if it exceeds the required amount.
The reference flow is used for the autosampler purge. This flow can be set to values between **20 mL/min** and **70 mL/min** (**150 - 200 mL/min in total when using He^M**), with the goal to minimize or eliminate the blank contribution of the autosampler.

For single mode N analyses, a second trap can be installed filled with **Carbosorb®** or **Ascarite®** to remove **CO_2**. The analysis time has to be reduced even further. In this case the entry Switch Gas **CO_2** can be deleted in the **Time Event List** of the **Isodat Method**.





Creating a Sequence for Dual Measurement

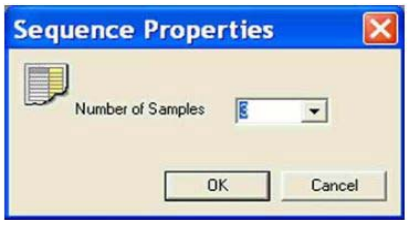
❖ To create a new sequence for dual measurement

1. Place a sample (for example, 0.281 mg of benzoic acid) in the solids autosampler.
2. Open **Acquisition**.
3. Click the **New** button to create a new sequence.The **File New** dialog box opens, see Figure 117.

Figure 117. Creating a New Sequence

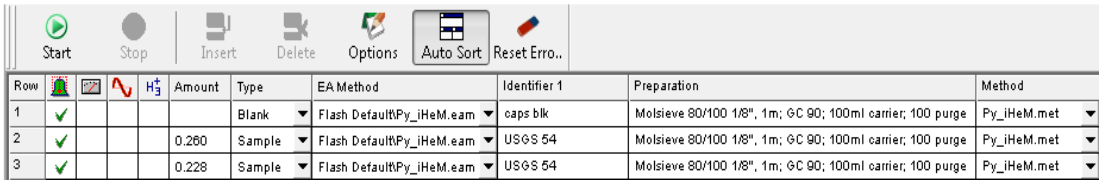
4. In the **File New** dialog box, double-click **Sequence**. The **Sequence Properties** window opens, see Figure 118.

Figure 118. Defining Number of Samples



5. In the **Sequence Properties** window, enter the **Number of Samples**, for example, 3.6.
6. Click **OK**. The sequence grid opens, see Figure 119.

Figure 119. Creating a Sequence for Dual Measurement



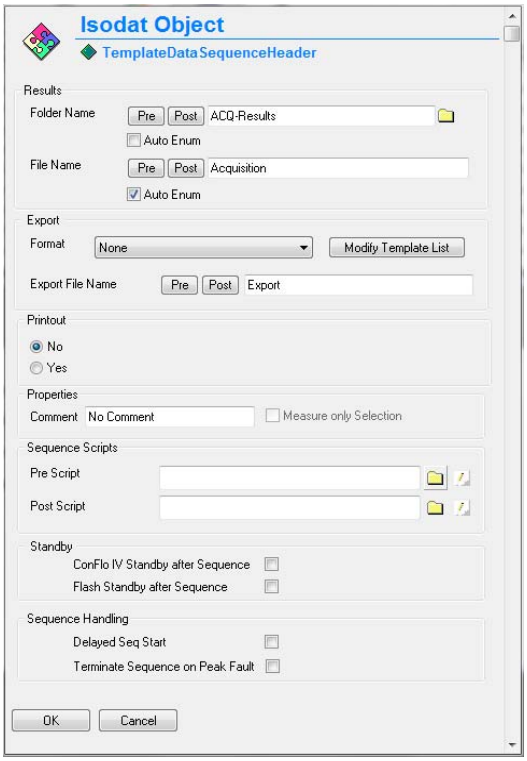
7. For each row, enter the **Amount** of the individual sample, for example **0.260**.
8. Enable ☑ to perform a **Peak Center** prior to measurement, see Figure 119.
9. Select an EA Method, for example **Py_iHeM.eam**.
10. Edit text in **Identifier 1** to identify the sample, for example benzoic acid.
11. Select **Method**, for example **Py_iHeM**.
12. **Save** the sequence with a name of your choice.
13. Continue with "Start Data Acquisition" on page 105.

Start Data Acquisition

❖ To start the data acquisition

1. In the Sequence window, click **Start** to start the sequence.The **Start Sequence** window opens, see Figure 120.

Figure 120. Defining Results Export and Printout Parameters



2. Select your destination **Folder Name** and **File Name** for the **Results**.
3. Select the **Format** for the export file.
4. Select **Printout**, for example, Yes.
5. Click **OK**.

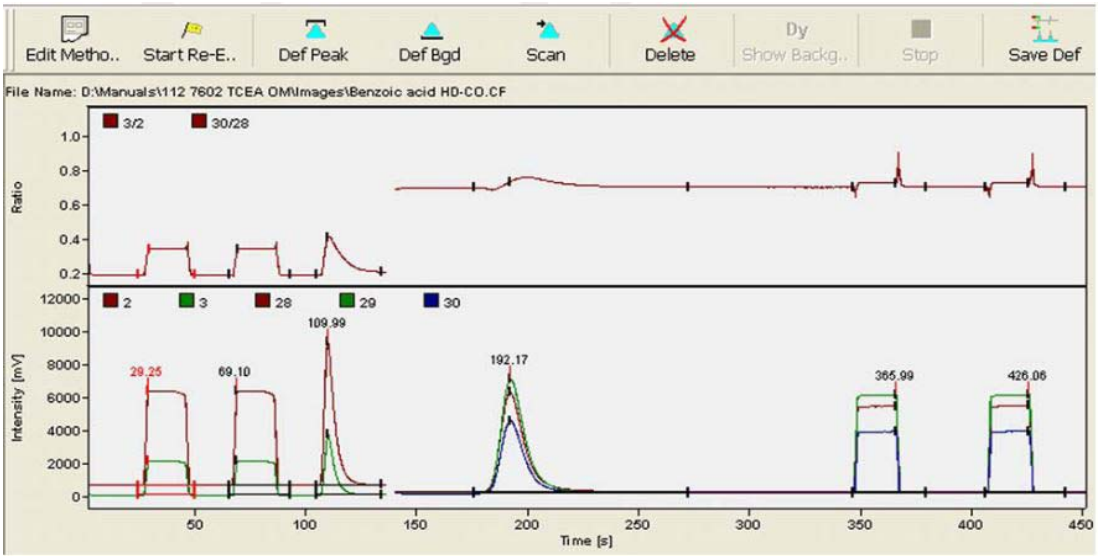
Dual Measurement Results

The following events occur during the acquisition:

- Peak center procedure is performed.
- The H_2 reference gas pulse is activated after 20 seconds for 20 seconds.
- The H_2 reference gas pulse is activated after 60 seconds for 20 seconds.
- The sample is dropped into the reactor.
- The H_2 sample peak appears approximately 15 seconds after reaction start. See Figure 121.
- The CO sample peak appears approximately 100 seconds after reaction start. See Figure 121.
- The CO reference gas pulse is activated after 340 seconds for20 seconds.
- The CO reference gas pulse is activated after 400 seconds for 20 seconds.
- The acquisition stops after 450 seconds.
- After finishing data acquisition, the printer creates a data output sheet as defined by the Result Workshop template (***.irw**) selected. The results are exported to a spreadsheet file if defined in Figure 120.

- The chromatogram and result grid are shown in Figure 121, Figure 122 and Figure 123:

Figure 121. Dual Measurement Chromatogram



The H_2 page displays information about the hydrogen-related peaks, see **Peaks Nr.** 1, 2 and 3 in Figure 122

Figure 122. Dual Measurement H_2 Result Grid

H2 | CO | Sequence Line | Error | Extended

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 2 [mV]	Ampl. 3 [mV]	BGD 2 [mV]	δ 2H [‰] vs. V-SMOW	AT% 2H [%]	Amt% [%]
1	24.9	29.3	25.4	5725	2053	637.6	-169.779	0.012929	-
2*	66.1	69.1	27.3	5723	2051	637.0	-169.895	0.012927	-
3	105.2	110.0	29.2	8695	3675	638.0	-75.440	0.014398	4.9000000

The CO page displays information about the CO-related peaks. See **Peaks Nr.** 4, 5 and 6 in Figure 123.

Figure 123. Dual Measurement CO Result Grid

H2 | CO | Sequence Line | Error | Extended

Peak Nr.	Start [s]	Rt [s]	Width [s]	Ampl. 28 [mV]	Ampl. 30 [mV]	BGD 28 [mV]	Area [Vs]	δ 13C [‰] vs. PDB	δ 18O [‰] vs. V-SMOW	Amt% [%]
4	176.3	192.2	96.6	6055	4434	218.8	80.238	12.284	29.314	26.2200000
5	346.5	366.0	33.5	5232	3784	225.0	99.372	0.571	9.649	-
6*	406.5	426.1	36.2	5252	3796	225.8	99.709	0.000	9.050	-

H3-Factor Determination

Protonation reactions in the ion source result in H3+-ion production. The H3+ portion of the **m/z** ion beam is determined as the H3-factor. The H3-factor is used to correct the H3+ contribution to the m/z3 signal. A low and stable H3-factor is needed for a good DH/H2 determination.

H3-factor determination is based on the peak area information of a “standard gas on/off chromatogram” (*.dxf) with standard gas pulses of different amplitudes. How to perform a H3-Factor Determination is explained in Chapter 3 of the *ConFlo IV Operating Manual*.

Measuring Sulfur Isotopes

This section provides the instructions for measuring sulfur isotopes.

Introduction

In comparison to isotope ratio measurements of nitrogen (^{15}N) and carbon (^{13}C) in organic and inorganic matter, the analysis of sulfur (^{34}S) has always been more challenging.

Analyzing biological sulfur makes experimental difficulties caused by the low abundance of sulfur in organisms (for example 0.2 wt% [mg/mg] in plants), as well as by the fact that sulfur is present as a mixture of organic and inorganic compounds.
Difficulties in using the Elemental Analyzer arise due to a large amount of carbon in the same samples, which quickly exceeds the capacity of combustion reactors. This causes incomplete combustion.

Thermo Fisher Scientific has developed a technique for precise, accurate and fast sulfur measurement, which puts it on a par with carbon and nitrogen in terms of ease of use and sample size.

Due to the high natural abundance of the heavier isotope ^{34}S, less amplification is required and may become necessary for all Delta IRMS before Delta^{plus} XP (since 2002).
It is recommended to use a smaller resistor ($1 * 10^{10}$ Ω) on the cup for mass 66 (usually $3 * 10^{10}$ Ω).

Procedure

Sulfur measurements are performed using a specially equipped Elemental Analyzer. Combustion and reduction are carried out in a single reactor filled with tungsten oxide (WO_3) and copper (Cu) as reducing agent.

The technique used for sulfur determination is based on the quantitative **Dynamic Flash Combustion** method. The samples - sometimes together with vanadium pentoxide (V_2O_5) - are wrapped in tin capsules and placed into the autosampler. Then they are continuously purged with helium to remove any traces of water and nitrogen. When a sample is dropped into the reactor, the helium stream is temporarily enriched with pure oxygen.
The sample and its container melt as the tin promotes a violent reaction flash combustion). Under these favorable conditions, even thermally resistant substances are completely oxidized.

In the reactor, for example barium sulfate ($BaSO_4$) is thermally decomposed within a tin capsule. The following reactions can then take place. See *Bailey, S.A. and Smith, J.W., 1972*):

$$BaSO_4 \rightarrow BaO + SO_2 + \frac{1}{2}O_2$$

$$BaSO_4 \rightarrow BaO + SO_3$$

$$SO_3 \rightarrow SO_2 + \frac{1}{2}O_2$$

Note *Bailey, S.A. and Smith, J.W. (1972)*: An improved method for the preparation of sulfur dioxide from barium sulfate $BaSO_4$) for isotope ratio studies. *Anal. Chem.* **44**, 1542-1543.

Although the process does not require oxygen (O_2), better combustion has been experienced when oxygen (O_2) is injected and vanadium pentoxide (V_2O_5) is added to the sample. If either the oxygen (O_2) pressure is low or a bad catalyst is selected or the reactor has too much ash, combustion will proceed slowly.

A slow stream of sulfur dioxide (SO_2) through the system causes adsorption at the tubing wall.

Note It is important that enough reduced copper (Cu) is present in the combustion tube.
If this is not assured, sulfur trioxide (SO_3) will only be reduced partially and isotope fractionation will occur.
Light isotopes ($^{32}SO_3$) are reduced more easily than **heavy** isotopes ($^{34}SO_3$). Therefore, sulfur dioxide (SO_2) gas is depleted in ^{34}S compared to the original sample, and the **δ** value becomes more negative.

Sulfur Measurement Kits

To perform a sulfur measurement your Elemental Analyzer needs to be modified. The required hardware is available in a kit. This kit contains amongst some helpful chemicals and spare parts:

- a dedicated reactor
- a dedicated separation column for S analysis
- a special tubing to minimize water adsorption

Table 22. Parts of Sulfur Measurement Kit for ConFlo (P/N 115 7100)

Quantity	Description	Part Number
1	Attachment for exhaust tube	112 1390
1	Self-adhesive heating foil for ConFlo	114 1180
1	Power supply for self-adhesive heating foil for ConFlo	204 8580

WARNING When working with sulfur dioxide (SO_2), good ventilation is essential. Otherwise, the gas can be hazardous to your health.

WARNING TOXIC SUBSTANCES HAZARD: To ensure operational safety, a CO, SO_2 and H_2 detector with an alarm must be installed. The exhaust tube should be installed on top of the ConFlo interface to remove the toxic sulfur dioxide (SO_2) from inside of ConFlo out of your working area. See Figure 5 on page 8.

Preparing the System for a Sulfur Measurement

❖ **To prepare the system for a sulfur measurement**

1. Ensure proper ventilation by connecting the exhaust to an outlet.
2. Ensure heating of the pressure regulator for SO_2.
3. Install the SO_2 GC column.
4. Installing the Self-Adhesive Heating Foil when using a ConFlo III.

Note SO_2 is liquid at higher pressures. Therefore, the manometer should be heated to avoid condensation of SO_2. The temperature must be between 60 °C and 70 °C. It may vary with ventilation.

a. Remove the backing paper from the self-adhesive heating foil.

b. Paste the heating foil in the middle of the manometer's rear side (of Ref 2).

5. Ensure the inlet heater and the ion source are heated.
6. Install the properly packed reactor within the Elemental Analyzer.
7. Replace all stainless steel tubing with Teflon®/Sulfinert® tubing to avoid water condensation in the system.

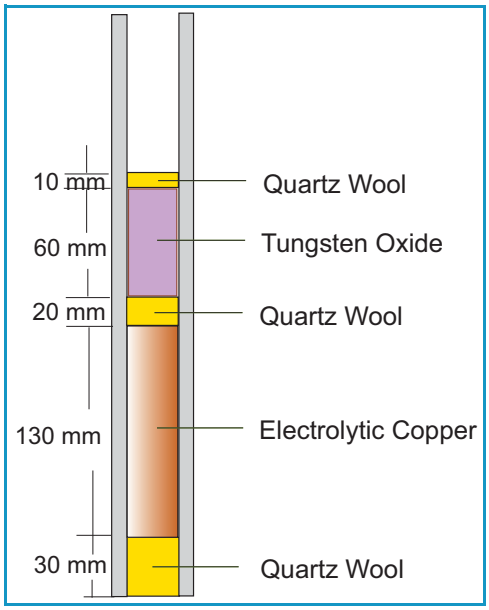
> **Note** Take care that the wires of the heating foil point downwards when pasting it on the manometer's rear side.

8. Simply insert the wires into the plug socket where they fix themselves.

Reactor Filling

We recommend to use a Thermo Fisher Scientific ready for use reactor (P/N 468 02021). The reactor is 470 mm long. If the reactor must be packed by the user, the packing should be as shown in Figure 124.

Figure 124. Packing of the Reactor



About **150** to **200** analyses can be performed using the reactor type described above.

Before Starting a Sulfur Measurement

We assume that the user already has working experience with the isotope ratio mass spectrometer. Before starting a sulfur measurement make sure that:

1. The Elemental Analyzer is set up, that is:
   - The SO_2 reactor is packed and installed.
   - The water trap is installed.
2. The special SO_2 stainless steel (or Teflon®) GC column is installed.
3. The connection between reactor, water trap, GC column and ConFlo is made up of Teflon® tubing.

4. The gases needed (He, O_2) are available and have been connected to the corresponding positions.
5. Leak-check all connections outside of ConFlo by brushing all fittings carefully with soapsuds.
6. The exhaust tube is connected to the ConFlo and ventilation is working.
7. SO_2 is connected to the ConFlo.
8. The pressure regulator for SO_2 is heated.
9. The IRMS has been calibrated for SO_2 measurements.
10. The Elemental Analyzer is switched on.
11. Adjust the gas pressure of Elemental Analyzer and ConFlo. Take the following settings as a guideline.
    - "Setting for Flash Elemental Analyzer" on page 111

Setting for Flash Elemental Analyzer

Table 23. Setting for Flash Elemental Analyzer in Eager*Smart* software

Carrier	180 mL/min
Oxygen	25 mL/min
Reference	20 mL/min
Cycle run time	1000 s
Sampling delay	10 s
Oxygen Injection End	3 s (or higher, depending on sample size and sample type)

12. Increase reactor temperature to 900 °C. After 10 minutes at this temperature, increase to 1020 °C. Alternatively, use the HeatUp function in the context menu of the Flash IRMS peripheral visualization
13. After 10 minutes at this temperature, increase to 1020 °C. Increase column temperature to 100 °C.
14. It is recommended to keep the reactor over night at end temperature to stabilize the conditions.

Note Teflon® tubings are not absolutely tight against atmosphere. Therefore, the backgrounds of argon and nitrogen are higher than those of carbon and nitrogen measurements. Nevertheless, take care of leaks.

Note When using reduced amplification (i.e. resistor of 1×10^{10} Ohm on cup for mass 66), the background values will be three times higher.

Note Background values may vary depending on sensitivity and focus settings.

Create a Gas Configuration for a Sulfur Measurement

A Gas Configuration determines a combination of masses, which are collected in the cups, for evaluation of ratios and eventually δ values. The Gas Configuration is specific for the particular gas and is combined with a magnet field value taken from the mass calibration of your IRMS. The ratio groups determine the reported ratios of predefined masses.

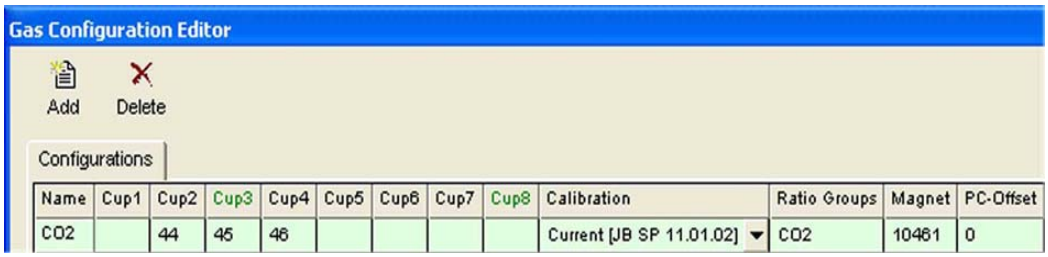
Prior to defining this Gas Configuration ensure that the connected IRMS has the cups for the simultaneous detection of masses 64 and 66 and mass calibration for these cups has already been performed.

For a ^{34}S measurement, a Gas Configuration must be available for the masses 64 (i.e. $^{34}S^{16}O^{16}O$) and 66 (i.e. $^{32}S^{16}O^{16}O$). Otherwise, it must be created as follows.

❖ To create a gas configuration for sulfur measurement

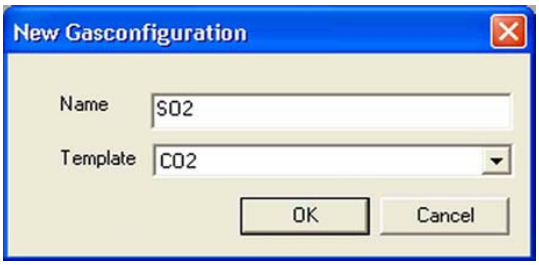
15. Open the **Acquisition** module. 

16. Open the**Gas Configuration Editor**. It is only available if no acquisition is running.



- Per default, the Gas Configuration **CO_2** is being created as the first one.
- If the Gas Configuration **SO_2** has already beencreated, it occurs in the list above.
- If the Gas Configuration **SO_2** has not been created yet, it does not occur in the list above. Then, proceed as follows.

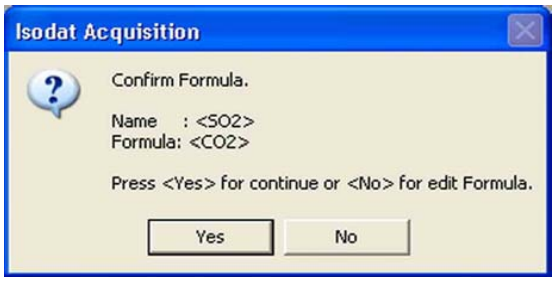
17. **Add** a new Gas Configuration. 



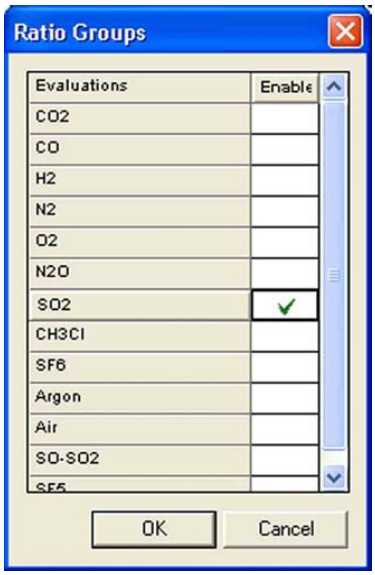
a. Type **SO_2** for the **Name**.

b. Select a Gas Configuration as **Template**, for example **CO_2**. In the pull-down menu, only the already existing Gas Configurations are displayed. When creating the first Gas Configuration, **CO_2** is displayed.

c. Confirm by **OK**.



18. Type **No**. If you would type Yes, this would automatically mark the template (i.e. **CO_2**) instead of **SO_2** in the **Ratio Groups** window below.



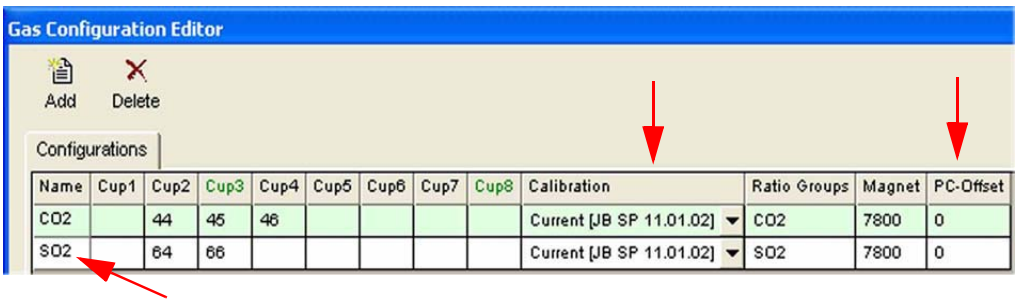
a. Mark **SO_2**.

b. If **Ratio Groups** other than **SO_2**are marked, clear them all.

19. Confirm by **OK**.

20. The new Gas Configuration **SO_2** appears in the list as a row of its own.

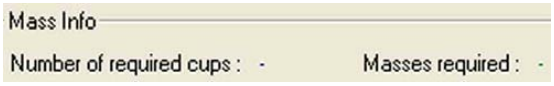
Figure 125. Creating a New Gas Configuration



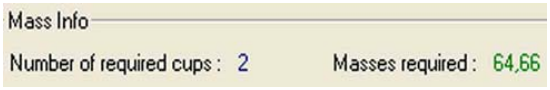
21. In the **Calibration** column select your current calibration file.

Note Figure 125 shows a common cup configuration as used in most Delta mass spectrometers, i.e. universal triple collector. If you have a special cup configuration, the respective masses will be collected in other cups.

22. Fill in the correct masses (64 and 66 replace for example 44, 45 and 46) to the appropriate cups specific for your IRMS.



When highlighting the specific gas configuration by a click on its row, the number of cups required for measurement is displayed together with the assigned masses.



23. Select a **Calibration**, which is valid for the selected cups.
24. Press the **Save & Close** button 

.

Note A value of -1 denotes unlimited

Starting a Sulfur Measurement

EA IsoLink Setting

See Table 24.

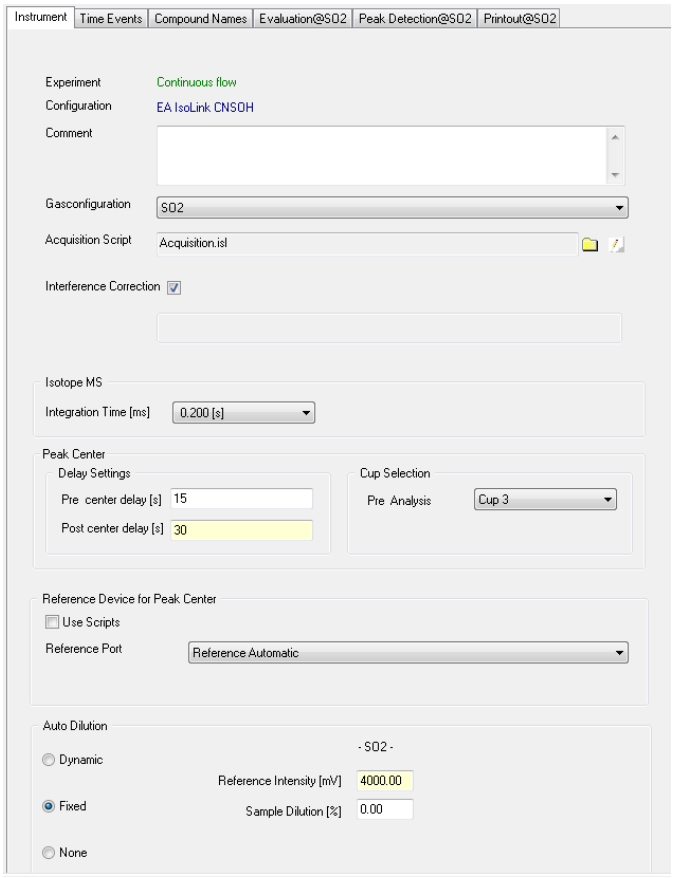
Table 24. Setting for EA IsoLink

Carrier	180 mL/min
Oxygen	25 mL/min
Reference	20 mL/min
Cycle run time	1000 s
Sampling delay	10 s
Oxygen Injection End	3 s (or higher, depending on sample size and sample type)

Defining an Isodat Method

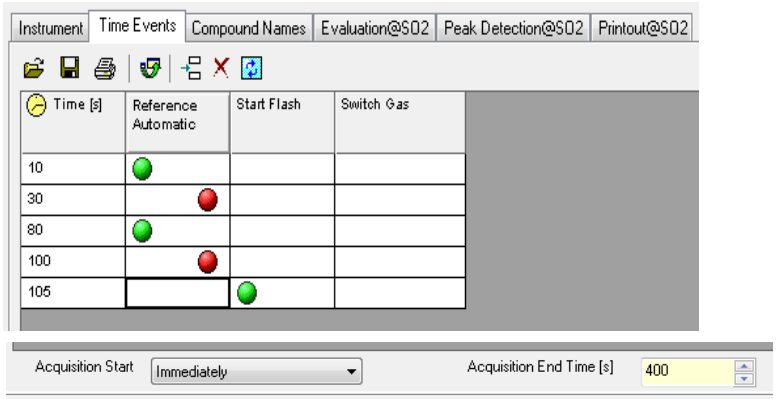
Instrument

Figure 126. Starting a Sulfur Measurement - Instrument Tab



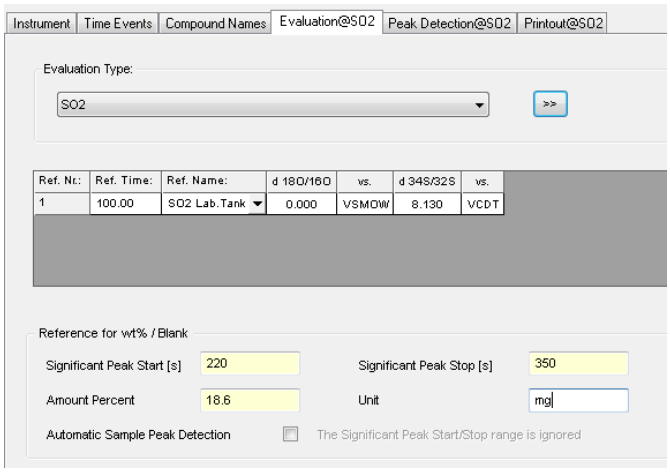
Time Events

Figure 127. Starting a Sulfur Measurement - Time Events Tab



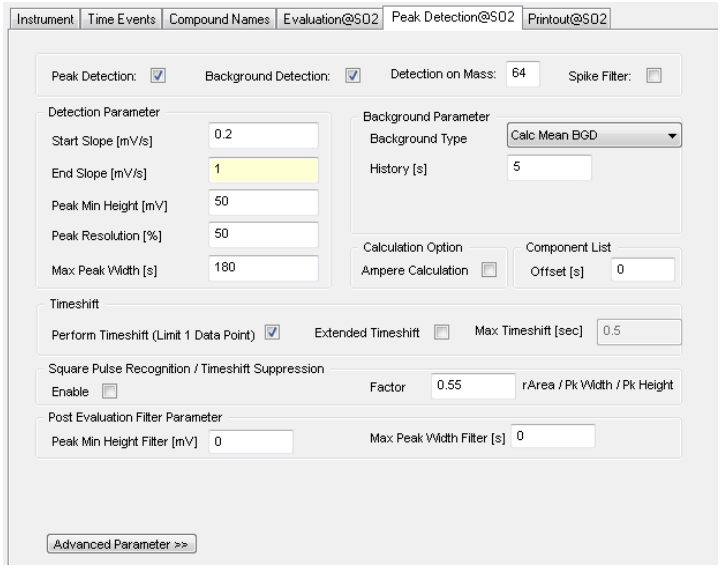
Evaluation

Figure 128. Starting a Sulfur Measurement - Evaluation Tab



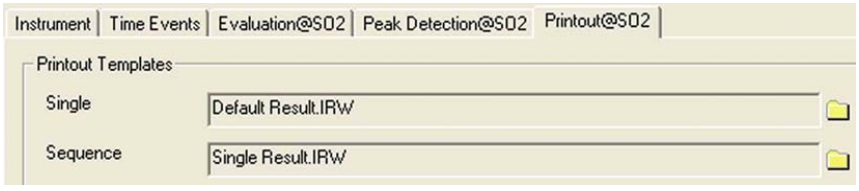
Peak Detection

Figure 129. Starting a Sulfur Measurement - Peak Detection Tab



Printout

Figure 130. Starting a Sulfur Measurement - Printout Tab



Events During Acquisition

See Figure 127 on page 115 and Figure 130 on page 116.

1. Peak center procedure.

2. First **SO_2 Reference Gas** pulse activated at **20 s** (duration: **20 s**).
3. Second **SO_2 Reference Gas** pulse activated at **90 s** (duration: **20 s**). It is assigned as Standard pulse for **δ** value calculation. See **Time** column in Figure 127 on page 115.
4. EA starts at **105 s**. At this time the oxygen is injected into the reactor.
5. Autosampler activated by EA after a sampling delay defined in the EA method, e.g. 10 s after oxygen injection.
6. Sample peak appears approximately **100 s** after Autosampler activation.
7. Acquisition stops at **400 s**.

7

Maintenance

This chapter provides information on the post-analysis operation, the operation sequences for the current and periodic maintenance of the instrument, and for maintaining the MAS Plus autosampler.

Contents

WARNING When, for technical reasons, it is necessary to work on instrument parts which might involve an hazard (moving parts, components under voltage, and so on), the authorized Technical Service must be contacted. This type of situations can be identified because access to these parts is possible only by using a tool. The removable protective covers bear a warning symbol suggesting to refer to the documentation accompanying the instrument. When a maintenance operation is performed, the operator must have received proper training to carry out specific actions.

WARNING When the instrument is switched off, consider that its does not cool down immediately, but heat tends to concentration in the upper part of the furnaces area. It should be made clear that it is better to cool down the furnaces first before switching off the instrument. Switching it off means that the fan in the back does not remove the hot air concentrating at the top and the surface thus becomes very hot. The openings provided for the chamber aeration will cause a slow cooling of the same, which however, in the vicinity of the areas marked with the symbol “hot surfaces”, might even reach temperatures higher than ambient temperature. Therefore in the minutes immediately following the instrument switching off, the operator must consider this risk and pay adequate attention during any maintenance operations following the use of the instrument.

Post-Analysis Operations

For maintaining high analytical performance and to reduce operating costs, we recommend you follow these practical suggestions.

- "Putting the Instrument in Standby Mode" on page 120
- "Maintaining the Instrument" on page 122
- "Maintaining the Instrument" on page 122

Putting the Instrument in Standby Mode

When the work session is over, the instrument can be placed into **Standby mode**. This mode is intended to reduce the gas consumption significantly and will reduce operating costs. The standby conditions can be defined by the operator.

There are these options:

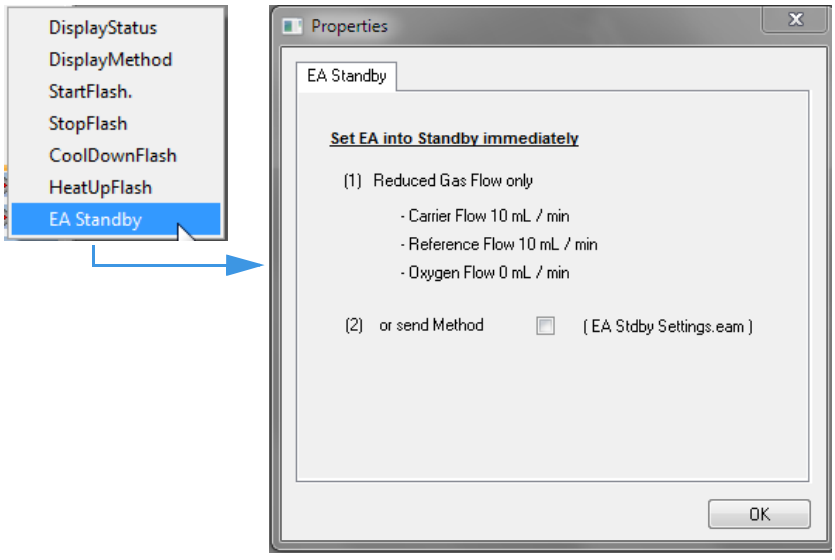
- **Reduce the flow only** — This option reduces the carrier and reference flow to 10 mL/min and the oxygen to 0 mL/min.
- **Define an EA method** — This option allows to define settings in an EA method which are uploaded to the EA.

The Standby function can be activated immediately or automatically at the end of the analytical sequence.

❖ To set the EA in standby immediately

1. Right-click on the EA window in Isodat Software Suite and open **EA Standby** options. When clicking this button it opens the following page. See Figure 131.

Figure 131. Standby option for EA



Here you decide which standby properties you want to use when setting the EA in standby.

- **Reduced gas flows (default)**

- **Upload user defined method** — This method can be found in the following folder: **C:\Thermo\Isodat NT\Global\User\ConFlo IV Interface\EA IsoLink Device\Method\Flash Default**

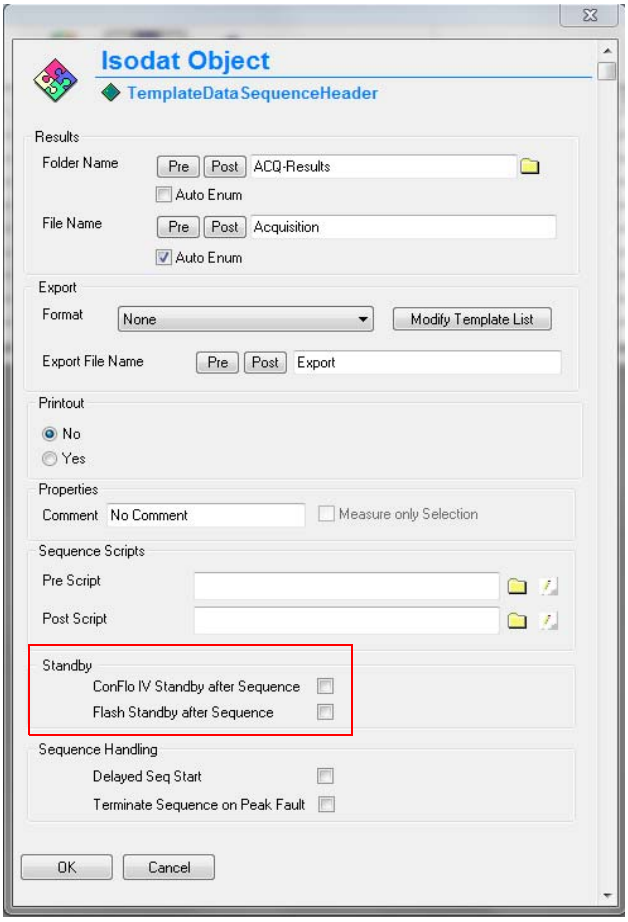
  The method can be modified to your purposes. Alternatively, it can be created a new by renaming the given EA file name (**EA Stdby Setting.eam**)

> **Note** If the method does not exist in the given folder Isodat Software Suite uses the default settings for the EA (reduced gas flow options)

❖ To automatically set the standby function after sequence end

In the Sequence Options of an Isodat Software Suite Software Suite sequence there are two boxes. See

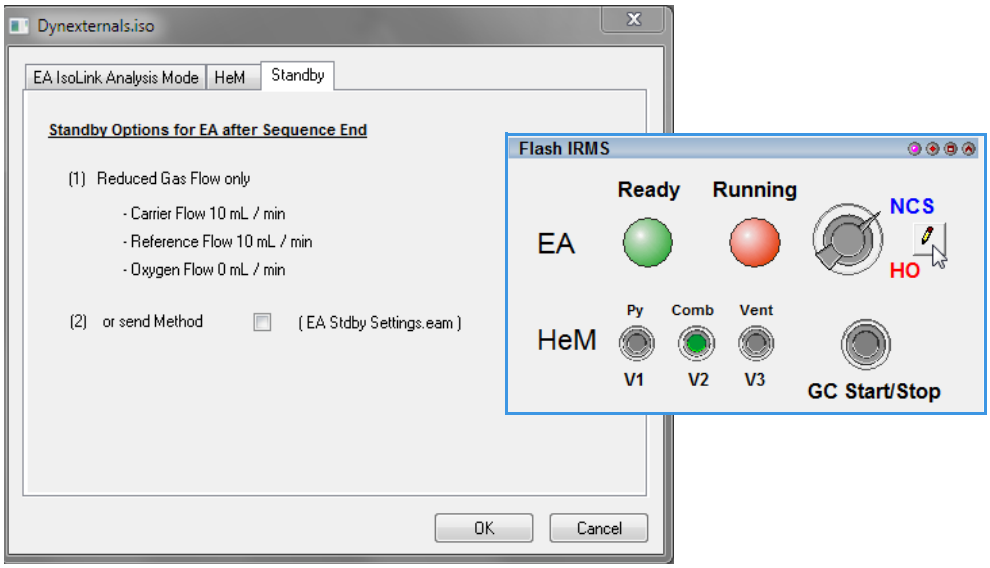
Figure 132. Sequence Standby Options



- **ConFlo IV Standby after Sequence** — Reduces the helium flow in the **ConFlo IV** to a minimum required to protect the ion source from ingress of air through the **Open Split**. The following action are taken:
  - The ConFlo is set into **LF Mode**.
  - The sample dilution is set to V1.
  - All reference gases are closed.
  - Reference dilution is set to zero.

- **Flash Standby after Sequence** — When selected, Isodat reduces the flows of the EA (default) or uploads the ***EA Stdby Settings.eam*** to the EA if the box is checked in the EA options. For this you must click on the options button in the Flash IRMS visualization and check the box under (2). See Figure 133.

Figure 133. DynExternals



Maintaining the Instrument

Note The instrument will be generally serviced by Thermo Fisher Scientific authorized technical personnel for all the warranty period or, after warranty, possibly according to a Programmed Service Contract. For more information contact your local Thermo Fisher Scientific Office.

- **Current Maintenance** — Replacement of reactors and adsorption filters and their filling materials. For instrument configurations using special steel reactors for combustion it may be necessary also to clean and remove the ashes from the crucible done with same material. See "Current Maintenance Program" on page 122.
- **Periodic Maintenance** — Replacement of the gas chromatographic column. The column lifetime in EA IsoLink IRMS System for CNSOH instrument is evaluated in years. Replacement of the seals of the reactors coupling unions placed on the furnace compartment base.

Note For some maintenance operations, furnaces and oven need to be at room temperature.

Current Maintenance Program

Each reactor, each filter and relevant fillings, need to be replaced according to the analytical configuration used. Please refer to the *CookBook* for the best analytical setup for your samples.

Replacing Reactors and Adsorption Filters

The replacement of reactors and adsorption filters is performed after a preset number of analyses according to the setting entered in the section "Replacing the Filling Materials" on page 123. Replace and install reactors and filters.

Replacing the Filling Materials

The replacement of reactors and adsorption filters requires the replacement of their filling materials. This operation comprises two steps. Alternatively, prepacked reactors can be obtained via your local Thermo Fisher Scientific Office.

- Removing the exhausted filling material from the reactor.
- Restoring the sequence of the layers of filling materials using new reagents.

Perform these operations according to the instructions given in the following operating sequences.

- "To replace the filling material in quartz reactors" on page 123
- "To replace the filling material in adsorption filters" on page 124

❖ To replace the filling material in quartz reactors

Material required
Tool for cleaning quartz reactors P/N 27606010 (included in the Standard Outfit of all EA IsoLink IRMS System for CNSOH Configurations)
Filling materials depending on analytical setup. Consult the *CookBook* for different reactor setups.

CAUTION Before starting the operation, check that the furnaces are at room temperature.

Remove the quartz reactor from the furnace, then do the following:

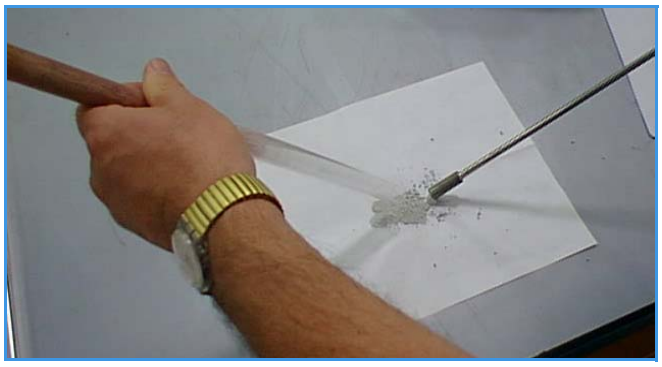
1. Introduce the cleaning tool into the reactor as shown in Figure 134.

   Figure 134. Removing the Filling Material from a Quartz Reactor

2. Rotate the tool exerting a slight pressure to scrape off the filling material.
3. Collect the removed filling material as shown in Figure 135.
4. Repeat steps 1 and 2 until complete elimination of the exhausted filling materials.

Figure 135. Collection of the Material Removed from a Quartz Reactor



5. At the end of the operation restore the layers of filling materials introducing into the reactor the new ones.

❖ **To replace the filling material in adsorption filters**

Material Required
Filling materials

Remove the adsorption filter from the detector compartment, then do the following:

1. Unscrew the filter nut and remove the filling material.
2. Restore the sequence of the layers of filling materials introducing the new ones into the filter.

Ashes Removal and Crucible Cleaning

❖ **To remove the ashes and clean the crucible**

CAUTION Perform the operation with the furnaces at room temperature.

Materials Required
Tool for cleaning quartz reactors P/N 27606010
Quartz wool

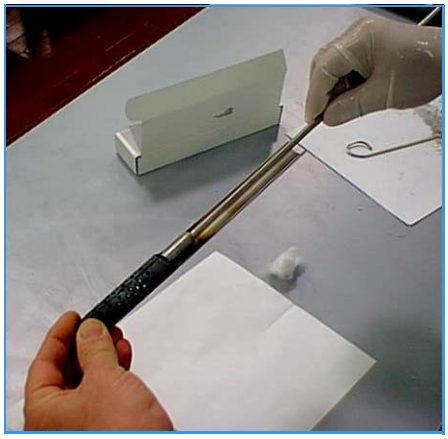
Remove the crucible from the reactor.

If the crucible is made of quartz, remove the ashes by using a spatula.

If the crucible is made of steel then do the following:

1. Introduce the cleaning tool into the crucible as shown in Figure 136.

Figure 136. Removal of Ashes and Quartz Wool from the Crucible



2. Rotate the cleaning tool exerting a slight pressure in a way to scrape off the ashes and quartz wool, then collect the material removed. At the end of the operation, introduce new quartz wool into the crucible.

Maintaining the Reactor of the Pyrolysis Unit

Note Please refer also to the section "Installing the Reactors into the Furnaces" on page 56.

❖ To clean the reactor

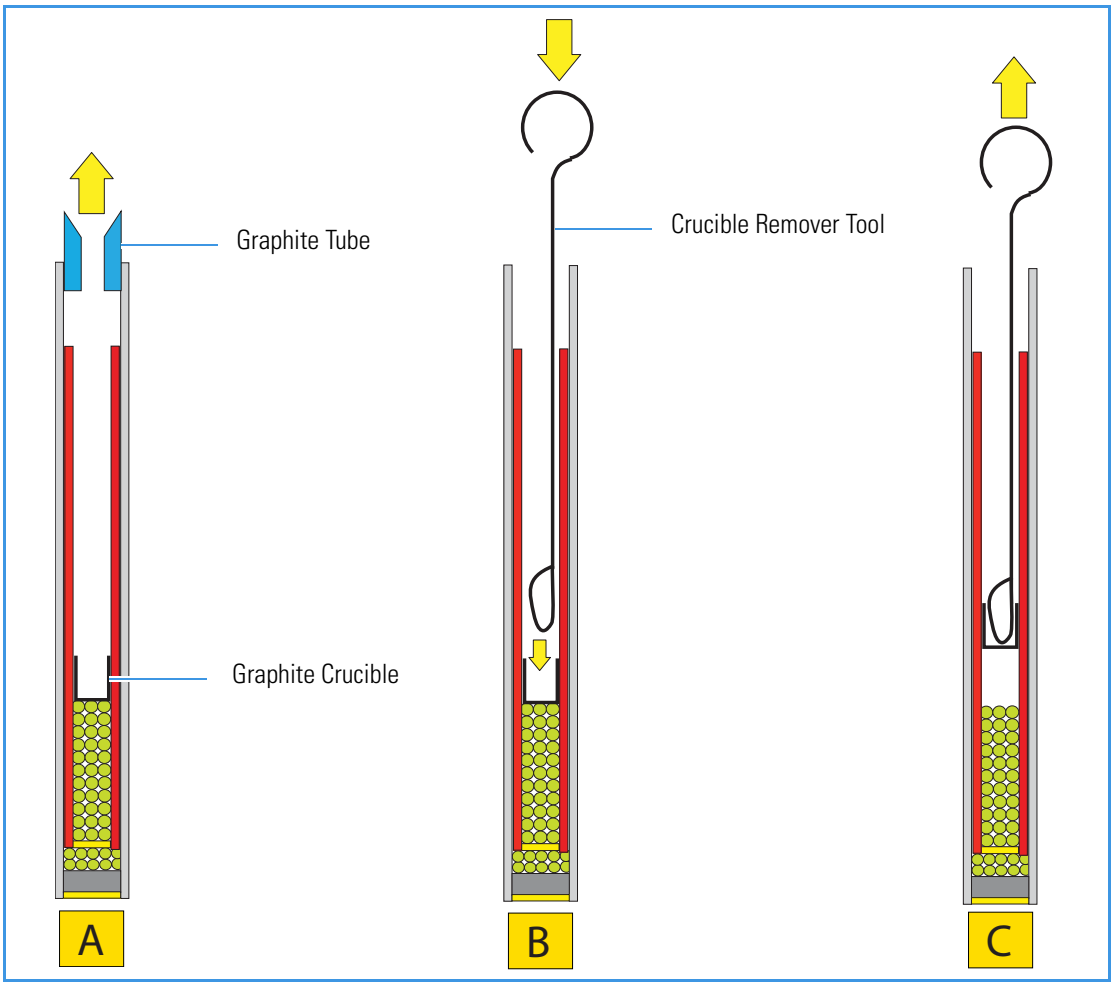
1. Take out the glassy carbon tube.
2. Remove the crucible, silver wool and quartz wool. Empty the granules in a separate bowl.
3. Clean the inside of the glassy carbon tube with the bottle brush.
4. Insert new quartz wool and silver wool.
5. Clean the granules with a tissue or a sieve until they are shiny again.
6. Remove all granules that are not shiny anymore or show a porous surface.
7. Clean the outside of the crucible with a tissue, and remove the molten silver inside or use a new one.

❖ To clean/replace the graphite crucible

Clean or replace the graphite crucible after approximately 400 analysis, according to sample nature and samples weight.

ATTENTION The use of gloves is strongly suggested. The graphite crucible is very hot after removal from the reactor.

Figure 137. Graphite Crucible Removal



1. Cool the furnace to a temperature below **600 °C**.
2. Put the ConFlo in standby or close needle valve to IRMS.
3. After reaching the temperature switch **Off** the flow of helium. Wait approximately five minutes to depressurize the system.
4. Remove the autosampler.
5. Carefully remove the graphite tube (for example with a pair of tweezers), see **A** of Figure 137, and place it on a clean cloth.
6. Insert the crucible remover inside the graphite crucible. See **B** in Figure 137.
7. Pull the graphite crucible out of the reactor. See **C** in Figure 137.
8. Drop a new graphite crucible into the reactor, or remove the deposits outside and inside of the graphite crucible and use it again.
9. Remount the parts proceeding in the reverse order.

❖ Maintenance measures for pyrolysis unit

To facilitate proper work, adhere to the described maintenance recommendations and procedures. This will also increases the life time of the pyrolysis reactor and the furnace heater.

CAUTION As the lifetime of the furnace heater is limited, do not heat it to higher temperatures than necessary. Put it to a lower standby temperature, when not in use for several days, or turn it off for longer idle times.

- To measure:

Type of analysis	Suggested operating temperature
Oxygen of organic samples	1325 °C - 1350 °C
Oxygen of inorganic samples	1450 °C
Hydrogen of organic samples	1450 °C
Hydrogen of inorganic samples	1450 °C
Water (sample amount > 1 µL)	1400 °C

- To cool down the furnace:
  If you do not want to perform any analysis **for about one day** (for example, over night), put the instrument in stand-by condition, and set the oven temperature to **150 °C**.
- If you do not want to perform any analysis **for more than one week**, put the instrument in stand-by condition, cool down both furnaces and oven to ambient temperatures.
- If the pyrolysis unit has not been in use **for a long time** or if it was **off**, heat the column to a temperature of **190 °C** for at least **24 hours**. Make sure to maintain a flow through both separation columns during baking out
- Always watch the background of the masses **28** and **40**. If the values exceed those you were used to, perform a leak test. A small leak will crack the ceramic tube, and after a while the glassy carbon reactor, too.
- After around **400** measurements (depending on size and type of the samples) clean or replace the graphite crucible as described in the "To clean/replace the graphite crucible," operating sequence. After cleaning the crucible may be used again. See also reactor maintenance.

Replacing the Gas Chromatographic Column

The instrument rarely requires the gas chromatographic column replacement, however, in case, operate according to the following operating sequence.

Note To replace the GC column in the ramped GC oven, please refer to the Ramped GC Oven Operating Manual.

❖ **To replace the gas chromatographic column in the GC oven of the EA.**

Material Required
Open end wrenches for the column fittings

1. Open the instrument right door and remove adsorption filters from their securing clips.

Note According to the instrument configuration, there can be one or two adsorption filters in the detector compartment.

2. Undo the 4 fixing screws securing the protection panel. See Figure 138.

Figure 138. Access to the Detector

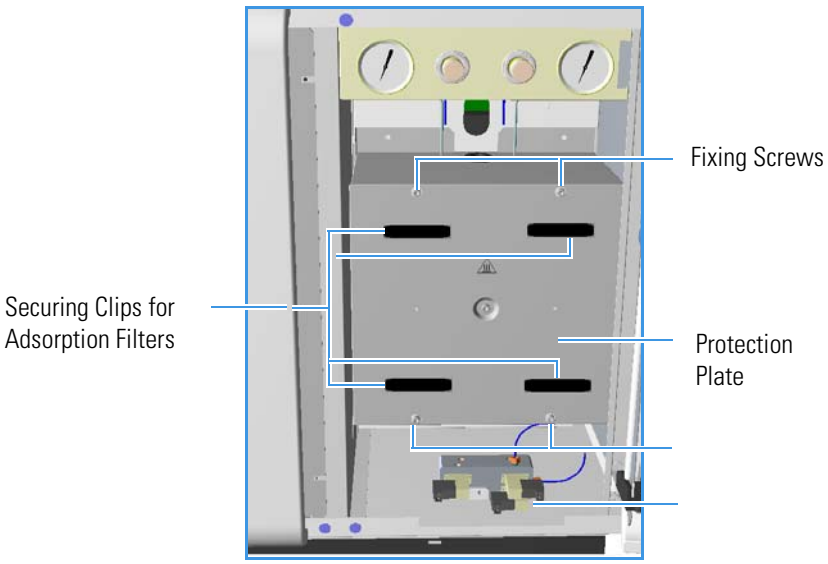
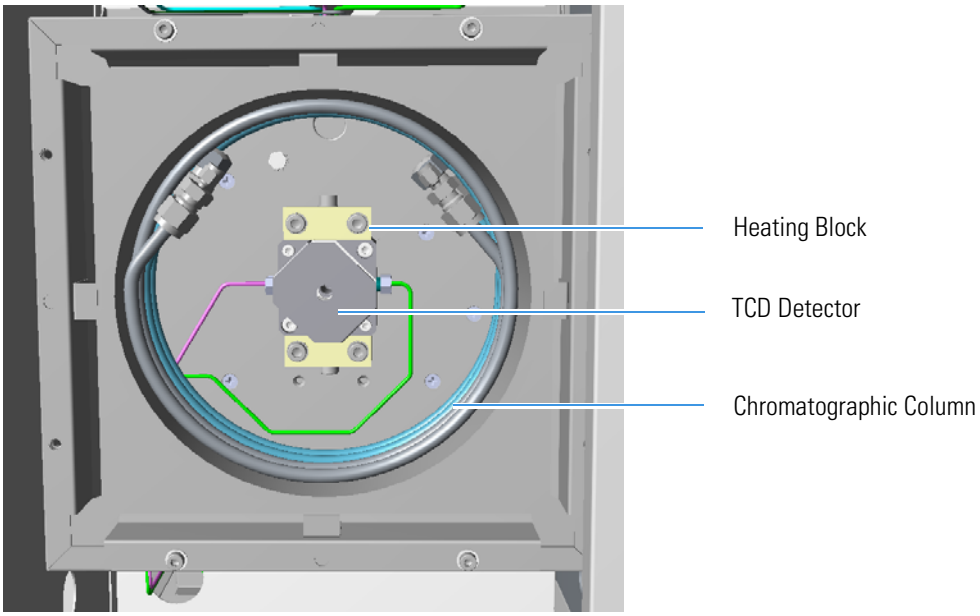


Figure 139 shows the detector compartment, the heating block where the detector is housed, and the gas chromatographic column.

Note According to the instrument configuration there can be one or two analytical columns.

Figure 139. Compartment Housing the Chromatographic Column



3. Unscrew the fittings from the column ends and remove the column from the compartment.
4. Introduce the new column and connect its ends to the fittings.
5. Re-mount the protection panel and the adsorption filters.

Maintaining the MAS Plus Autosampler

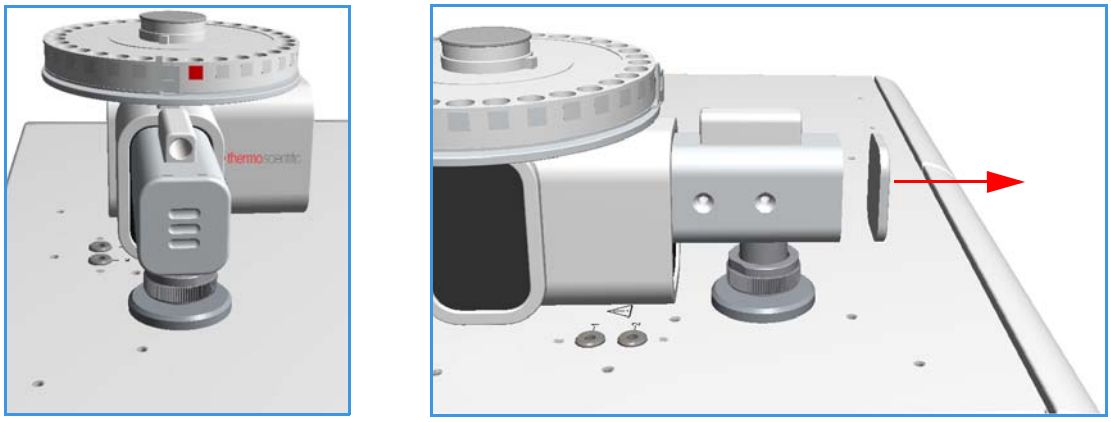
The MAS Plus autosampler does not normally require maintenance. However, when the instrument is extensively used, it is a good practice to clean from time to time the shaft housed in the autosampler operating as described in the following operating sequence.

❖ To clean the shaft of the MAS Plus autosampler

Materials Required
Phillips screwdriver
Cloth
Silicon grease (For use at pressures down to 10^{-6} mm Hg)

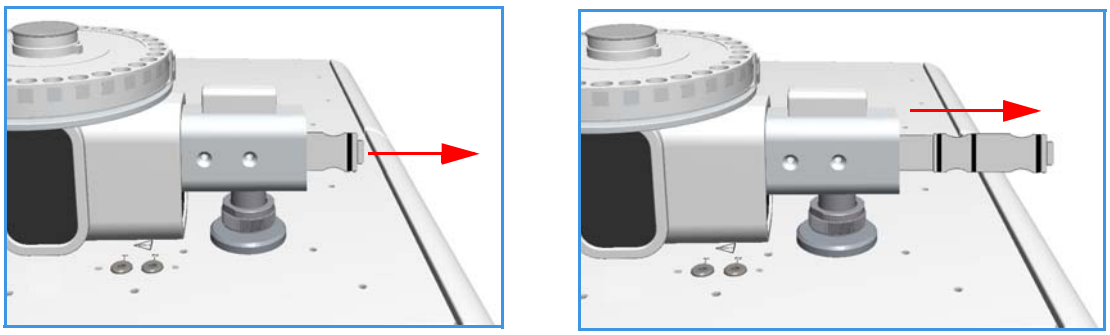
1. In Isodat Software Suite, right click in the Flash IRMS dialogue box and then select settings for EA.
2. In Eager*Smart* Data Handling Software Main Menu **Edit | Edit Elemental Analyzer parameters,** or just click the  icon. The **Analyzer Parameter** window is visualized.
3. Click **Send** and wait 2-3 minutes to discharge the gas out the circuit.
4. Remove the frontal cover of the MAS Plusautosampler as shown in Figure 140.

Figure 140. Removal of the MAS Plus Autosampler Cover



5. From Main Menu select the menu **View | View Elemental Analyzer status**, or click the icon  .
6. In the **Status** window select the **Special function** tag, then click **Step sampler tray position**. The autosampler mechanism pushes the shaft forward and then ejects it.
7. Take out the shaft from the autosampler, as shown in Figure 141.

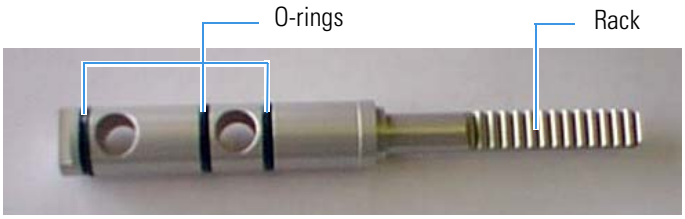
Figure 141. Removal of the Shaft



8. Eliminate possible traces of dirt from the shaft seals using a dry clean cloth.
9. Place and smear a slight layer of silicon grease on the o-ring. Do not use solvents.

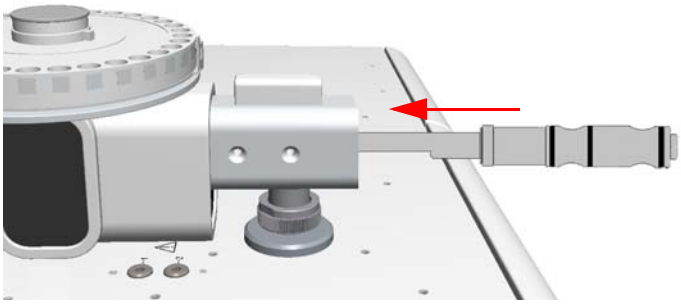
   Figure 142 shows the shaft of the MAS Plus autosampler.

Figure 142. Shaft of the Autosampler



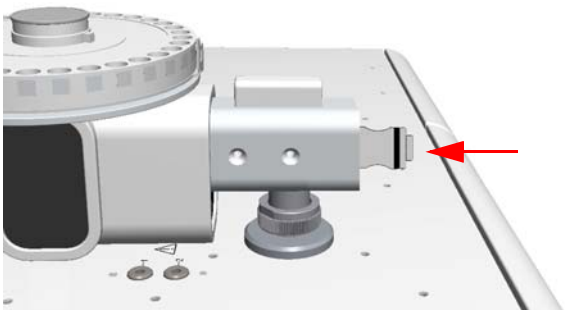
10. Re-introduce the shaft into the autosampler until in place keeping its rack turned downward, as shown in Figure 143.

Figure 143. Reinstalling the Shaft (1)



11. Slightly pushing the shaft with one finger of your hand, as shown in Figure 144, click **Step Sampler tray position** again. The autosampler mechanism first tries to eject the shaft, then, on the motor reversal, the mechanism will hook the shaft and draw it inside the autosampler (2).

Figure 144. Reinstalling the Shaft (2)



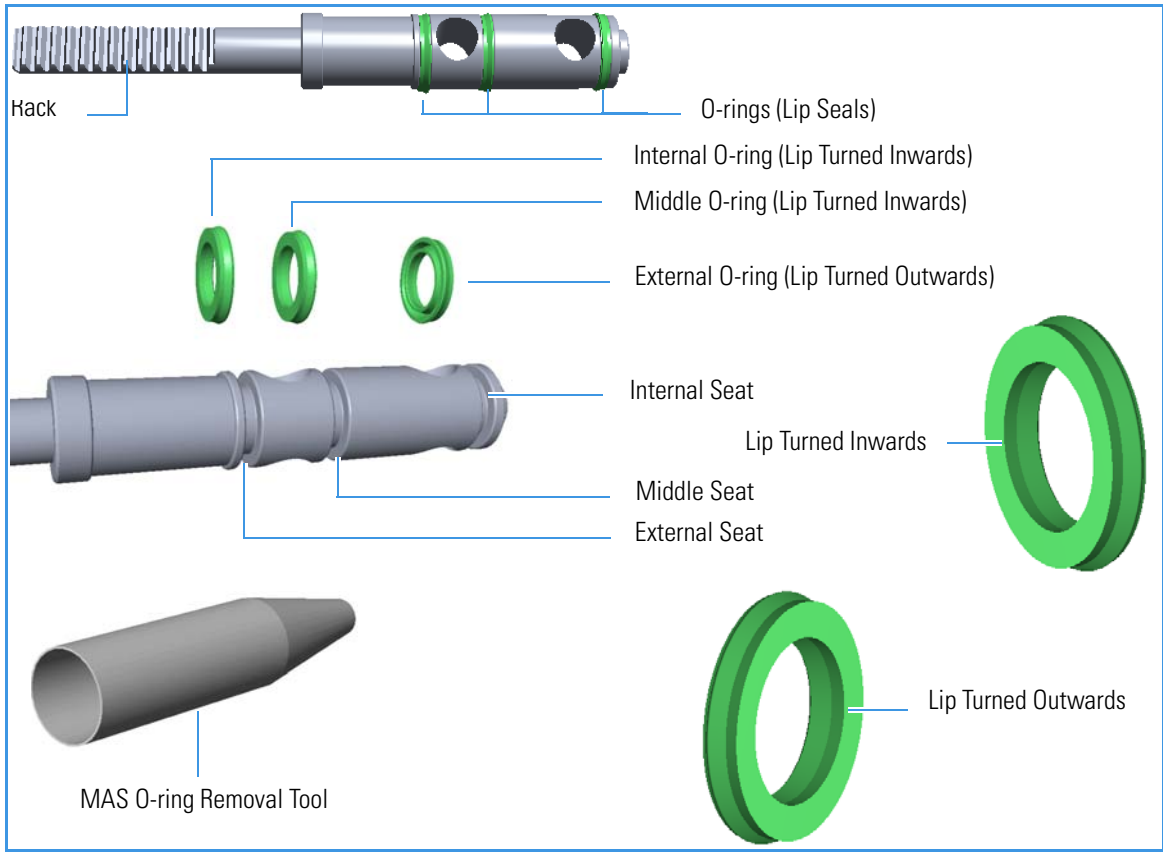
12. Re-install the autosampler front cover.
13. Return to **Analyzer Parameter** window. Restore the operating conditions setting the carrier gas flow to the initial value.

❖ Replacing the o-rings of the MAS shaft

Materials Required
Tweezers or spatula
Green O-rings package (set of 3) for MAS Plus shaft (P/N 290 30343)
MAS O-ring Removal Tool (P/N 205 02613)

The shaft of the MAS plus has three conical O-rings (lip seal) that must be replaced when worn-out.

The MAS O-ring Removal tool allows an easier replacement of the internal and middle O-rings into their relevant seats.

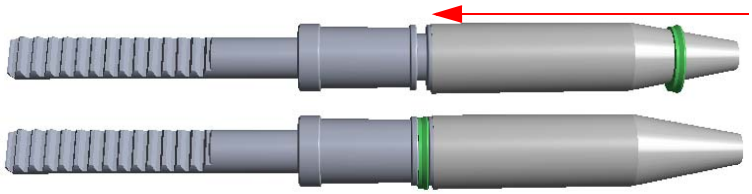


1. Remove the three from the shaft body by using tweezers or a spatula paying attention to not scratch the shaft.
2. Take the new green O-rings package and the removal tool.
3. Insert the first O-ring into the internal seat of the shaft.
   a. Place one of the three O-ring, with the lip turned inwards, on the conical section of the tool.

b. Move the O-ring along the tool up to reach the border.

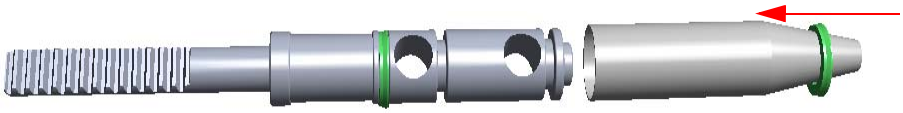
c. Move the tool on the shaft body up to reach the internal seat.

d. Move and inset the O-ring into the internal seat, then remove the tool.



4. Insert the second O-ring into the middle seat of the shaft.

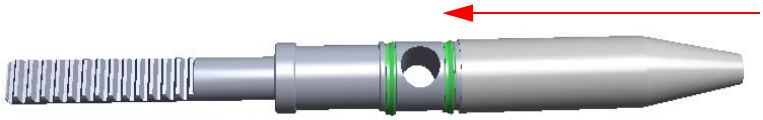
a. Place the second O-ring, with the lip turned inwards, on the conical section of the tool.



b. Move the O-ring along the tool up to reach the border.

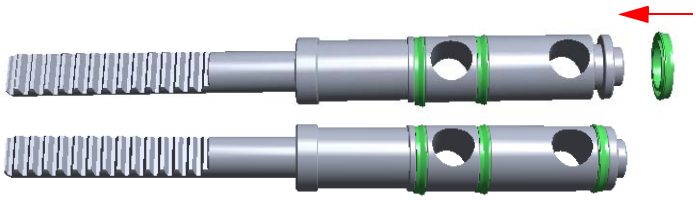
c. Move the tool on the shaft body up to reach the middle seat.

d. Move and inset the O-ring into the middle seat, then remove the tool.



5. Insert the third O-ring into the external cavity of the shaft. The use of the tool is not necessary.

a. Manually place the last O-ring, with the lip turned outwards, into the external seat.

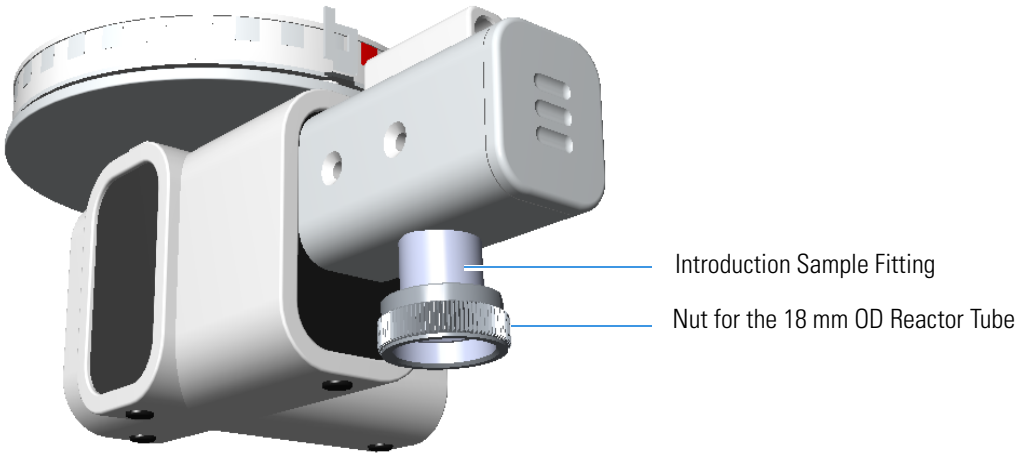


Upgrading EA for 25 mm OD Macro Reactor

This procedure gives the instructions for replacing the fittings for the 18 mm OD standard reactor with the fittings for the 25 mm macro reactor.

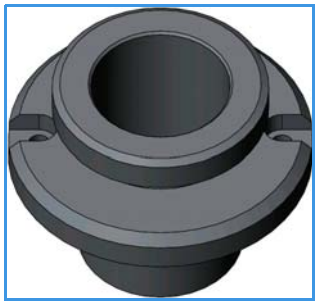
WARNING This operation must be carried out with the furnaces at room temperature.

1. If already installed, remove the MAS Plus and the reactor from the left/right furnace of interest.
2. Using the 23-33 mm open-ended wrench, unscrew and remove the introduction sample fitting with the nut for the 18 mm OD reactor (P/N 35008417) from the MAS Plus paying attention to do not damage the internal gasket.



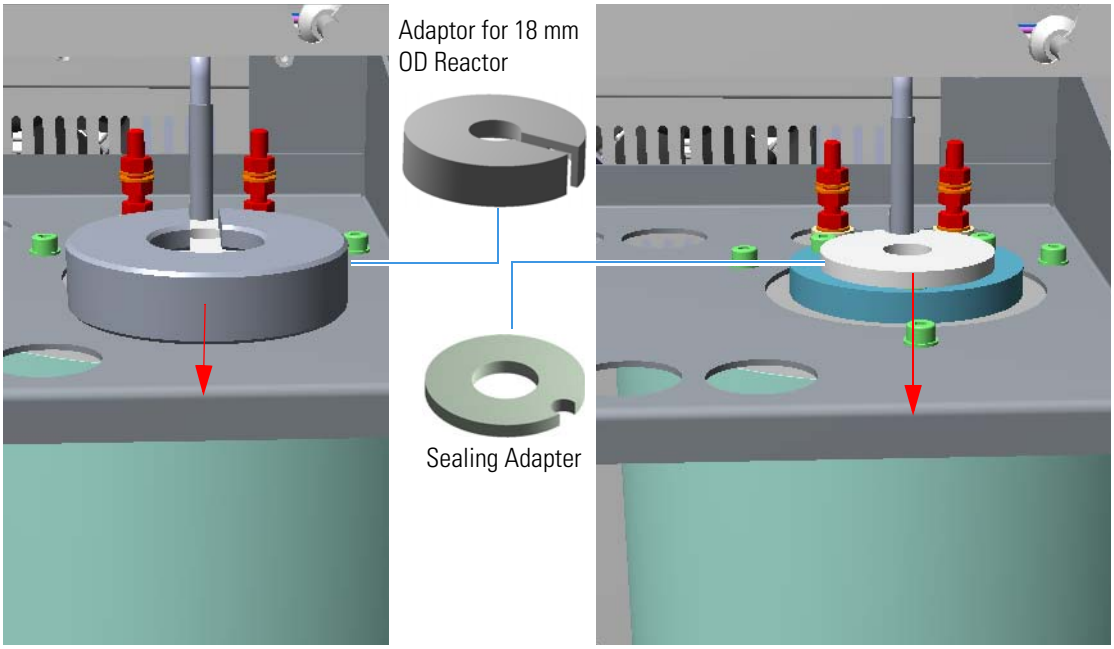
3. Screw manually the introduction sample fitting with the nut for the 25 mm OD macro reactor (P/N 35008418, provided in the MAS Plus standard outfit) into the MAS Plus. Tighten the fitting by using the 23-33 mm open-ended wrench.
4. Access the furnace compartment lifting the cover and removing the protecting plate.

5. On the top panel of the instrument remove the left/right upper connector for the 18 mm OD reactor unscrewing the two fixing Allen screws with the proper Allen wrench. Place and fix the upper connector for 25 mm OD macro reactor.



Upper Connector for
25 mm OD Macro Reactor

6. On the top of left/right furnace remove the adaptor for 18 mm OD reactor and its sealing adapter.



7. Reinstall the protecting plate and the cover of the furnace compartment.
8. Install the macro reactor tube into the furnace and reinstall the MAS Plus.

Cleaning the Instrument

❖ **To clean the instrument**

WARNING Cleaning must be performed with the instrument off, the furnaces at room temperature and the power cord disconnected.

1. Externally clean the instrument with a soap and water solution, or with a household non-abrasive product. Avoid that any liquid seeps into the instrument.
2. If you just suspect that a substance used for cleaning or a product submitted to analysis has infiltrated inside the instrument, immediately shut down the instrument and call an authorized customer support engineer for proper actions.The service engineer must be fully informed on the nature of the concerned substance.

WARNING It is your responsibility to avoid that dangerous liquids and/or materials seeping inside the EA IsoLink IRMS System for CNSOH during operation and maintenance.

If you have any doubt about compatibility of decontamination or cleaning agents with parts of the equipment or with material contained in, please contact your Thermo Fisher Scientific Representative.

Troubleshooting

This chapter provides information necessary to find out instrument troubles and to solve them.

Contents

Analytical Troubleshooting

If the instrument has been correctly installed, the gas characteristics are as required and maintenance has been regularly carried out, EA IsoLink IRMS System for CNSOH will provide correct results.

The lack of the above conditions will be indicated by anomalies in the chromatograms and the relevant analytical reports. Table 25 reports the most common anomalies with the relevant diagnosis and remedy.

Table 25. Analytical Troubleshooting Guide (Sheet 1 of 2)

Problem	Diagnosis	Remedy
High nitrogen blank.	Presence of leak.	Check that helium and oxygen lines are sealed and in case eliminate possible leak.
	Oxygen line or cylinder contaminated.	Purge for 10-20 minutes. Replace the contaminated cylinder.
	Autosampler not purged.	Check that the helium flow is correct.
High constant nitrogen blank in several sequential analyses.	Oxygen cylinder contaminated.	Replace the oxygen cylinder.
	Presence of leak in the autosampler system.	Identify leaks and remove them.
Carbon peak tailing or split.	Too much ashes inside the reactor.	Check ashes and remove them.
	The sample analyzed was too large.	Weigh a lower amounts of sample.
Hydrogen peak is a split peak.	The tube connecting reactor and column is clogged.	Cut off the clogged tube portion.
Bad separation between nitrogen and carbon peaks.	High nitrogen blank value.	Check the nitrogen blank value. Eventually repeat the analysis.
	Copper exhausted.	Replace the reactor.

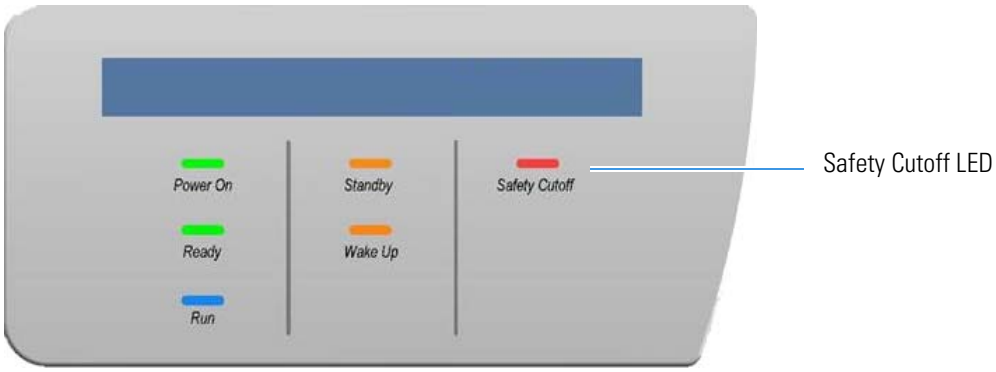
Table 25. Analytical Troubleshooting Guide (Sheet 2 of 2)

Problem	Diagnosis	Remedy
Peak between nitrogen and carbon peaks.	Oxygen line contaminated.	Exclude autosampler and check the oxygen blank.
	Inadequate oxygen purity.	Use Oxygen with adequate purity. Exclude autosampler and check the oxygen blank.
	Inadequate oxygen dosage.	Increase oxygen injection time.
High carbon blank.	Tin containers contaminated.	Check the tin container box, tweezers, work bench are clean.
	Memory effect due to bad combustion of previously run analyses.	Remove ash and analyze lower amounts of sample.
Decreasing nitrogen blank values.	Oxygen line contaminated.	Wait 10-20 minutes for complete purging of the oxygen line. Repeat blank analysis.
Increasing nitrogen blank values.	Copper exhausted	Replace the reactor.
Retention times very delayed respect the normal chromatogram.	Presence of leaks in the pneumatic circuit.	Perform Leak Test.
	Presence of obstructions in the pneumatic circuit.	Reach and remove the obstruction dissecting the pneumatic circuit.

Safety Cutoff

Instrument malfunctioning, due to a component failure or to abnormal operating conditions, is identified by the red lighting of the Safety Cutoff LED indicator. See Figure 145

Figure 145. Safety Cutoff LED



When lit, this LED indicates that the furnaces and detector oven power has been cut off for safety reasons.

The Safety Cutoff status is followed by an error message about the possible cause of error.

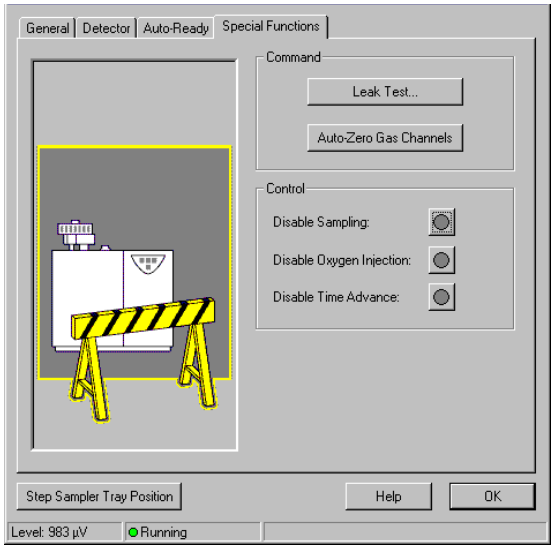
❖ To display the error message

Proceed as follows:

Proceed as follows:

1. In Isodat Software Suite, right click in the Flash IRMS dialogue box and then select settings for EA.
2. In the Main Menu for IRMS click the icon  .
3. In the visualized dialog window, select the **Special Functions** tabbed page. The dialog window of Figure 146 is visualized.
4. Read the error message in the reading box located on the lower right side of the window, below the buttons **Help** and **OK**.
5. Refer to Table 26 to find out the error status and have mode information.

Figure 146. Special function window



The following Table 26 reports the error messages and the explanation of the relevant correlated problem.

Table 26. Error Messages (Sheet 1 of 2)

Message	Description	What to do
Under voltage protection (Safety Cut Off message)	Voltages supplied to the electrical circuits are too low or out of tolerance.	Check all the voltages and main power.
Oven over limit (Safety Cut Off message)	The oven temperature exceed 190 °C or does not reach the setting temperature.	Check the functionality of the PT 100 probe. Replace the HWD 1112 board
Left furnace over limit (Safety Cut Off message)	The left thermocouple is damaged or interrupted.	Verify the continuity of the thermocouple and replace it.

Table 26. Error Messages (Sheet 2 of 2)

Message	Description	What to do
Right furnace over limit (Safety Cut Off message)	The right thermocouple is damaged or interrupted.	Verify the continuity of the thermocouple and replace it.
Thermal pre-protection (Safety Cut Off message)	Anomalous temperature inside the transformers compartment.	Check the functionality of the fans. Replace the AS 1112 board.
Oxygen pressure too low (NO Safety Cut Off message)	If the oxygen pressure is too low (<150 kPa) the flow of oxygen does not reach the set point.	Verify that the pressure at the manometer is at second stage higher or equal to 350 kPa.

CAUTION The error message is generally due to the specific cause indicated. Sometimes, it may generated by different electric factors or caused by failures not depending on the system. In this case contact the Technical Service.

EFC-t Module

The failures that may be generated on the EFCt Module are connected to the breakage, or to the malfunctioning of solenoid valves and flow sensors.

Refer to the Table 27 to find the component responsible of the EFC-t module malfunctioning and to solve the relevant problem.

Table 27. EFC-t module troubleshooting

Failure	Defective Component	Remedy
Oxygen does not flow to the point 2 of the autosampler	EV1	Check voltage supply Replace the solenoid valve
	EV2	Check voltage supply Replace the solenoid valve
The Helium flow measured on point 1 or 2 cannot be adjusted	Flow sensor 1 or 2.	Replace flow sensor
	EVP1 or EVP2	Check voltage supply Replace the solenoid valve
The pneumatic circuit is perfectly close but the flow value do not decrease up to zero performing the Leak Test.	EV3 and/or EV4	Check voltage supply Replace the solenoid valve

9

Running the Flash IRMS as Stand-alone Instrument

This chapter provides instruction for installing and configuring the Eager*Smart* Data Handling Software.

Contents

ATTENTION If you want to operate with the EA IsoLink CNSOH as a stand-alone instrument, you need to install Eager*Smart* software on you computer.

ATTENTION Eager*Smart* does not support the He^M. It is thus required that the He^M valve block is bypassed. Please refer to the proper Technical Note.

Eager*Smart* Software Installation

Eager*Smart* is compatible with commercially available computers and Windows® 2000, XP, Vista, 7, or 8 operating system

The required software package includes the following items:

- USB stick containing Eager*Smart* Data Handling Software
- Eager*Smart* Data Handling Software Manual

❖ **To install Eager*Smart* Data Handling Software**

Material required
Eager*Smart* Data Handling Software package

Eager*Smart* Data Handling Software is the dedicated software that fully controls all the operations of the EA IsoLink IRMS System for CNSOH, Flash 2000, Flash 4000, and EA 1110 Elemental Analyzers. It is designed to be compatible with commercially available computers, and requiring the use of Windows™ 2000 / XP / Vista / 7 / 8 operating system. The free space on the PC hard disk must be at least 1 GB.

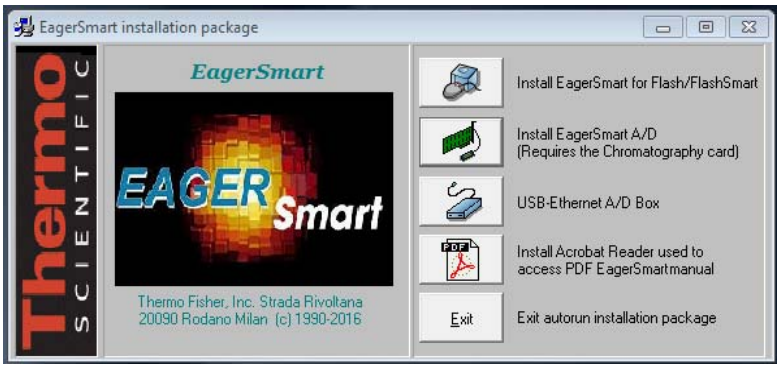
Eager*Smart* Data Handling Software is installed by using the USB stick provided in the standard outfit kit and operating as follows:

IMPORTANT Prior installation of Eager*Smart* Data Handling Software make sure any precedent version of Eager software is removed from the disk.

❖ To install Eager*Smart* Data Handling Software

1. Remove a precedent version of Eager software installed on your computer.
   a. Select **Control Panel | Add/Remove Programs**.
   b. In the dialog window visualized, select the precedent **Eager** version to remove.
   c. Click **Add/Remove**.
2. Install the new version of Eager*Smart* Data Handling Software. When the USB stick is introduced in a free USB port of the computer, the following installation window is visualized. See Figure 147.

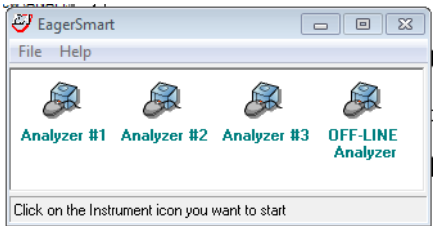
Figure 147. Eager*Smart* Data Handling Software Installation Menu



Note If when the USB stick is introduced in a free USB port of the computer, the installation menu does not automatically appear, through the Windows command **Start-Run**, start the program **Autorun** on the USB stick.

3. Start installation by clicking the push-button  **Install EagerSmart for Flash**.
4. Follow the instructions prompted step by step.
5. At the end of installation, in the page **Start-Program Eager*Smart***, double-click the **Eager*Smart* for Flash** icon. The window of Figure 148 is visualized.

Figure 148. Selection of the instrument

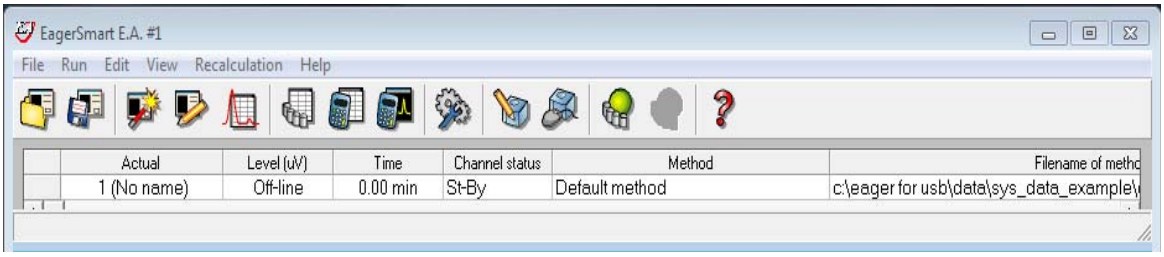


6. Click the icon of the instrument selected.The program is designed to work with four instruments. Each icon corresponds to one instrument. The instrument name shown below the icon can be changed. To do this, click on the existing name and overwrite the new one.
7. Eager*Smart* Data Handling Software proceeds with the registration and the activation of some drivers needed for the correct functioning of the software.
   a. Click **Ok** to the answers prompted step by step.
   b. At the end of the operation, reboot the computer. Start Eager*Smart* Data Handling Software again selecting **Start | Programs | Eager*Smart***.
8. Follow the prompted indications. At the end of the installation, the Main Menu is visualized.

Eager*Smart* Main Menu

The Main Menu of Eager*Smart* Data Handling Software, shown in Figure 149, is the starting point to enter all menus and relevant functions.

Figure 149. Eager*Smart* Data Handling Software Main Menu



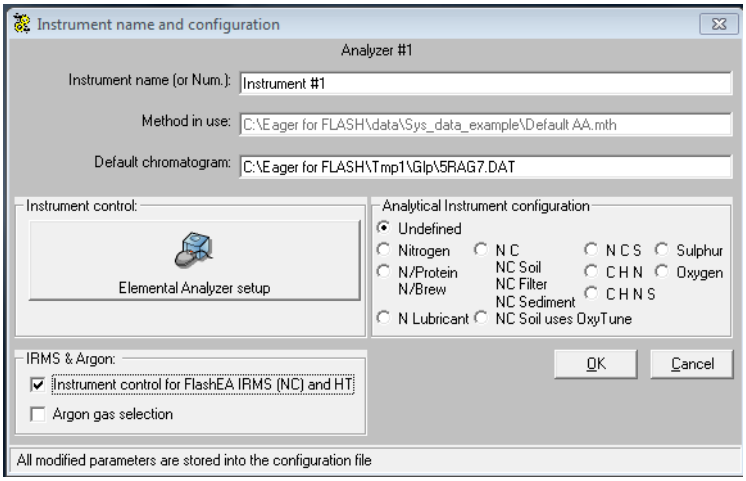
Configuring the Analyzer

The analytical conditions are set in our laboratories during the final test of the analyzer. To put the analyzer in operating conditions, you only have to follow the instructions reported in the next operating sequence "To configure the analyzer" on page 143.

❖ To configure the analyzer

1. In Main Menu of Figure 149 on page 143, choose **File | Instrument Name and Configuration**. The following window is visualized. See Figure 150.

Figure 150. Instrument Name and Configuration



a. In the field **Instrument name**, type the instrument **serial number** (6 digits; for example 991234). See the label located on the instrument rear panel.

b. In the field Analytical Instrument Configuration select Undefined.

c. In the field **IRMS & Argon** select **Instrument control for Flash EA IRMS (NC) and HT**.

d. Click **OK**. The system ask to exit and reboot Eager*Smart*.

e. At the reboot of Eager*Smart*, the simplified Main Menu for IRMS is visualized. See Figure 151.

Figure 151. Main Menu for IRMS

Menus and buttons of the Main Menu for IRMS are described in the following Table 28 and Table 29 respectively.

Table 28. Main Menu for IRMS: Description of Menus

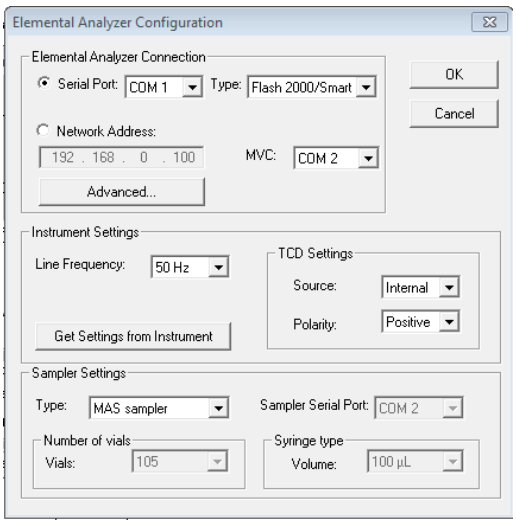
Menu	Description	Sub-menus and Options
File	Monitors the analysis in real time.	• Instrument name and configuration • View sample being acquired • Exit Eager*Smart*
Tools	Used when the ashes removal, the reactor replacement, or both, are required as maintenance.	• Ashes removal • Reactor replacement
Help	Accesses to the help system of Eager*Smart*.	• About Eager*Smart*

Table 29. Main Menu for IRMS: Description of Icons (Graphic Buttons)

Icon	Function	Description
	Edit elemental analyzer parameters	Gives the access to the three pages containing the commands for setting temperatures, flows, times, detector, and the analyzer control functions.
	Elemental analyzer status	Comprises four pages displaying the analyzer conditions.

f. In Main Menu for IRMS select **File | Instrument name and configuration** and click **Elemental analyzer setup** for visualizing the dialog window of Figure 152, where the configuration parameters must be set.

Figure 152. Configuration Dialog Window



g. In the field **Elemental Analyzer Connection** select the computer serial port (COM1, COM2, and so on), to which the instrument is connected.

h. In the field **Instrument Settings** choose the following settings:

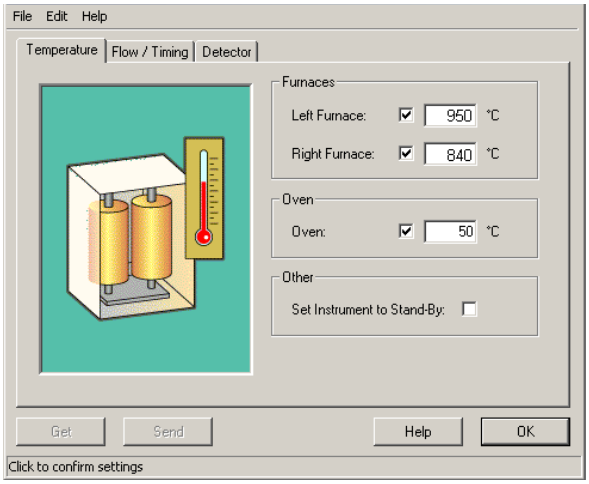
- Line Frequency = 50 Hz
- TCD Settings Source = Internal
- TCD Settings Polarity = Positive

i. In the field **Sampler Setting** select the type of autosampler installed on the instrument.

- In the case of autosampler for liquid samples, also specify the serial port of the computer to which the autosampler is connected, and the number of vials.
- Click **Ok** to go back to the window of Figure 152, then click **Ok** to return to Main Menu for IRMS.

2. In Main Menu for IRMS click the icon  . The following window appears where the analyzer operating parameters are visualized. See Figure 153.

Figure 153. Example of Analyzer Parameters



a. Click **Send** to transfer the operating parameters to the instrument. From now on the analyzer is working.

b. The furnaces begin to heat, and the helium flows in the circuit. After about 50 minutes the furnaces reach the temperature settings, and the LED **Ready** on the status panel lights up. The instrument is now ready to run analyses. However, before starting an analytical cycle, a **leak test** must be carried out for checking the **Carrier** and **Reference** pneumatic circuits are free of leaks.

> **Note** The leak test must be performed also any time a component of the pneumatic circuit is replaced for checking that reactors, filters, if any, and gas chromatographic columns have been properly installed. See the section "Performing a Leak Test" on page 147 for details.

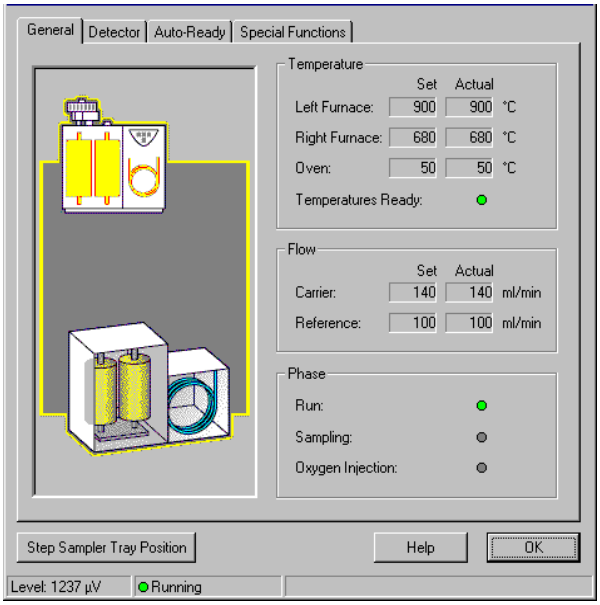
Performing a Leak Test

For performing the leak test follow the operating sequence "To check the leaks" on page 147.

❖ To check the leaks

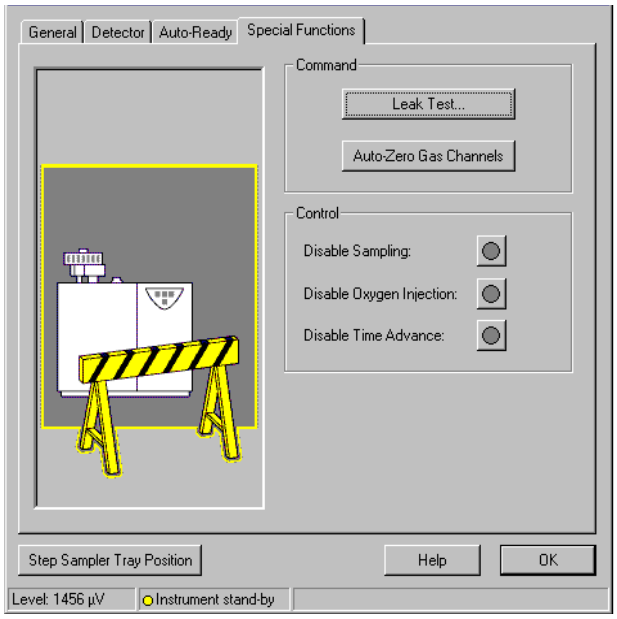
1. In Main Menu for IRMS click the icon  . The following dialog window is visualized. See Figure 154.

Figure 154. Analyzer Status Page



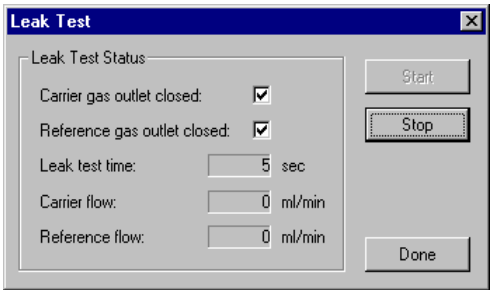
2. Select **Special Functions** tabbed page. See Figure 155.

Figure 155. Special Function Tab



3. In the field **Command** click **Leak Test**. The window of Figure 156 is visualized indicating the status of the leak test.

Figure 156. Leak Test Status Window



a. Click **Start** to begin the leak test. The system automatically selects **Carrier** and **Reference gas outlet closed** check boxes. The system will ask if the zero of flow controllers has to be calibrated; reply **Yes** to this question. This operation will take place in less than one minute.

b. Gas outlets are closed by the solenoid valves. Wait for some time (see **Leak test time)**, according to the instrument configuration, to let the gas circuit reach the equilibrium pressure. From the values of **Carrier Flow** and **Reference Flow** must be within **0** and **3 mL/min**. Higher values indicate that the system is not leak-free.

> **Note** Leaks in the system are generally due to incorrect closure of the reactors and filters locking nuts. Rarely, leaks may be due to the autosampler.

c. Click **Stop** and **Done** for ending the leak test and restoring the flow operating values.

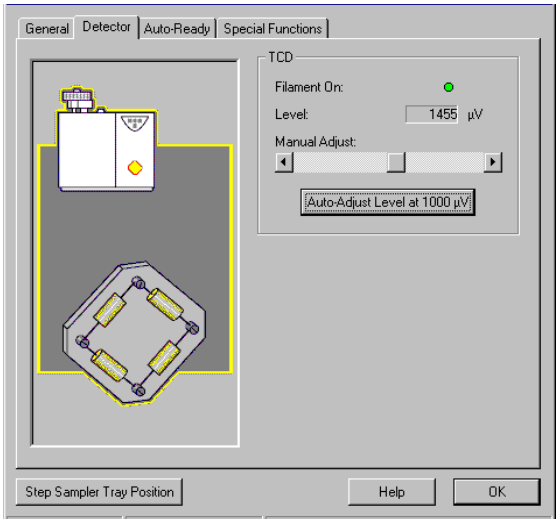
Adjusting the Detector Signal Level

To adjust the level of the TCD detector's signal, follow the instructions reported in the operating sequence "To adjust the level of the detector signal" on page 148.

❖ To adjust the level of the detector signal

1. In Main Menu select **View | View Elemental Analyzer Status**, or just click the icon  , then select the **Detector** tabbed page. See Figure 157.

Figure 157. Detector Status Tab



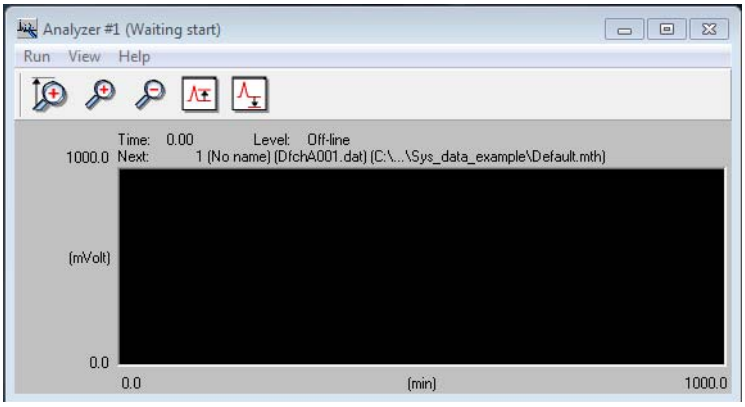
2. In the field **TCD**, click **Auto-Adjust Level at 1000 µV**. At the end of the operation, the value 1000 is set representing the analysis starting point.

View Sample Being Acquired

This section provides instruction for monitoring the sample currently being acquired.

View Sample Being Acquired is a tool to monitor the sample currently being acquired. See Figure 158.

Figure 158. View Sample Being Acquired Page



Note This page shows the real chromatogram being acquired.

Icon	Description	See on:
	Fit to Higher Peak	page 149
	Expand (scale/2)	page 150
	Shrink (Scale*2)	page 150

Icon	Description	See on:
	Step Up	page 150
	Step Down	page 150

Run Menu — gives access to the following functions:

- **Start Single Sample Data Acquisition** — Starts the acquisition of single sample of the instrument currently in use. All parameters set in **Detection Parameters** and **Integration Parameters** are used for starting the acquisition.
- **Stop Data Acquisition** — Stops the acquisition of the instrument currently in use.
  The system, after a confirmation, stops the acquisition saving the chromatogram data up to the moment of the **stop** command, and a report is generated if required.
- **Exit Monitor Detector** — Exits from detector monitoring. The program returns to the Main Menu, but the acquisition continues in the background.

View Menu — Gives access to the following functions:

- **Fit to Highest Peak** — Expands or reduces the chromatogram in order to fit all peaks being part of the partially acquired chromatogram centering the signal on the window display.
  This modification has effect only on the real time display, but it does not affect the integration and the chromatogram printout.

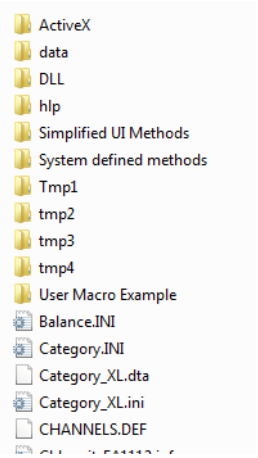
- **Step Up** — Lifts the chromatogram lowering the beginning of the scale by about 1/10 of the full scale. The result is a lift of the chromatogram baseline. This modification has effect only on the real time display, but it does not affect the integration and the chromatogram printout.
- **Step Down** — Lowers the chromatogram increasing the beginning of the scale by about 1/10 of the full scale. This modification has effect only on the real time display, but it does not affect the integration and the chromatogram printout.
- **Expand (scale/2)** — Expands the chromatogram by dividing the full scale in two. The system automatically re-draws the entire chromatogram with the new required scale. This modification has effect only on the real time display, but it does not influence the integration and the chromatogram printout.
- **Shrink (scale*2)** — Reduces the chromatogram by doubling the full scale. The system automatically re-draws the entire chromatogram with the new required scale. This modification has effect only on the real time display, but it does not affect the integration and the chromatogram printout.
- **Set Manual Scale** — Sets manually the values for the beginning and the full scale. You can enter the signal on the window display or you can display the signal with a desired attenuation. This modification has effect only on the real time display, but it does not affect the integration and the chromatogram printout.
- **Set Focus to Eager Main Menu** — Sets the focus on Main Menu. The function is only used when you wants to use the keyboard instead of the mouse. All functions, like in all other Windows™ applications, can be accessed with **Alt** key. After the use of **View chromatogram being acquired**, the function returns the focus on the Main Menu functions.

Configuring Eager*Smart* Data Handling Software

To control the EA IsoLink IRMS System for CNSOH version, it is necessary to properly configure the Eager *Smart* Data Handling Software:

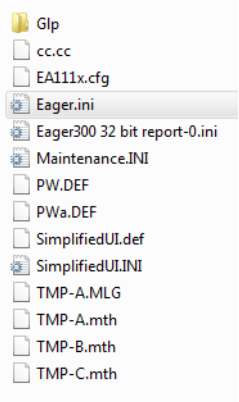
❖ To Configure Eager*Smart* Data Handling Software for HT

1. Exit Eager*Smart* Data Handling Software if active.
2. On the desktop select the directory where Eager*Smart* Data Handling Software is installed.



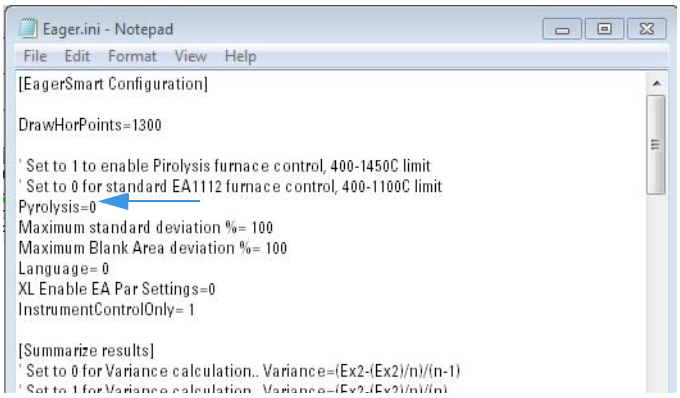
3. Double-click twice to open the relevant contents.

4. Note the four sub-folders named **Temp 1**, **Temp 2**, **Temp 3**, and **Temp 4**. Each of them corresponds to the relevant instrument (Analyzer #1, Analyzer #2, and so on), that may be configured during the Eager*Smart* Data Handling Software installation procedure.



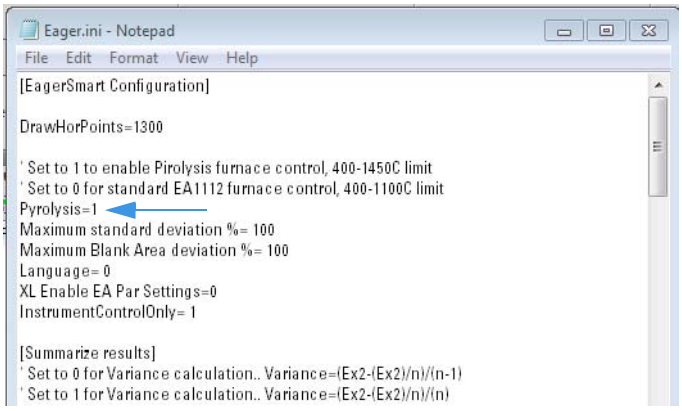
5. Double-click the sub-folder name that corresponds to your EA IsoLink IRMS System for CNSOH for opening the relevant contents. For example: **Temp 1 = Analyzer #1**. Double-click on the **Eager.ini** file for opening the file. See Figure 159.

Figure 159. Eager.ini File (1)



6. Search **Pyrolysis=0** line. Indicates the configuration of Eager*Smart* Data Handling Software to control a standard EA IsoLink IRMS System for CNSOH. For controlling a EA IsoLink IRMS System for CNSOH, modify the **Pyrolysis=0** line to **Pyrolysis=1**. See Figure 160.

Figure 160. Eager.ini File (2)

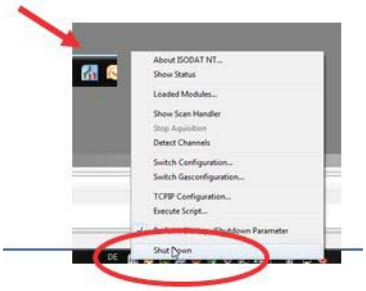


7. Save the modification and close the file. Run Eager*Smart* Data Handling Software. A message appears indicating that the instrument is now configured to operate under pyrolysis condition.

Using Isodat Software Suite and Eager*Smart* Data Handling Software at the Same Time

❖ To install the software version

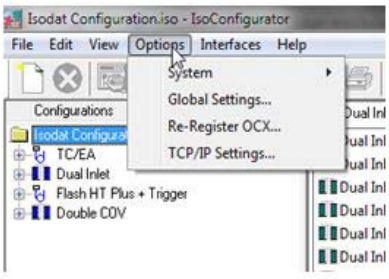
1. Make a backup of Isodat Software Suite with the Version Handler if Isodat Software Suite is installed.
2. Make an additional backup of the **User** folder under Isodat Software Suite.
3. Close down Isodat Software Suite completely by right-click task bar.



4. Install Eager*Smart* Data Handling Software.

❖ To switch fromEager*Smart* Data Handling Software to Isodat Software Suite

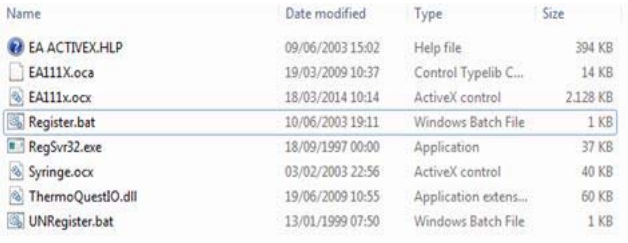
1. Before using either software make sure the other software is completely closed.
2. Open **Acquisition**. The OCX is automatically registered for Isodat Software Suite if Eager*Smart* Data Handling Software is completely closed down first. If problems occur a Safety Cut-Off message is visualized. Go to the Isodat Software Suite **Configurator** and **Re-Register OCX**, then wait 10 seconds.



3. Close **Configurator** and start **Acquisition**.

❖ To switch from Isodat Software Suite to Eager*Smart* Data Handling Software

1. Before using either software make sure the other software is completely closed.
2. Start Eager*Smart* Data Handling Software. The OCX is automatically registered for Eager*Smart* Data Handling Software if Isodat Software Suite is completely closed down. If problems occur you may register and unregister the OCX using the two bat –files in the following folder of Eager*Smart* Data Handling Software:



Name	Date modified	Type	Size
EA ACTIVEX.HLP	09/06/2003 15:02	Help file	394 KB
EA111X.oca	19/03/2009 10:37	Control Typelib C...	14 KB
EA111x.ocx	18/03/2014 10:14	ActiveX control	2.128 KB
Register.bat	10/06/2003 19:11	Windows Batch File	1 KB
RegSvr32.exe	18/09/1997 00:00	Application	37 KB
Syringe.ocx	03/02/2003 22:56	ActiveX control	40 KB
ThermoQuestIO.dll	19/06/2009 10:55	Application extens...	60 KB
UNRegister.bat	13/01/1999 07:50	Windows Batch File	1 KB

EAGER For FLASH | Active X | Vers_1_x

Glossary

This section lists and defines terms used in this guide. It also includes acronyms, metric prefixes, symbols and abbreviations.

A B C D E F G H I J K L M N O P Q R S T U V W X Y Z

A ampere

ac alternating current

ADC analog-to-digital converter

Ar Argon

b bit

B byte (8 b)

baud rate data transmission speed in events per second

C Carbon

°C Celsius

cm centimeter

CPU central processing unit (of a computer)

CSE Customer Service Engineer

<Ctrl> control key of the keyboard

d depth

DAC digital-to-analog converter

dc direct current

DS data system

EMC electromagnetic compatibility

ESD electrostatic discharge

°F Fahrenheit

FSE Field Service Engineer

ft foot

g gram

GND electrical ground

h height

h hour

H hydrogen

harmonic distortion A high-frequency disturbance that appears as distortion of the fundamental sine wave

He Helium

HPAR High Performance Alloy Reactor

HV high voltage

Hz hertz (cycles per second)

IEC International Electrotechnical Commission

in. inch

I/O input/output

k kilo (10^3 or 1024)

K Kelvin

kg kilogram

kPa kilopascal

l length

l liter

LAN Local Area Network

lb pound

LED light-emitting diode

m meter (or milli [10^{-3}])

M mega (10^{6})

µ micro (10^{-6})

min minute

mL o ml milliliter

mm millimeter

m/z mass-to-charge ratio

N nitrogen

n nano (10^{-9})

O oxygen

Ω ohm

p pico (10^{-12})

Pa pascal

PCB printed circuit board

PN part number

psi pounds per square inch

RAM random access memory

<Return> <Return> key on the keyboard

RF radio frequency

ROM read-only memory

RS-232 industry standard for serial communication

s second

slow average A gradual long-term change in average RMS voltage level, with typical duration greater than 2 s.

surge A sudden change in average RMS voltage level, with typical duration between 50 µs and 2 s.

S sulfur

TCD Thermal Conductivity Detector

transient A brief voltage surge of up to several thousand volts, with a duration of less than 50 µs.

V volt

V ac volts, alternating current

V dc volts, direct current

VGA Video Graphics Array

w width

W Watt

When a unit of measure has a quotient (e.g. Celsius degrees per minute or grams per liter) this can be written as negative exponent instead of the denominator:
For example:
°C min^{-1} instead of °C/min
g L^{-1} instead of g/L

ThermoFisher
SCIENTIFIC