

**Organic Elemental Analysis**

# **Flash 2000**

Elemental Analyzer

## **Operating Manual**

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[HOME](#)

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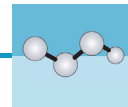
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# Declaration

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.

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## Preface

This *Operating Manual* contains descriptions of the features and components of the Flash 2000 Elemental Analyzer, inside, you will find all of the information necessary for routine operation of your Elemental Analyzer, including operating sequences, sample injection techniques, diagrams and descriptions of the major components.



The Flash 2000 Elemental Analyzer

This manual is organized as follows:

**Chapter 1, “Preliminary Information,”** provides information on the classification of the equipment, its safety and configurations. Basic technical features are also described.

**Part 1, “Description,”** describes the structure of the instrument and the pneumatic circuits.

**Chapter 2, “Structure of the Instrument,”** describes the structure of the instruments and its major features.

**Chapter 3, “Instrument Description,”** gives you a detailed description of the instrument components.

**Chapter 4, “Pneumatic Circuits,”** describes the pneumatic circuit of each instrument configuration in the pre-run condition.

**Part 2, “Preparation,”** , provides description of the reactors and adsorption filters required to run analyses with instructions for their preparation, and also description of gas chromatographic columns. Moreover it contains instructions to install and remove the system reactors and filters, to perform sample preparation and weighing, and also how to start up the instrument before running analyses.

**Chapter 5, “Preparation of Reactors and Adsorption Filters,”** , provides instructions for the preparation of the reactors and the adsorption filters, and it also reports the types of analytical columns currently used.

**Chapter 6, “Connecting Reactors and Adsorption Filters,”** , contains the instructions to install reactors and adsorption filters into the elemental analyzer, and it also provides information on how to remove them.

**Chapter 7, “Preparing the Sample,”** , describes some techniques for the sample preparation, also it provides basic instructions to homogenize and weigh the sample.

**Chapter 8, “Analytical Methods,”** , describes the analytical methods used for all configurations of the Flash 2000 elemental analyzer.

**Part 3, “Analysis,”** , contains information and operating sequences to perform the analysis preparation, execution and interpretation.

**Chapter 9, “Instrument Start-up,”** , contains information and operating procedures to prepare the instrument for running analyses.

**Chapter 10, “Guide to Run Analyses,”** , contains information and operating sequences to run sample analyses, and it also describes the comparison methods for a correct evaluation of results. Practical advise for daily operation is also provided.

**Chapter 11, “Applications,”** , contains guidelines referring to the applications of the Flash 2000 elemental analyzer.

**Chapter 12, “Use of Eager Simplified User Interface,”** , provides information about the use of the “easy” version of the Eager 300 software.

**Part 4, “Maintenance and Troubleshooting,”** , contains information and operating sequences to perform the necessary maintenance of the instrument and also information concerning troubleshooting in case of malfunctioning.

**Chapter 13, “Maintenance,”** , provides information on the current and periodic maintenance of the instrument, and it also contains the operating sequences for installation and maintenance of the MAS 200 R Autosampler and the CM2 Manual Sampler.

**Chapter 14, “Troubleshooting,”** , provides the information necessary to give you some hints on instrument problems and how to solve them.

**Appendix A, “Customer Communication.”** , contains information for direct contact with Thermo Fisher Scientific offices worldwide. This appendix also contains a one-page [Reader Survey](#).

The [Abbreviations](#) contains definitions of terms used in this guide. It also includes abbreviations, acronyms, metric prefixes, and symbols.

The [Index](#) contains an alphabetical list of key terms and topics in this guide, including cross references and the corresponding page numbers.

## Compliance

We thoroughly test and evaluate our products to ensure full regulatory compliance with applicable domestic and international regulations. Your system (hardware and software) is CE Compliant and meets Electromagnetic Compatibility (EMC) and safety standards.



**CAUTION Instrument Damage.** Flash 2000 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 may be impaired. If you maintain a system outside the specifications listed in this guide, failures of many types may occur. The repair of such failures may be excluded from the documents regarding your standard warranty and service contract coverage.

## Safety Alerts and Special Notices

In this paragraph, safety alerts and other special notices appear in boxes.

*Safety alerts* are a combination of *safety symbols* and *signal words* designed to alert you to protect yourself and/or your instrument. Please read about the types of safety alerts, signal words, and the safety symbols that are presented in this guide and presented on the instrument.

## Safety Symbols

Safety and special notices that may be found on your instrument and in this manual include the following:



The *General Warning* symbol/sign is a triangle with an exclamation mark that is used next to the signal word. In the vocabulary of ANSI Z535 signage this symbol indicates a possible personal injury hazard exists. The ISO 3864-2 standard refers to this as the general warning sign. This symbol alerts you to an action or sequence that, if improperly performed could result in damage to the instrument or possible personal injury. This symbol is followed by signal words such as Danger, Warning, or Caution indicating the risk.



This symbol indicates that an electrical shock hazard *will, could, or may* occur.



This symbol indicates a hot surface. Make sure the instrument is at room temperature before touching, or else you *will, could, or may* incur burn injuries.



This symbol indicates a risk of fire or flammability, or that fire/flammability damage *will, could, or may* occur.



This symbol indicates that a biohazard *will, could, or may* occur.



This symbol indicates that chemical damage or physical injury *will, could, or may* occur.



This symbol indicates the presence of radioactive material.



This symbol indicates that eye damage *will, could, or may* occur. Eye protection must be worn.



This symbol indicates the user must wear gloves when performing the sequence.

## Types of Alerts and Signal Words

Safety alerts that may or may not be associated with the use of this instrument. These instructions are defined as follows:

**DANGER** safety alerts an imminent hazard exists that **WILL** result in death or serious personal injury.

**WARNING** safety alerts you to an action or sequence that, if improperly performed, could result in damage to the instrument or possible physical harm to the user. This symbol may be followed by icons indicating special precautions that should be taken to avoid injury.

**CAUTION** safety alerts you to an action or sequence that, if performed improperly, could damage the instrument.



**Note** alerts you to important information related to the test.

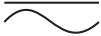















## Special Notices


**Tip** Helpful information that can make a task easier.

## 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 Flash 2000. See the asterisk.

Symbol	Description
==	Direct Current
*	  Alternating Current

Symbol	Description
	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	Description
* 	Symbol in compliance to the Directive 2002/96/EC on Waste Electrical and Electronic Equipment (WEEE) placed on the european market after August, 13, 2005.

## Using the Flash 2000 Document Set

The Flash 2000 Document Set (CD-Rom PN 317 095 10) includes all manuals in electronic format, and serves as your library for information about the hardware and software of your elemental analyzer.

The Flash 2000 Document Set (PN 317 120 50) as paper copy is also available Furthermore, Thermo Fisher Scientific part numbers (PN) for the paper copy manuals are provided for each book title.

- *Site Preparation and Installation Requirements* (PN 317 120 51)  
This manual describes how to set up a workspace for the instrument and accessories and how to connect the main unit to the gas supplies and peripheral devices.
- *Operating Manual* (PN 317 120 52)  
This manual provides the descriptions of the hardware and software and detailed instructions for their use.
- *Consumables and Spare Parts Catalog* (PN 317 082 50)  
This catalog contains a list of consumables and spare parts for the Flash 2000.

**Other available manuals are:**

- *Eager Xperience Software Manual* (PN 317 110 55)  
This manual contains instruction to operate with Eager Xperience to the fully control of the Flash elemental analyzers.
- *AI 3000/AS 3000 II Autosampler for Flash Operating Manual* (PN 317 094 45)  
This manual contains description of the features and components of the AI 3000/AS 3000 II Autosampler for Flash elemental analyzers.
- *Flash HT Elemental Analyzer User Guide* (PN 317 082 71)  
This guide is an additional section of the *Flash 2000 Operating Manual* containing the instruction to operate on the Flash HT Elemental Analyzer.



# Preliminary Information

This chapter provides information on the classification of the equipment, its safety and configurations. Basic technical features are also described.

## Contents

- [Classification of the Instrument](#)
- [Technical Features](#)
- [Safety Information](#)
- [Safety Cut Off Device](#)
- [Instrument Cleaning](#)
- [Instrument Configuration](#)
- [Standard Outfit](#)

## Classification of the Instrument

### Environmental Conditions

- Internal use.
- Altitude up to 2000 meters.
- Temperature from 15 to 35 °C.
- Maximum relative humidity between 30% and 85%.
- Voltage variations not exceeding  $\pm 10\%$  of the nominal value.
- Transients according to installation categories **II**.
- Degree of pollution according to IEC 664 (3.7.3) **2**.

## Technical Features

The following table summarizes the major technical features of the Flash 2000 elemental analyzer.

**Table 1.** Technical features of the instrument

Features	Description
Instrument configurations	Fourteen
Detector	Thermal conductivity detector (TCD)
External interface	RS 232 serial line
Instrument control	Eager Xperience for Windows™
Power supply	230 Vac; 50/60 Hz; 1400 VA
Dimensions (cm)	Height 50 (54 with fittings); Width 59, Depth 58
Mass (kg)	65

## 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, etc., the degree of insulation of peripheral devices should be equivalent or higher (double or reinforced) than that of the Flash 2000. 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 Safety 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<sub>2</sub>) as gas for sample oxidation.



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

## Purity of Gases

The Flash 2000 uses Helium and Oxygen with **99.995%** minimum purity.

## Nominal Pressure of Gases

The nominal pressure of the gases to supply the Flash 2000, as indicated on the instrument rear panel, are:

- Maximum 250 kPa (2.5 bar) for He
- Maximum 250-300 kPa (2.5-3 bar) for O<sub>2</sub> according to the analytical configuration

## 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 traps.

For more details please refer to [Chapter 14, “Troubleshooting.”](#)

## Instrument Cleaning

### ❖ Instruments Cleaning



**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, carefully avoid seeping of the products used inside 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 elemental analyzer during operation and maintenance.

## Instrument Configuration

The Flash 2000 elemental analyzer is available in fourteen different versions. Analytical techniques, pneumatic circuits and standard outfits are different for each version.

**Table 2.** Flash 2000 Series: Instrument versions

Configuration	Description
CHN Analyzer	For the determination of the amount (%) of carbon, hydrogen and nitrogen, contained in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.

**Table 2.** Flash 2000 Series: Instrument versions, continued

<b>Configuration</b>	<b>Description</b>
CHN-O Analyzer	<p>For the determination of the amount (%) of carbon, hydrogen, nitrogen and determination of oxygen, contained in organic and inorganic chemicals and in substances of different nature and origin, be they solid, liquid or gaseous samples.</p> <p><i>The determination of carbon, hydrogen and nitrogen is performed in a single sample analysis, whereas oxygen determination is performed separately.</i></p>
CHNS Analyzer	<p>For the simultaneous determination of the amount (%) of carbon, hydrogen, nitrogen and sulfur, contained in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.</p>
CHNS-O Analyzer	<p>For the determination of the amount (%) of carbon, hydrogen, nitrogen, sulfur and determination of oxygen, contained in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.</p> <p><i>The determination of carbon, hydrogen, nitrogen and sulfur is performed in a single sample analysis, whereas oxygen determination is performed separately.</i></p>
S Analyzer	<p>For the determination of the amount (%) of sulfur contained in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.</p>
O Analyzer	<p>For the determination of the amount (%) of oxygen contained in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.</p>
N Analyzer	<p>For the determination of the total amount of nitrogen present in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.</p>
N Lubricant Analyzer	<p>For the determination of the total amount of nitrogen present in lubricants, lubricant additives, fuel additives, petrochemical products.</p>

**Table 2.** Flash 2000 Series: Instrument versions, continued

Configuration	Description
NC Analyzer	For the determination of the amount (%) of nitrogen and carbon contained in materials of different kinds: <ul style="list-style-type: none"> <li>• <i>Synthetic materials</i>: polymers, rubbers, tyres, etc.</li> <li>• <i>Explosives</i>: nitrocellulose, TNT, gun powder, etc.</li> <li>• <i>Special materials</i>: carbon fibers, glass fibers, conductive polymers, graphite, etc.</li> <li>• <i>Metallurgy</i>: metal powders, steels, etc.</li> <li>• <i>Environmental analyses</i>: muds, discards, organic wastes, etc.</li> </ul>
NCS Analyzer	For the simultaneous determination of the amount (%) of nitrogen, carbon and sulfur contained in organic and inorganic chemical products and in substances of different nature and origin, be they solid, liquid or gaseous samples.
NC-Soils Analyzer	For the determination of the nitrogen and carbon content in soil samples.
NC-Sediments Analyzer	For the determination of the nitrogen and carbon content in sediments.
NC-Filters Analyzer	For the determination of the nitrogen and carbon content in particulate samples filtered on Whatman filters.
N/Protein Analyzer	For the determination of the nitrogen and protein amount in products of biological origin; it can also be used in determining nitrogen content in samples of different nature, generally agricultural products and foodstuff.
N-Brew Analyzer	For the determination of nitrogen content in samples of different nature, belonging to the brewing industry, such as malt, barley, wort and beers.
NC-IRMS Analyzer <sup>1</sup>	Flash 2000 analyzer able to be coupled to an Isotopic Ratio Mass Spectrometer (IRMS).for the determination of Nitrogen and Carbon.
Flash 2000 HT analyzer <sup>1</sup>	Flash 2000 analyzer with the left furnace at high temperature (1450 °C) for the determination of Oxygen and Hydrogen by pyrolysis and with the right furnace at 1000 °C for the determination of Nitrogen and Carbon (or Sulfur).

1.For details about these configurations refer to the Flash 2000 HT User

## Standard Outfit

Flash 2000 elemental analyzer is provided with its own standard outfit. Use the standard outfit checklist accompanying the instrument to verify that all items have been received.



---

Part

1

# Description

---

## Structure of the Instrument

This chapter describes the structure of the instrument and its major features.

### Contents

- [The Units Constituting the Instrument](#)
- [Analytical Section](#)
- [Control Section](#)
- [Automation](#)
- [Pneumatic Circuit](#)

## The Units Constituting the Instrument

The instrument, in its different configurations, consists in a single structure subdivided into two sections:

- Analytical Section
- Control Section

## Analytical Section

It comprises the following major components:

### Furnaces

Each furnace consists of a candle surrounded by an electrical resistor. The candle is plunged in a refractory material housed in a metal compartment.

- **Furnace Temperature**  
The temperature is monitored by a thermocouple appropriately located in the furnace.
- **Furnace Cooling**  
The cooling time varies according to the operating temperature setting.

The Flash 2000 analyzer can be equipped with one or two furnaces according to the instrument configuration.



- **LEFT Furnace**  
Present in all configurations.
- **RIGHT Furnace**  
Present only when required by the instrument configuration.

## Thermal Conductivity Detector (TCD)

It is 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 pyrolysis process.

The column can be kept at room temperature, or it can be placed in the thermostatic chamber of the TCD detector according to the instrument configuration.

The CHNS-O and CHN-O instrument versions use two analytical columns placed inside the thermostatic chamber.

## Adsorption Filters

They can be made of glass or Plexiglas according to the analytical configuration. Refer to [Chapter 5, "Preparation of Reactors and Adsorption Filters,"](#) .

## Reactors

These are tubes made of quartz or special alloy filled with different materials according to the analytical configuration. Refer to [Chapter 5, "Preparation of Reactors and Adsorption Filters,"](#) .

## Autosampler

It performs the automatic injection into the reactor of samples.

## Control Section

The control section consists of two major components:

### Pneumatic Compartment

It consists of two pressure reducers, two pressure gauges and of several lines fitted with an thermoregulator electronic flow controller (EFCt), which ensures the switching between helium and oxygen, and controls the flow values.

For more details, please refer to [Chapter 4, "Pneumatic Circuits,"](#) .

## Electronic Compartment

It comprises the electronic boards for the instrument power supply and control.

## Automation

The instrument is fully controlled by the computer through **Eager Xperience** dedicated software which is also used for data acquisition, data handling and interpretation of the acquired results. Therefore the instrument is not provided with independent keyboard and display. On the instrument front there is a synoptic panel where you can monitor the instrument statuses.

**Eager Xperience** is designed to be compatible with commercially available computers and Windows™ 2000/XP/Vista operating system.

**Table 3.** Hardware minimum requirements

Components	Description
Computer	Any PC can be used, including laptop computer
	Operating System: Windows™ 2000/XP/Vista
	Pentium Processor minimum 256 MHz
	Hard drive with at least 1 GB free
	One free COM port for instrument contr
	One free COM port for balance, if required
	One free COM port for AI 3000/AS 3000 II autosampler, if required
	CD driver
Monitor	Color 1024 x 768 or better
Printer	Any printer accepted by the operating system

## Pneumatic Circuit

Each configuration of the Flash 2000 elemental analyzer has its own dedicated pneumatic circuit. For details, please refer to [Chapter 4, “Pneumatic Circuits,”](#) .



# Instrument Description

This chapter gives you a detailed description of the instrument components.

## Contents

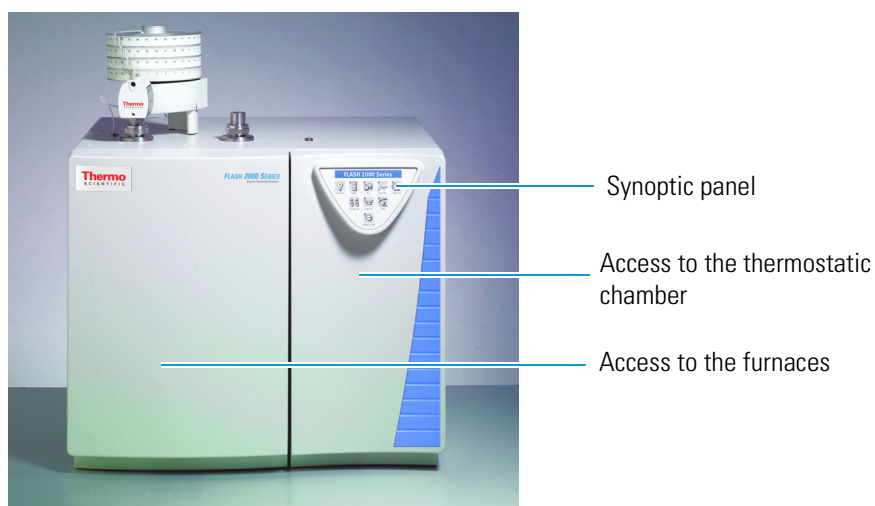
- Front Panel
- Rear Panel
- Top Panel
- Furnaces Compartment
- Fixing Plates for the Reactors
- Fittings for Gas Connections
- Detector Compartment
- Description of the Detection System
- Electrical Compartment
- Connections Panel
- Transformers Compartment
- Synoptic Panel
- Autosamplers
- Manual Injection Device for Liquids

## Front Panel

It comprises:

- Furnaces compartment  
Also refer to paragraph “Furnaces Compartment” on page 28.
- Synoptic panel  
Also refer to paragraph “Synoptic Panel” on page 38.
- Compartment for the TCD detector thermostatic chamber and for the gas chromatographic column.  
Refer to paragraph “Detector Compartment” on page 30.

**Figure 1.** Instrument front panel



## Rear Panel

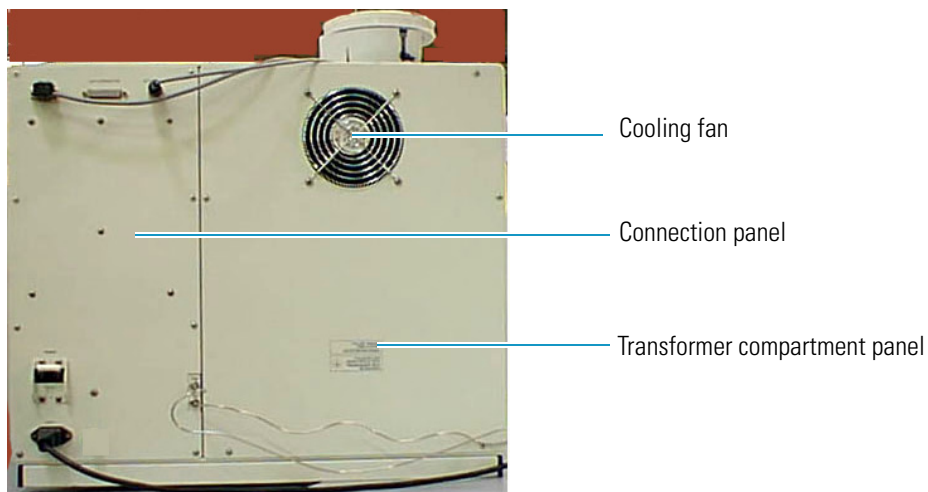
It comprises:

- Panel for connections including:
  - interface section
  - gas inlet section
  - electrical section

Also refer to paragraph “Connections Panel” on page 35.

- Cooling fan
- Transformers compartment  
Also refer to paragraph “Transformers Compartment” on page 37.

**Figure 2.** Instrument rear panel



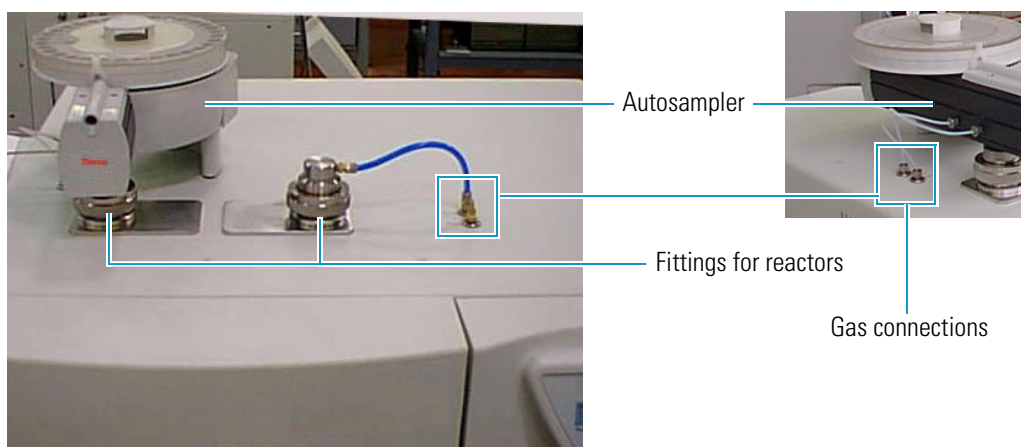
## Top Panel

It comprises:

- Fittings for mounting and securing the furnaces reactors
- Fittings for gas connection.

Also refer to paragraph “[Fixing Plates for the Reactors](#)” on [page 29](#)

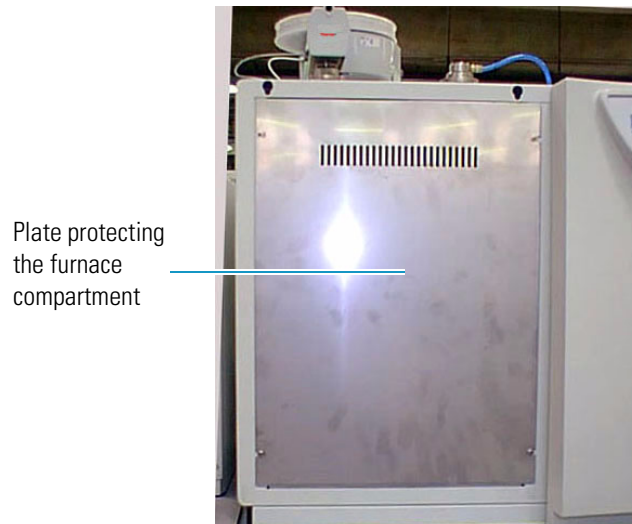
**Figure 3.** Instrument top panel



## Furnaces Compartment

The furnaces compartment can be reached from the instrument front and removing (lifting) the cover. The furnaces are accessible by removing the protecting plate.

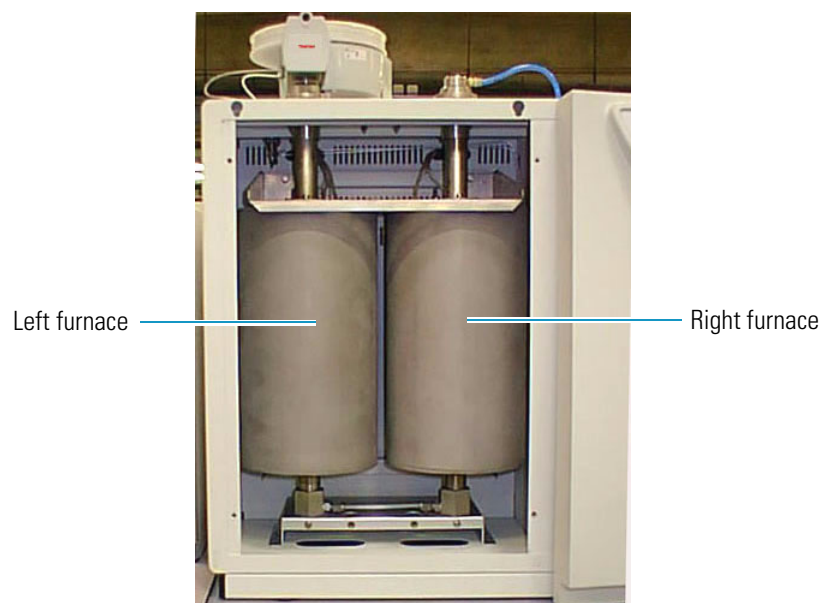
**Figure 4.** Furnace compartment with protecting plate



**WARNING** Do not open the furnaces compartment during operation because very high temperatures are reached.

The protecting plate can be removed only when the furnaces temperature shown is near the room temperature.

**Figure 5.** Furnaces compartment



The furnaces can reach the following maximum temperatures:

- LEFT Furnace: 1100 °C (1500 °C for HT)
- RIGHT Furnace: 1100 °C

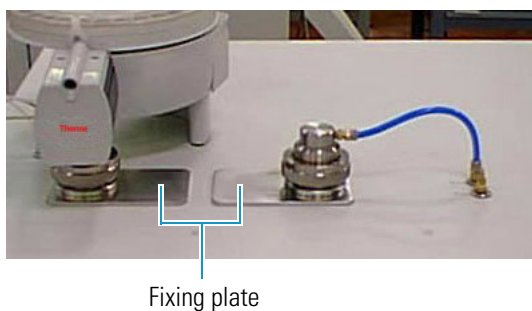
The furnace temperature is monitored by a thermocouple located inside the furnace.  
The furnaces are cooled when required by the operator.  
The cooling time depends on the operating temperature.

## Fixing Plates for the Reactors

These plates, on top of the furnaces compartment, accommodate the following components:

- Connections and fittings for the reactors.
- Automatic or manual sampler.

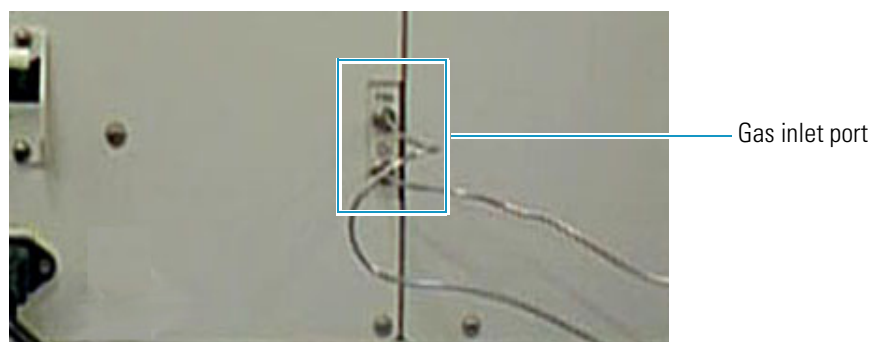
**Figure 6.** Fixing Plates for the Reactors



## Fittings for Gas Connections

They are located on the middle bottom part of the instrument rear panel. Gas inlet ports are directly connected to the pressure regulators.

**Figure 7.** Gas connection ports





**Table 4.** Gas Inlet Ports and Pressure Setting

Port	Description	Pressure value to be set
He	Inlet port for Helium	250 kPa (2.5 bar)
O <sub>2</sub>	Inlet port for Oxygen	250-300 kPa (2.5-3 bar) according to the instrument configuration

Gas pressures must be set and controlled through the pressure regulators and the gauges of the instrument. [Table 5](#) provides indications on the most currently used units of pressure.

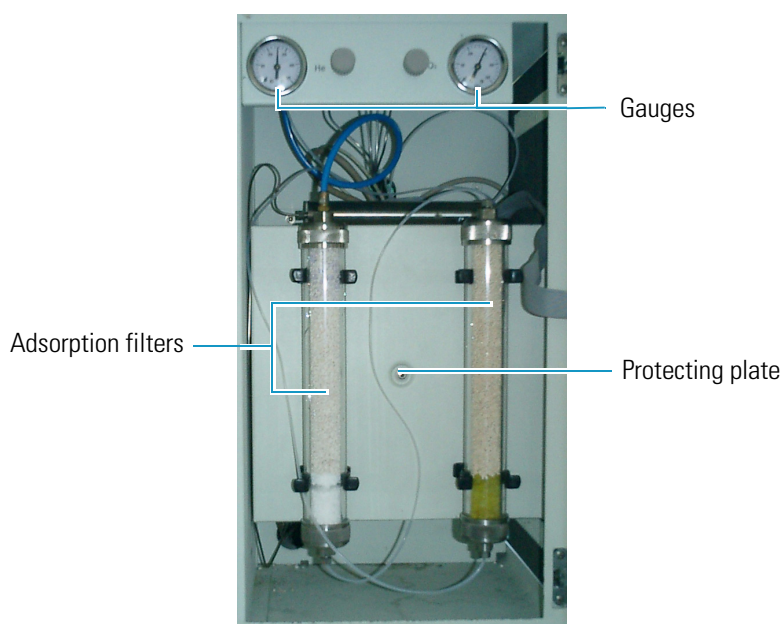
**Table 5.** Pressure units conversion

To convert	into	multiply by
kPa	bar	0.01
	psi	0.145
bar	kPa	100
	psi	14.51
psi	kPa	6.89476
	bar	0.0689476

## Detector Compartment

It is located on the right front part of the instrument and can be reached by opening the door. [Figure 8](#) shows the inside of the detector compartment.

**Figure 8.** View of the detector compartment



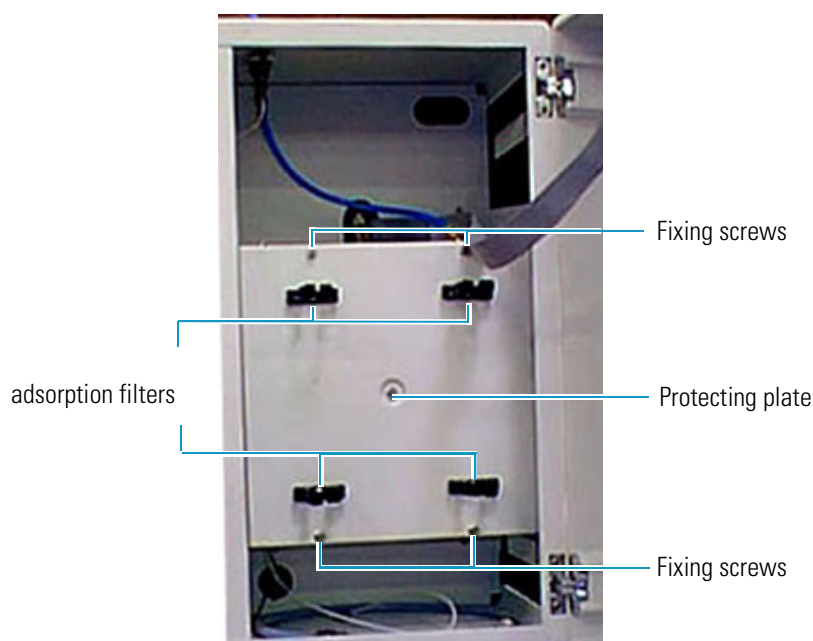
The detector compartment houses the pressure regulators, the gauges and the thermal conductivity detector (TCD), located behind the protecting plate and the gas chromatographic column. Refer to “[Access to the Detector](#)” on [page 31](#). The adsorption filters are housed in this compartment.

**Note** One or two adsorption filters may be required, according to the instrument configuration.

❖ **Access to the Detector**

1. Open the right side door to have access to the thermostatic chamber,
2. To reach the detector, remove the adsorption filters from the fastening clips.
3. Undo the four fixing screws on the protecting plate.

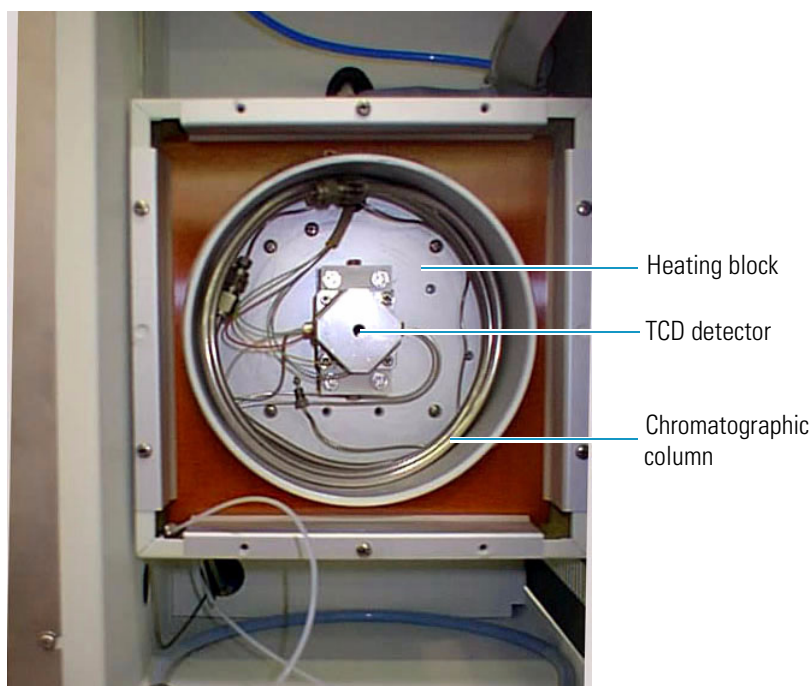
**Figure 9.** Access to the detector



[Figure 10](#) shows the detector compartment, the heating block surrounding the detector, and the gas chromatographic column.

**Note** One or two analytical columns may be required, according to the instrument configuration.

**Figure 10.** TCD detector compartment



## Description of the Detection System

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 essentially 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 at 5 V constant voltage 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, refer to the relevant paragraph, [page 35](#)).

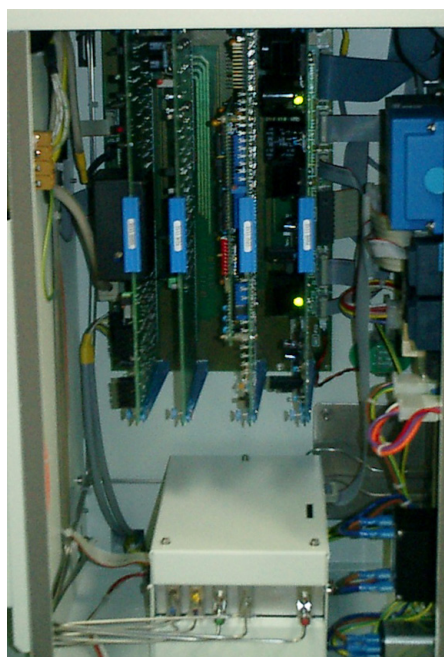


**CAUTION** Before opening the electrical compartment, cut off power supply to the instrument and disconnect the power cord.

The electrical compartment, shown in [Figure 11](#), comprises:

- Low voltage section
- Mains voltage section
- EFC electronic flow controller for gas regulation

**Figure 11.** Electrical compartment internal view



### Low Voltage Section

It contains the electronic boards to operate and control the instrument. These boards (in double EUROCARD format, except the EV Control Box and FP1112 boards) are interlocked through a mother board.

### Function of the Electronic Boards

The following [Table 6](#) reports the function of each electronic board present in the low voltage section:

**Table 6.** Description of the Function of the Electronic Boards

Board	Function
MB 1112	<p>Mother board. It provides interlocking between low voltage boards and with the rest of the instrument.</p> <p>This board can be connected to a NiCd 3,6 V; 280 mA/h rechargeable battery located nearby.</p> <p><i>The rechargeable battery replacement must be performed by specialized technical personnel</i></p>
CPU 1112	<p>This board has full control of the instrument operation. It controls the communication between operator and machine through Eager Xperience.</p> <p>Actuates the Safety Cut Off device, which puts the instrument in safe conditions, when an alarm condition occurs.</p>
EV Control Box	<p>Receives voltage supply from the TRF 1112 board. It operates the gas controlling solenoid valves contained in the EFC flow controller.</p> <p>The board is provided with a T0,16A 127/III (5 x 20 mm) protection fuse.</p>
HWD 1112	<p>Provides power supply to the HWD detector filaments.</p> <p>Allows the detector oven thermoregulation and also amplifies and converts the detector signal to send it to the PC.</p>
TCR 1112	Operates and controls the furnaces thermoregulation.
PWR 1112	<p>Receives voltage supplies from the TRF 1112 transformers board.</p> <p>It generates voltage supply for the electronic control circuits.</p>
FP 1112	Synoptic panel

## Main Voltage Section

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

The following [Table 7](#) reports the function of each component present in the mains voltage section.

**Table 7.** Description of the components of the Main voltage section

Component	Description
TRF 1112 Transformers Board	<p>Receives the mains power and supplies it to the following devices:</p> <ul style="list-style-type: none"> <li>• Cooling fan</li> <li>• Furnaces transformers</li> <li>• Heater of the detector thermostatic chamber</li> </ul> <p>Six fuses are provided on the board. See <a href="#">Table 8</a>.</p>
AC 1112 Furnaces Power Supply	<p>Supplies 48 Vac power to the furnaces. It contains the SSR relays for the furnaces control.</p> <p>Also refer to paragraph “<a href="#">Devices for the Furnaces Control</a>” on <a href="#">page 37</a>.</p> <p>Two fuses are provided on the board. See <a href="#">Table 8</a>.</p>
Breaker	Instrument ON/OFF main switch.

**Table 8.** Fuses of the High voltage section

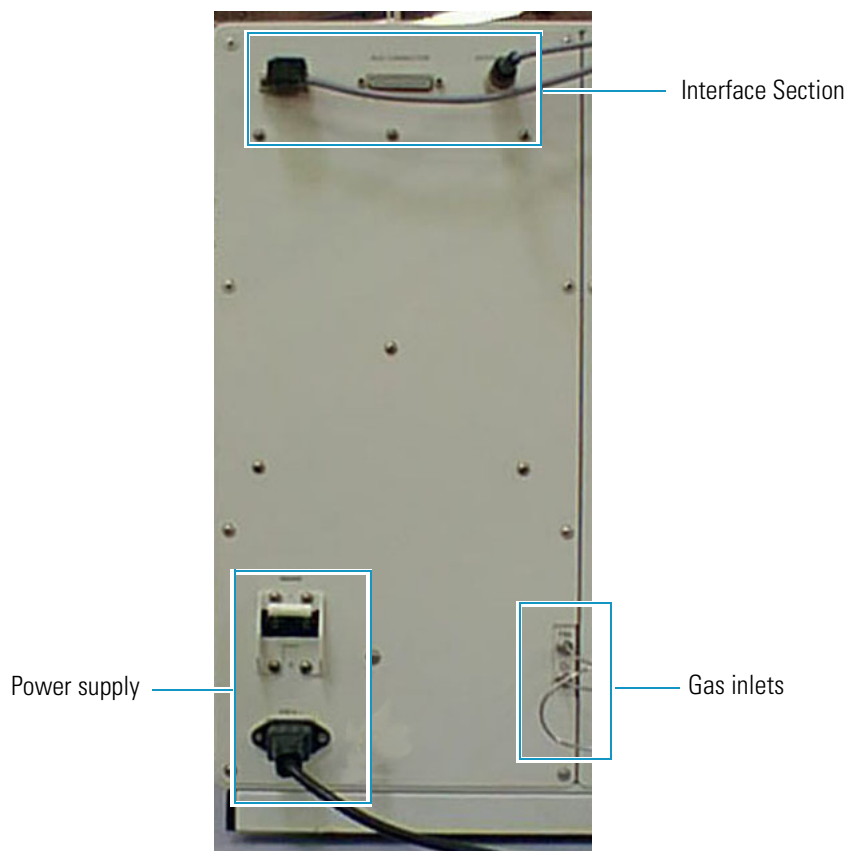
Board	Fuse	Type	Protection
TRF 1112	F1	F1A; IEC 127/I (5 x 20 mm)	Power supply to LEFT and RIGHT furnaces transformers
	F2	F0.315A; IEC 127/I (5 x 20 mm)	Fan
	F3	F1.6A; IEC 127/I (5 x 20 mm)	Main power (Breaker)
	F4	F1A; IEC 127/I (5 x 20 mm)	LEFT and RIGHT furnaces transformers
	F5	F0.315A; IEC 127/I (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 panel, shown in [Figure 12](#), is subdivided into three sections:

- interface
- power supply
- gas inlets

**Figure 12.** View of the Connections Panel



## Interface Section

It comprises:

- 9-pin connector marked RS 232 to dialog with the computer via serial line.
- 25-pin connector marked AUX CONNECTOR for the autosampler for liquids if required.
- 2-pin connector marked Autosampler for the MAS 200R autosampler for solids.

## Power Supply Section

It comprises:

- Breaker marked **MAINS** to switch the instrument on/off.  
Position **I** = instrument on.  
Position **O** = instrument off.
- 230 V; 50/60 Hz mains connector.

## Gas Supply Section

It comprises the gas inlet ports. Refer to paragraph “[Fittings for Gas Connections](#)” on [page 29](#).

## Transformers Compartment

Located in the right bottom part of the instrument, it is accessible from the rear panel by removing the relevant cover.

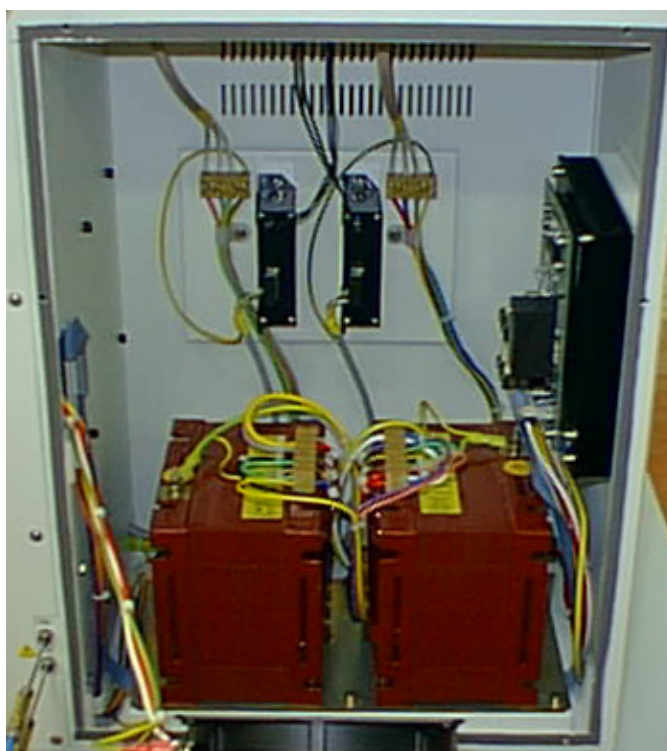
It contains the electrical devices to power the furnaces and control their temperature.



**CAUTION** Before opening the compartment, switch the instrument off and disconnect the power cord.

Figure 13 shows the devices contained in this compartment.

**Figure 13.** Transformer compartment internal view



## Devices for the Furnaces Control

The following [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.



**Table 9.** Description of the Devices Controlling the Furnaces, continued

Device	Function
SSR LEFT	Solid state relays contained in the AC 1112 board.
SSR RIGHT	Each SSR is coupled with a proper safety sensor, which detects any malfunction. The SSRs 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

The following [Table 10](#) reports the function of each device:

**Table 10.** Description of the Devices Supplying the Furnaces

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

## Synoptic Panel

This panel shows the instrument operating conditions, and it is located on the right side of the instrument front panel.


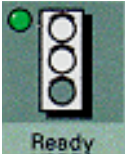







Each synoptic is provided with a LED, which lights up when the relevant function is active.

**Figure 14.** Synoptic panel



The following [Table 11](#) illustrates the meaning of each synoptic:

**Table 11.** Description of the synoptic

Synoptic	LED	Meaning
	Power On	When lit, the instrument is powered.
	Ready	When lit, the instrument is ready to run analyses.
	Run	When lit, an analysis is in progress.
	Stand By	When lit, the instrument is in stand-by condition. During this condition, gas flows are decreased to 10 ml/min, and the furnaces temperature is reduced to 50% of the set value.
	Wake Up	When lit, the instrument has been programmed for a timed automatic startup (Ready Condition).
	Furnace On	Two LEDs are provided, one for each furnace. When one is lit, the relevant furnace is powered.
	Oven On	When lit, the detector oven is powered.
	TCD	When lit, the detector filaments are powered.
	Safety Cut Off	It lights up when an alarm condition occurs.

## Autosamplers

The Flash 2000 elemental analyzer can be configured with the following autosamplers:

### MAS 200R Autosampler for Solid Samples

It is mounted directly on the connecting fitting of the concerned reaction tube.

It consists of:

- Anodized aluminium block provided on the left with fittings for carrier gas and purge gas lines connection.
- 32-position sample-holding tray.  
Up to 4 optional trays are available to accommodate 125 samples.

**Note** When the analyzer is equipped with two MAS 200R autosamplers, it is possible to switch from a channel to the other by using a dedicated Switching Box. See [“To Install the MAS 200R Autosampler on the Flash 2000”](#) on page 200.

### NoBlank Sampling Device

This device, interposed between MAS 200R autosampler and Flash 2000 instrument, reduces the Nitrogen blank value due to the sampler movement.

The device is particularly suggested for Flash 2000 configuration coupled to IRMS instrument.

### AI 3000/AS 3000 II Autosampler for Liquid Samples

It is mounted on the analyzer by means of the appropriate support.

It consists of:

- Sampling unit
- 8-position (AI 3000) or 105-position (AS 3000) sample tray

### CM2 Manual Sampler

It is mounted directly on the connecting fitting of the concerned reaction tube. It consists of:

- Base block provided with a hole for the manual introduction of samples.

### Manual Injection Device for Liquids

It allows the direct injection of a liquid sample into the reactor using a syringe.

# Pneumatic Circuits

This chapter describes the pneumatic circuit of each instrument configuration in the pre-run condition.

## Contents

- [Introduction](#)
- [Pressure Regulators](#)
- [EFC-t Module](#)
- [Pneumatic Circuit for CHNS-O and CHN-O Configurations](#)
- [Pneumatic Circuit for CHN and CHNS Configurations](#)
- [Pneumatic Circuit for O, S and NCS Configurations](#)
- [Pneumatic Circuit for NC, NC-Soils, NC-Filters and NC-Sediments Configurations](#)
- [Pneumatic Circuit for N, N Lubricant, N/Protein and N-Brew Configurations](#)
- [Pneumatic Circuit for NC-IRMS Configuration](#)
- [Pneumatic Circuit for Flash HT Configurations](#)

## Introduction

Each of the instrument configurations of the Flash 2000 Elemental Analyzer works with a different analytical technique, and therefore has a different pneumatic circuit.

All pneumatic circuits have the following common components:

- The EFC Electronic Flow Controller for gases.
- Inlet gases pressure regulators and relevant gauges.
- The TCD Thermal Conductivity Detector.

According to each analytical configuration, the pneumatic circuit can comprise:

- One or two reactors.
- One or two gas chromatographic columns.

- One or two adsorption filters or none.

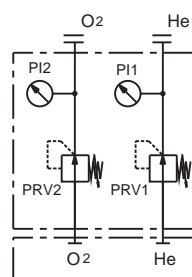
The filling of reactors and absorbent filters, and the type of analytical columns vary according to the instrument configuration. For more details refer to [Chapter 7, “Preparing the Sample,”](#)

**IMPORTANT** All the pneumatic circuits described in this chapter are represented in the PreRun condition.

## Pressure Regulators

Pressure regulators, located in the detector compartment and schematically shown in [Figure 15](#), allow the manual adjustment of the Helium and Oxygen inlet pressure.

**Figure 15.** Pressure Regulators



Pressure regulators are common to all analyzers. They consist of the following components. See [Figure 12](#):

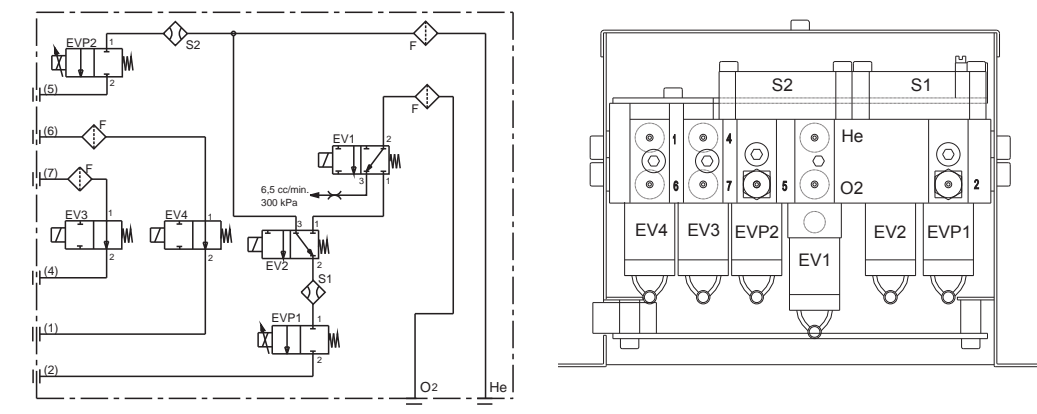
**Table 12.** Pressure regulators

Component	Description and function
He	Inlet port for Helium.
O2	Inlet port for Oxygen
PRV1	Helium pressure regulator
PI1	Helium pressure gauge
PRV2	Oxygen pressure regulator
PI2	Oxygen pressure gauge

## EFC-t Module

The EFC-t module, schematically shown in [Figure 16](#) is common to all analyzers. It consists of the following components. See [Table 13](#).

**Figure 16.** Thermoregulated EFC-t module



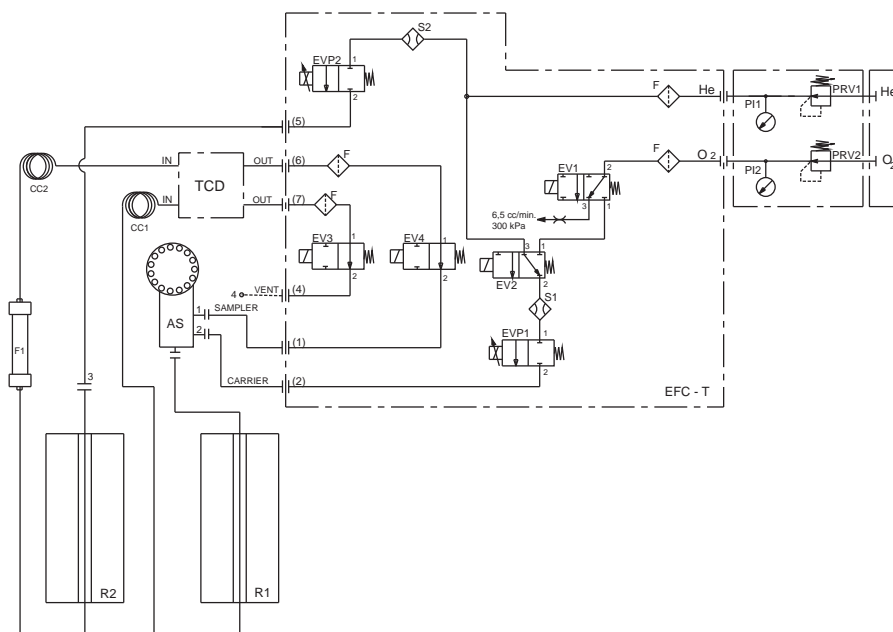
**Table 13.** Parts of the EFC-t module

Component	Description
He	Inlet port for Helium.
O2	Inlet port for Oxygen.
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 gas is exhausted to the outside through the Vent port. 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 is used to eliminate air from the MAS 200R autosampler. The valve is closed during the leak test.
S1	Electronic flow sensor for Helium as carrier gas and Oxygen during the sampling stage. It cooperates with the EVP1 electronic controller (proportional valve).
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 flowrates of gases according to the flow values set.
EVP2	Electronic flow controller for Helium as reference gas to control the flowrate according to the required flow value.

## Pneumatic Circuit for CHNS-O and CHN-O Configurations

The pneumatic diagram shown in [Figure 17](#) is common to CHNS-O and CHN-O configurations.

**Figure 17.** Pneumatic Circuit for CHNS-O and CHN-O Configurations



It comprises the following components:

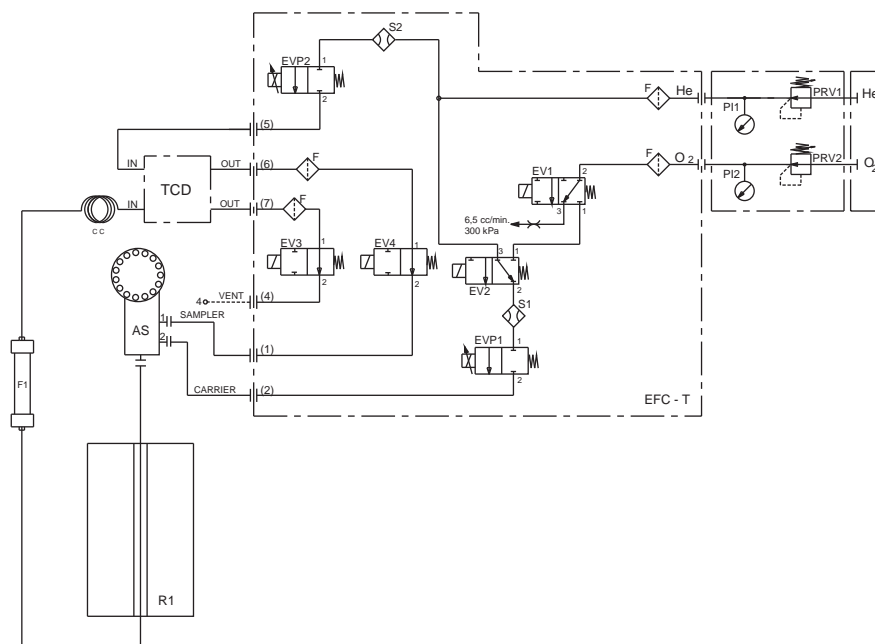
**Table 14.** Components of the Pneumatic Circuits for CHNS-O and CHN-O

Component	Description
EV1-EV2 EV3-EV4 EVP1-EVP2 S1-S2	They constitute the EFC module. Refer to paragraph “EFC-t Module” on <a href="#">page 43</a> .
AS	Autosampler
R1	Quartz reactor for CHNS determination
R2	Quartz reactor for Oxygen determination
F1	Adsorption filter
CC1	Gas chromatographic column for CHNS determination
CC2	Gas chromatographic column for Oxygen determination
TCD	TCD thermal conductivity detector

## Pneumatic Circuit for CHN and CHNS Configurations

The pneumatic diagram shown in [Figure 18](#) is common to CHN and CHNS configurations.

**Figure 18.** Pneumatic Circuit for CHNS and CHN Configurations



It comprises the following components:

**Table 15.** Components of the Pneumatic Circuits for CHNS and CHN

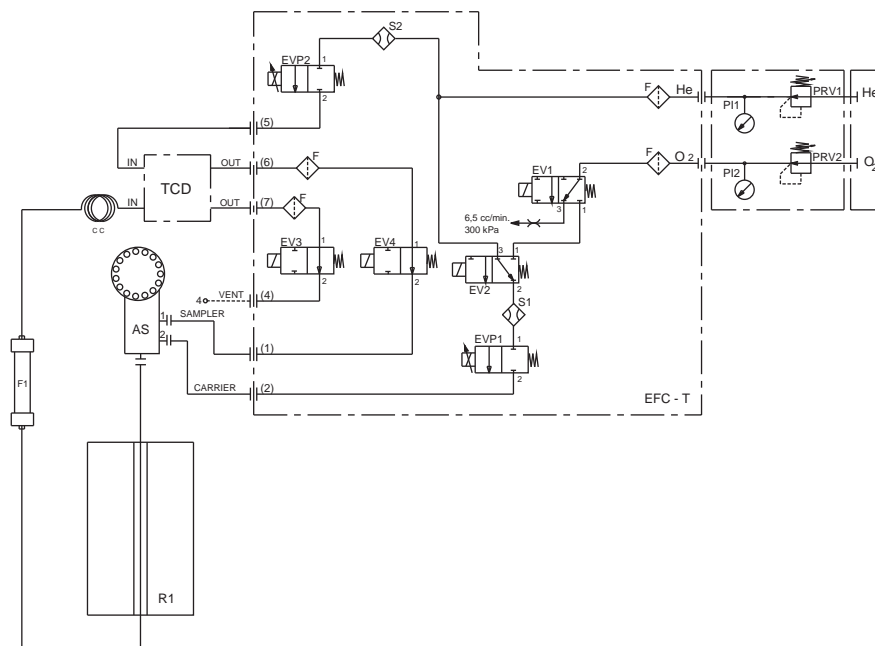
Component	Description
EV1-EV2 EV3-EV4 EVP1-EVP2 S1-S2	They constitute the EFC module. Refer to paragraph “EFC-t Module” on <a href="#">page 43</a> .
AS	Autosampler
R1	Reactor
F1	Adsorption filter
CC1	Gas chromatographic column
TCD	TCD thermal conductivity detector



## Pneumatic Circuit for O, S and NCS Configurations

The pneumatic diagram shown in [Figure 19](#) is common to O, S and NCS configurations. Pneumatic Circuit for O, S and NCS Configurations.

**Figure 19.** Pneumatic Circuit for O, S and NCS Configurations



It comprises the following components:

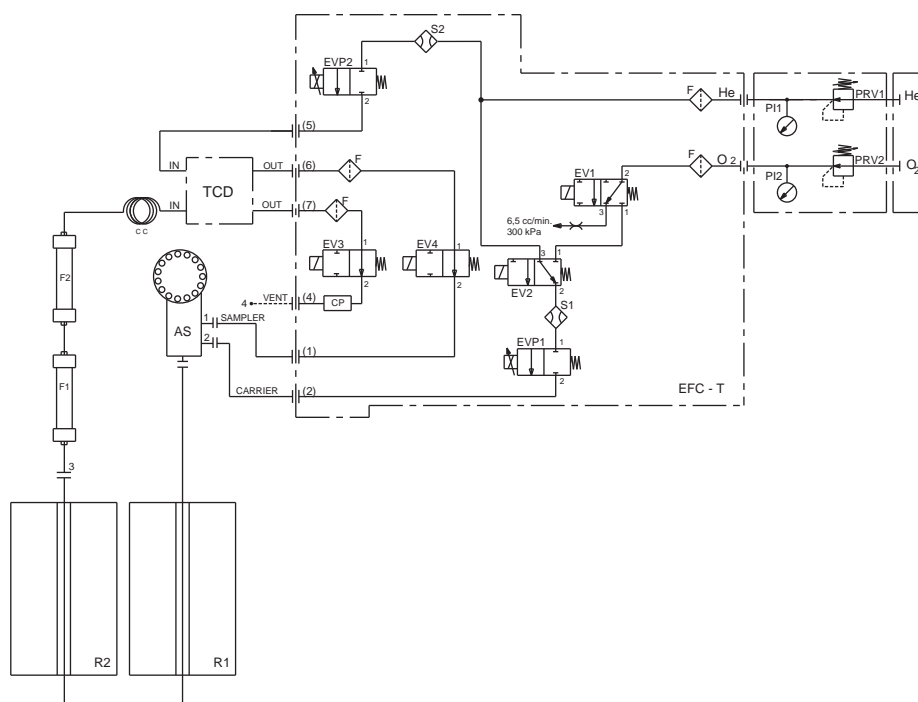
**Table 16.** Components of the Pneumatic Circuit for O, S and NCS

Component	Description
EV1-EV2 EV3-EV4 EVP1-EVP2 S1-S2	They constitute the EFC module. Refer to paragraph “EFC-t Module” on <a href="#">page 43</a> .
AS	Autosampler
R1	Reactor
F1	Adsorption filter
CC1	Gas chromatographic column
TCD	TCD thermal conductivity detector

## Pneumatic Circuit for NC, NC-Soils, NC-Filters and NC-Sediments Configurations

The pneumatic diagram shown in [Figure 20](#) is common to NC, NC-Soil, NC-Filters and NC-Sediments configurations.

**Figure 20.** Pneumatic Circuit for NC, NC-Soils, NC-Filters and NC-Sediments Configurations



It comprises the following components:

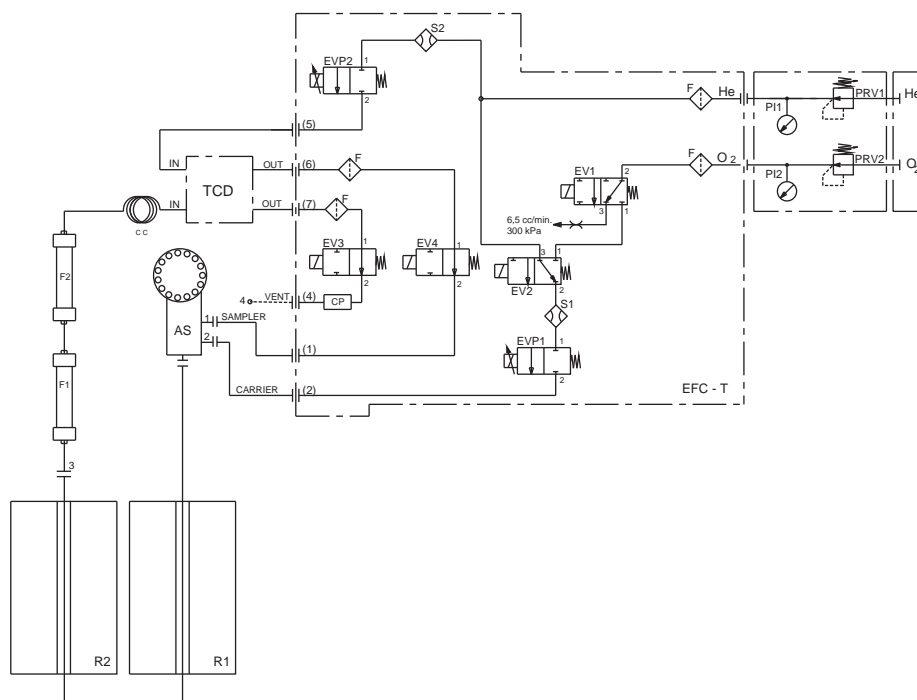
**Table 17.** Components of the Pneumatic Circuit for NC, NC-Soils, NC-Filters, NC-Sediments

Component	Description
EV1-EV2 EV3-EV4 EVP1-EVP2 S1-S2	They constitute the EFC module. Refer to paragraph “EFC-t Module” on <a href="#">page 43</a> .
AS	Autosampler
R1	Oxidation reactor
R2	Reduction reactor
F1	Adsorption filter for carbon dioxide
F2	Adsorption filter for water
CC1	Gas chromatographic column
TCD	TCD thermal conductivity detector

# Pneumatic Circuit for N, N Lubricant, N/Protein and N-Brew Configurations

The pneumatic diagram shown in Figure 21 is common to N, N Lubricant N/Protein and N-Brew configurations.

**Figure 21.** Pneumatic Circuit for N, N Lubricant N/Protein and N-Brew Configurations



It comprises the following components:

**Table 18.** Components of the Pneumatic Circuit for N, N Lubricant, N/Protein and N-Brew

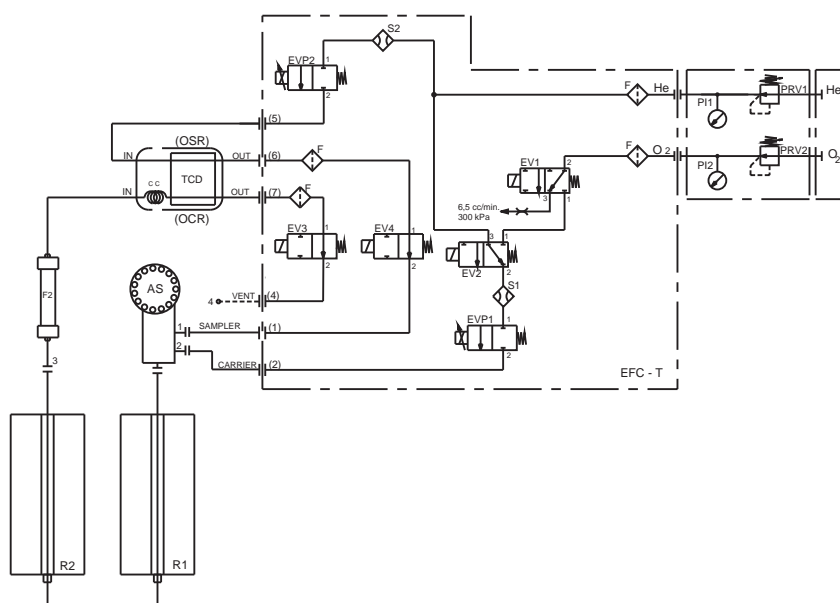
Component	Description
EV1-EV2 EV3-EV4 EVP1-EVP2 S1-S2	They constitute the EFC module. Refer to paragraph “EFC-t Module” on page 43.
AS	Autosampler
R1	Oxidation reactor
R2	Reduction reactor
F1	Adsorption filter for carbon dioxide
F2	Adsorption filter for water
CC	Gas chromatographic column
TCD	TCD thermal conductivity detector
CP	Pressure stabilizing cylinder

**CAUTION** The pressure stabilizing cylinder CP is not present in the N Configuration. It avoids the introduction of air from the point 4 during the extended combustion of samples with very high weigh.

## Pneumatic Circuit for NC-IRMS Configuration

The pneumatic diagram shown in Figure 22 is common to N and C configurations with the IRMS detector.

**Figure 22.** Pneumatic Circuit for NC-IRMS Configuration



It comprises the following components:

**Table 19.** Components of the Pneumatic Circuit for NC-IRMS Determinations

Component	Description
EV1-EV2 EV3-EV4 EVP1-EVP2 S1-S2	They constitute the EFC module. Refer to paragraph <Pantone>EFC-t Module page 43.
AS	Autosampler
R2	Oxidation reactor
F2	Adsorption filter for water
CC	Gas chromatographic column
TCD	TCD thermal conductivity detector
(OSR-OCR)	Oven without TCD detector - Oven with TCD detector

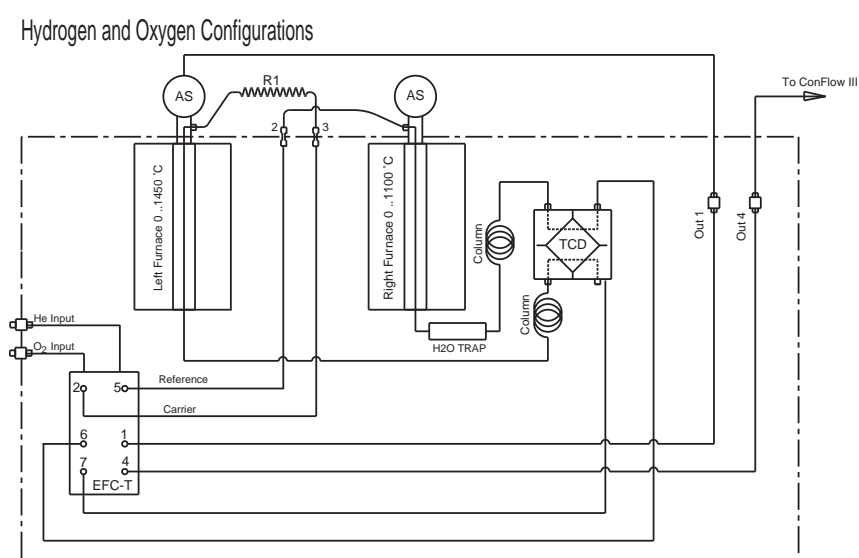
## Pneumatic Circuit for Flash HT Configurations

The left furnace at high temperature (1450 °C) is used for the determination of Oxygen and Hydrogen by pyrolysis while the right furnace, at 1000 °C, is used for the determination of Nitrogen and Carbon (or Sulfur).

### OH Configuration

The pneumatic diagram is shown in [Figure 23](#). The OH configuration use the left furnace at high temperature.

**Figure 23.** Pneumatic Circuit Flash HT for OH Configuration



It comprises the following components:

**Table 20.** Components of the Pneumatic Circuit for O and H Determinations

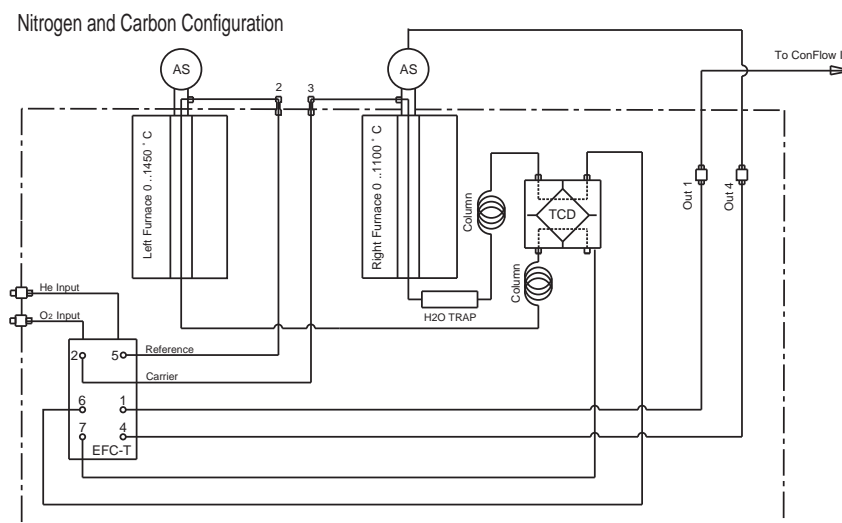
Component	Description
EFC-t	Electronic Flow Controller Termoregulated module
AS	Autosampler
Left Furnace	Left furnace at high temperature (1450 °C)
Right Furnace	Right furnace (1100 °C)
H <sub>2</sub> O Trap	Filter for water
TCD	Thermal Conductivity Detector

**Note** For details about this configuration refer to the Flash HT User Guide (PN 317 082 71).

## NC Configuration

The pneumatic diagram is shown in Figure 24. The NC configuration uses the right furnace.

**Figure 24.** Pneumatic Circuit Flash HT for NC Configuration.



It comprises the following components:

**Table 21.** Components of the Pneumatic Circuit for NC Determinations

Component	Description
EFC-t	Electronic Flow Controller Thermoregulated module
AS	Autosampler
Left Furnace	Left furnace at high temperature (1450 °C)
Right Furnace	Right furnace (1100 °C)
H <sub>2</sub> O Trap	Filter for water
TCD	Thermal Conductivity Detector

**Note** For details about this configuration refer to the Flash 2000 HT User Guide (PN 317 082 71).

## **4 Pneumatic Circuits**

Pneumatic Circuit for Flash HT Configurations

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Part

1

# Preparation

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# Preparation of Reactors and Adsorption Filters

This chapter provides instructions for the preparation of the reactors and the adsorption filters, and it also reports the types of analytical columns currently used.

## Contents

- [Introduction](#)
- [Filling Materials](#)
- [Introduction to the Preparation of Reactors and Filters](#)
- [Preparing the Reactors](#)
- [Preparing the Adsorption Filters](#)

## Introduction

Each instrument configuration requires its own dedicated reactors, adsorption filters and analytical columns. Except for a few “ready for use” reactors, the reactors and the adsorption filters must be prepared by the user.

## Reactors

The reactors can be quartz tubes or special steel tubes.

- The quartz and special steel tubes have a conical bottom end.
- The special steel reactors have their top end provided with two through-holes.

The filling materials used vary according to the analytical determination required. Refer to paragraph “[Filling Materials](#)” on [page 56](#).

**Note** The special steel reactors, used for combustion, require the presence of a crucible. For more details, refer to the operating sequence on [page 78](#).

## Adsorption Filters

They can be glass or Plexiglas filters.

The filling materials used vary according to the analytical determination required. Refer to paragraph “Filling Materials” on page 56.

## Gas Chromatographic Columns

The columns are made of steel, except in N, N/Protein and N-Brew configurations, which require PTFE columns.

**Note** Gas chromatographic columns are “ready for use”, and therefore they do not require any preparation.

The following Table 22 reports the characteristics of reactors, filters and gas chromatographic columns required for each analytical determination.

**Table 22.** Characteristic of reactors, filters and gas chromatographic columns

Characteristics					Analytical Determination																		
Material	Length (cm)	OD (mm)	ID (mm)		CHNS	CHN	NCS	S (TCD)	S (FPD)	O	N	N Lubricant	N/Protein	N-Brew	NC	NC-Soils	NC-Sediments	NC-Filters	IRMS (NC)	HT (NC)	HT (O/H)		
<b>Reactors</b>	Quartz	45	18	14	X	X	X	X	X	X	X				X	X	X	X	X	X	X		
	Special Steel	45	25	23								X	X	X			X	X	X				
<b>Filters</b>	Glass	11	10	8			X	X		X					X	X	X	X					
	Plexiglas	23	30	22							X	X	X	X					X	X			
<b>Columns</b>	Steel	100	6	5						X												X	
		200	6	5											X	X	X	X					
		300	6	5															X	X			
	PTFE	15	6	4					X														
		50	8	6						X	X												
		80	6	4				X															
		100	8	6								X		X									
		200	6	5		X	X	X															

## Filling Materials

The following table reports the materials used to fill reactors, adsorption filters and gas chromatographic columns.

**Table 23.** Material required for filling reactors, filters and chromatographic columns

Characteristics		Analytical Determination																
		CHNS	CHN	NCS	S (TCD)	S (FPD)	O	N	N Lubricant	N/Protein	N-Brew	NC	NC-Soils	NC-Sediments	NC-Filters	IRMS (NC)	HT (NC)	HT (O/H)
<b>Reactors</b>	Quartz Wool	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	
	Electrolytic Copper	x		x	x	x												
	Copper Oxide	x		x	x	x		x				x						
	Reduced Copper		x					x	x	x	x	x	x	x	x	x	x	x
	Chromium Oxide		x													x	x	
	Silvered Cobaltous/Cobaltic Oxide		x					x				x				x	x	
	Quartz Chips						x											
	Metallized Carbon						x											
	Oxidation Catalyst								x	x	x		x	x	x			
<b>Filters</b>	Quartz Wool			x	x	x	x	x	x	x	x	x	x	x				
	Soda Lime						x	x	x	x	x							
	Molecular Sieves 3 Angstrom							x	x	x	x							
	Magnesium Procreate (Andorran)			x	x	x						x	x	x	x	x	x	
	Silica Gel							x	x	x	x							
<b>Columns</b>	Multi-separation Column (PTFE)		x															
	Multi-separation Column (S.Steel)											x	x	x	x			
	Oxygen Separation Column						x											
	Nitrogen Separation Column (50 cm)							x		x								
	Nitrogen Separation Column (100 cm)								x		x							
	CHNS/NCS Packed Column	x		x														
	Sulphur Separation Column				x	x												
	IRMS Separation Column															x	x	x

## Introduction to the Preparation of Reactors and Filters

The preparation of reactors and adsorption filters must be done according to the specifications and quantities reported in the table referring to each instrument configuration. If required, also refer to [Chapter 4, “Pneumatic Circuits,”](#) .

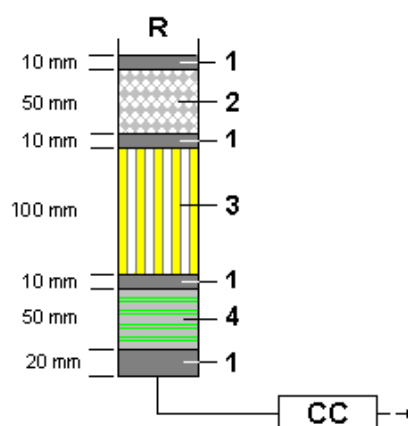
## CHN Configuration

The following [Table 24](#) reports the characteristics of the components required for **CHN determination**, and the type and size of the filling materials to be used for a proper preparation of the reactor.

**Table 24.** Components required for CHN determination

Reference	Component	Characteristic	Filling material
R	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Copper Reduced 3. Chromium Oxide 4. Silvered Copulates/Copulated Oxide
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meters <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the oxidation/reduction reactor. In this case it is necessary to eliminate the Quartz Wool between Chromium Oxide and high quality Copper reduced and between the high quality Copper reduced and Copulates-Cobalt Oxide.

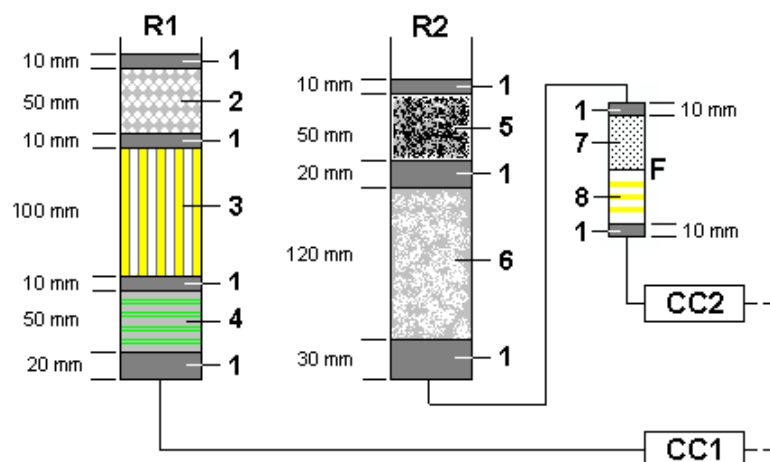
## CHN-O Configuration

The following [Table 25](#) reports the characteristics of the components required for **CHN-O determination**, the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filter.

**Table 25.** Components required for CHN-O determination

Reference	Component	Characteristic	Determination	Filling material
R1	Reactor	<i>Material:</i> Quartz	CHN	1. Quartz Wool 2. Copper Reduced 3. Chromium Oxide 4. Silvered Cobaltous/Colbaltic Oxide
CC1	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meter <i>Diameter:</i> 6 x 5 mm	CHN	---
R2	Reactor	<i>Material:</i> Quartz	Oxygen	1. Quartz Wool 5. Quartz Turnings 6. Nickel Plated Carbon
F	Adsorption filter	<i>Material:</i> Quartz	Oxygen	1. Quartz Wool 7. Soda Lime 8. Magnesium Perchlorate (Anhydron)
CC2	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 1 meter <i>Diameter:</i> 6 x 5 mm	Oxygen	---

### Size of the Filling Material



## 5 Preparation of Reactors and Adsorption Filters

Introduction to the Preparation of Reactors and Filters

### CHN Determination



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the oxidation/reduction reactor. In this case it is necessary to eliminate the Quartz Wool between Chromium Oxide and high quality Copper reduced and between the high quality Copper reduced and Cobaltous-Cobaltic Oxide.

### Oxygen Determination



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the reactor of pyrolysis. In this case it is necessary to reduce from 30 mm to 20 mm the Quartz Wool in the lower section of the reactor

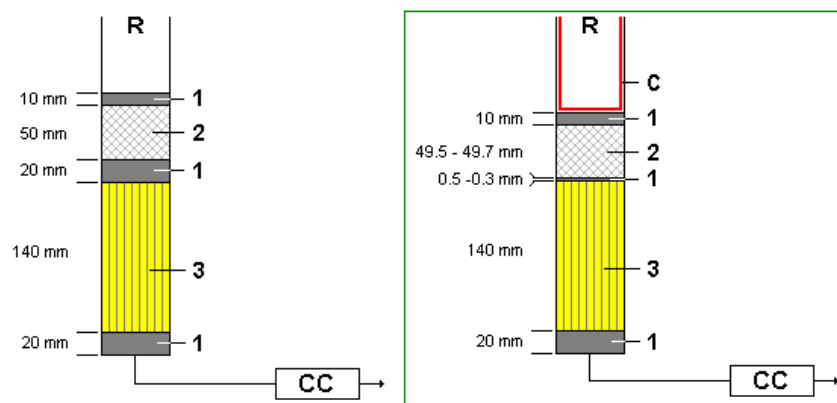
## CHNS Configuration

The following [Table 26](#) reports the characteristics of the components required for **CHNS determination**, and the type and size of the filling materials to be used for a proper preparation of the reactor.

**Table 26.** Components required for CHNS determination

Reference	Component	Characteristic	Filling material
R	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Electrolytic Copper 3. Copper Oxide
C	Crucible	<i>Material:</i> Quartz	---
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meters <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the oxidation/reduction reactor. In this case it is necessary to reduce the Quartz Wool between Copper Oxide and Electrolytic Copper up to obtain a thin layer and to reduce the Copper Oxide proportionally as shown in the previous figure.

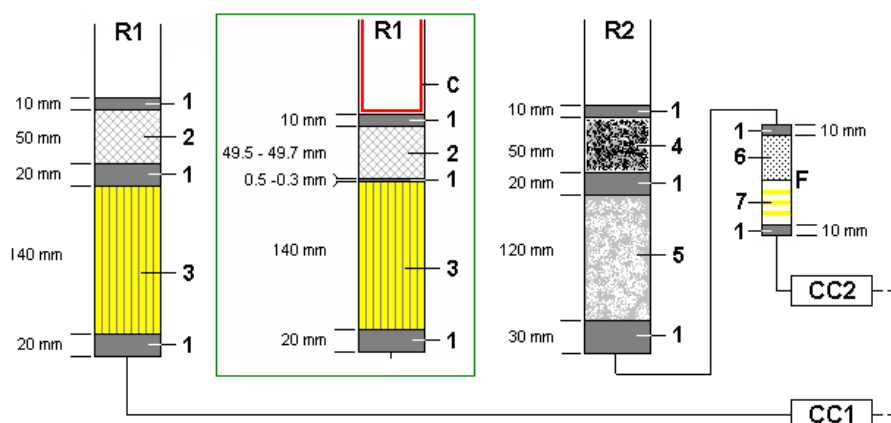
## CHNS-O Configuration

The following [Table 27](#) reports the characteristics of the components required for **CHNS-O determination**, and the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filter.

**Table 27.** Components required for CHNS-O determination

Reference	Component	Characteristic	Determination	Filling material
R1	Reactor	<i>Material:</i> Quartz	CHNS	1. Quartz Wool 2. Copper Oxide 3. Electrolytic Copper
C	Crucible	<i>Material:</i> Quartz	---	---
CC1	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meters <i>Diameter:</i> 6 x 5 mm	CHNS	---
R2	Reactor	<i>Material:</i> Quartz	Oxygen	1. Quartz Wool 4. Nickel Plated Carbon 5. Quartz Turning
F	Adsorption filter	<i>Material:</i> Glass	Oxygen	1. Quartz Wool 6. Soda Lime 7. Magnesium Perchlorate (Anhydrous)
CC2	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 1 meter <i>Diameter:</i> 6 x 5 mm	Oxygen	---

### Size of the Filling Material





## CHNS Determination



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the oxidation/reduction reactor. In this case it is necessary to reduce the Quartz Wool between Copper Oxide and Electrolytic Copper up to obtain a thin layer and to reduce the Copper Oxide proportionally as shown in the previous figure.

## Oxygen Determination



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the reactor of pyrolysis. In this case it is necessary to reduce from 30 mm to 20 mm the Quartz Wool in the lower section of the reactor.

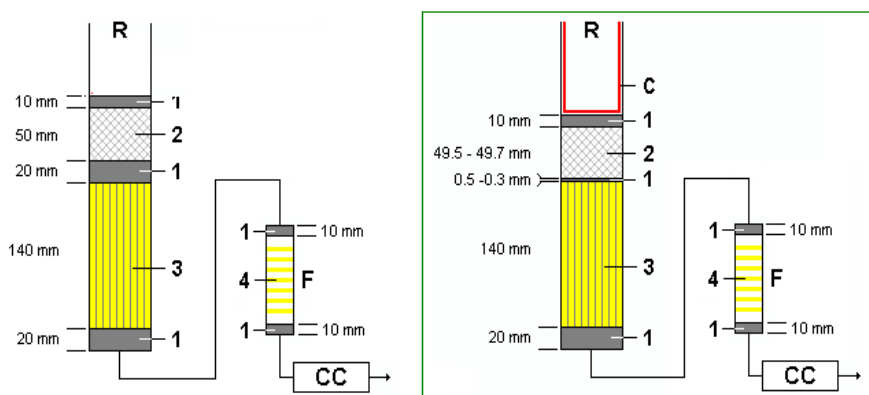
## S (Sulfur) Configuration

The following [Table 28](#) reports the characteristics of the components required for **S determination**, and the type and size of filling materials to be used for a proper preparation of reactor and adsorption filter.

**Table 28.** Components required for S (Sulfur) determination

Reference	Component	Characteristic	Filling materials
R	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Copper Oxide 3. Electrolytic Copper
C	Crucible	<i>Material:</i> Quartz	
F	Adsorption filter	<i>Material:</i> Glass	1. Quartz Wool 4. Magnesium Perchlorate (Anhydrous)
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 1 meter <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the oxidation/reduction reactor. In this case it is necessary to reduce the Quartz Wool between Copper Oxide and Electrolytic Copper up to obtain a thin layer and to reduce the Copper Oxide proportionally as shown in the previous figure.

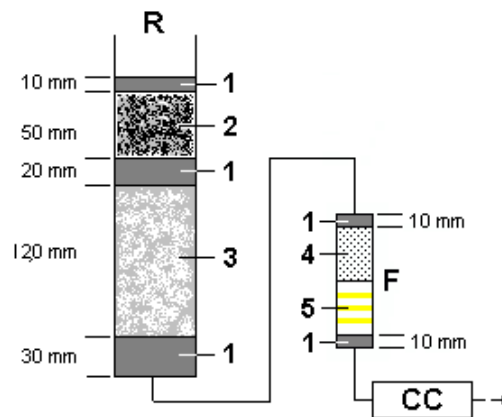
## O (Oxygen) Configuration

The following Table 29 reports the characteristics of the components required for O determination, and the type and size of the filling materials to be used for a proper preparation of reactor and adsorption filter.

**Table 29.** Components required for O (Oxygen) determination

Reference	Component	Characteristic	Filling materials
R	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Metallized Carbon 3. Quartz Turnings
F	Adsorption filter	<i>Material:</i> Glass	1. Quartz Wool 4. Soda Lime 5. Magnesium Perchlorate (Anhydron)
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 1 meter <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the reactor of pyrolysis. In this case it is necessary to reduce from 30 mm to 20 mm the Quartz Wool in the lower section of the reactor.

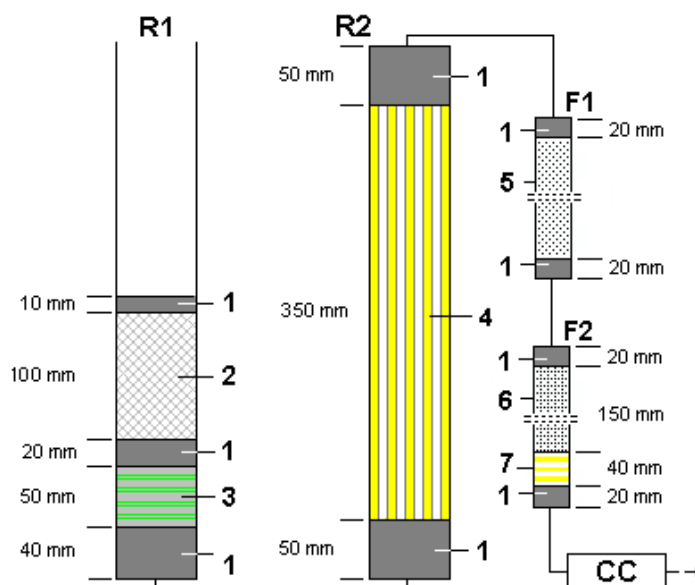
## N (Nitrogen) Configuration

The following [Table 30](#) reports the characteristics of the components required for **N determination**, and the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filters.

**Table 30.** Components required for N (Nitrogen) determination

Reference	Component	Characteristic	Filling materials
R1	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Copper Oxide 3. Silvered Cobaltous/Cobaltic Oxide
R2	Reactor	<i>Material:</i> Quartz	1 Quartz Wool 4. Copper Reduced
F1	Adsorption filter	<i>Material:</i> Plexiglas	1. Quartz Wool 5. Soda Lime
F2	Adsorption filter	<i>Material:</i> Plexiglas	1. Quartz Wool 6. Molecular Sieves 7. Silica Gel
CC	Gas chromatographic column	<i>Material:</i> PTFE <i>Length:</i> 50 cm <i>Diameter:</i> 8 x 6 mm	---

### Size of the Filling Material



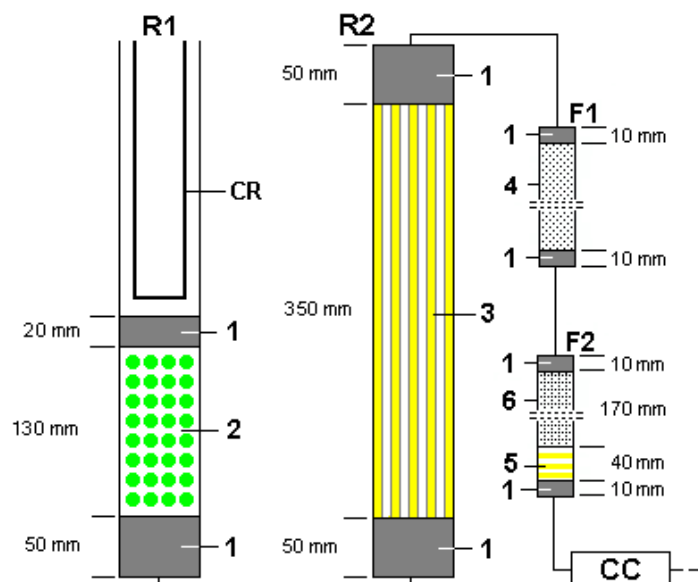
## N Lubricant Configurations

The following Table 31 reports the characteristics of the components required for N Lubricant determination, and the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filters.

**Table 31.** Components required for N Lubricant determination

Reference	Component	Characteristic	Filling materials
R1	Reactor (See note below)	<i>Material:</i> Special Steel	1. Quartz Wool 2. Oxidation Catalyst
R2	Reactor	<i>Material:</i> Special Steel	1. Quartz Wool 3. Copper Reduced
F1	Adsorption filter	<i>Material:</i> Plexiglas	1. Quartz Wool 4. Soda Lime
F2	Adsorption filter	<i>Material:</i> Plexiglas	1. Quartz Wool 5. Molecular Sieves 6. Silica Gel
CC	Gas chromatographic column	<i>Material:</i> PTFE <i>Length:</i> 100 cm <i>Diameter:</i> 8 x 6 mm	---
CR	Crucible	<i>Material:</i> HPAR	--

### Size of the Filling Material



**Note** The R1 combustion reactor requires the use of a crucible CR.

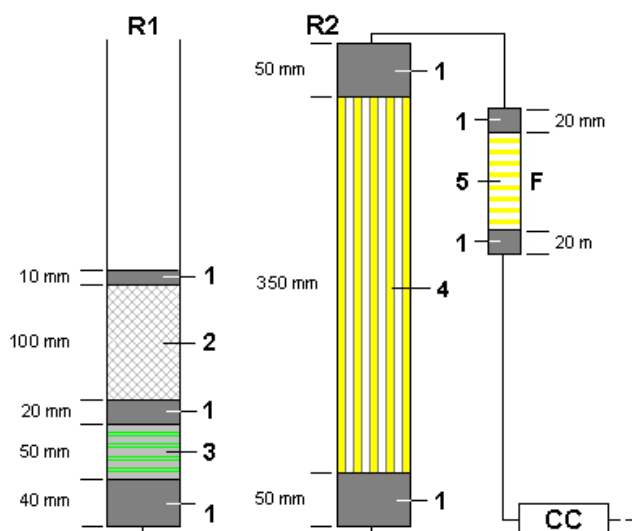
## NC Configuration

The following [Table 32](#) reports 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 reactors and adsorption filter.

**Table 32.** Components required for NC determination

Reference	Component	Characteristic	Filling materials
R1	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Copper Oxide 3. Silvered Cobaltous/Cobaltic Oxide
R2	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 4. Copper Reduced
F	Adsorption filter	<i>Material:</i> Glass	1. Quartz wool 5. Magnesium perchlorate (Anhydron)
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meters <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



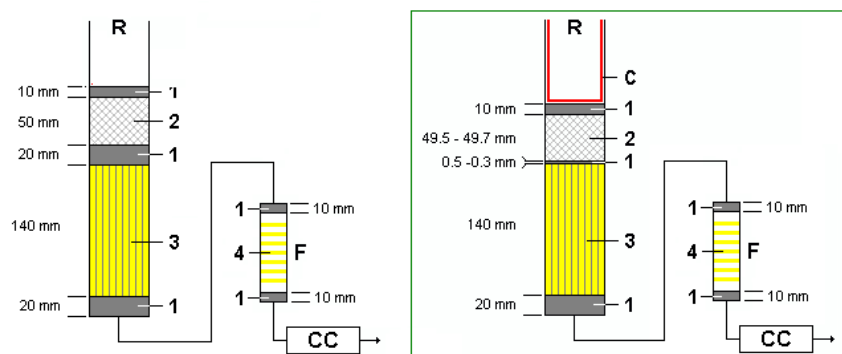
## NCS Configuration

The following Table 33 reports the characteristics of the components required for NCS determination, and the type and size of the filling materials to be used for a proper preparation of reactor and adsorption filter.

**Table 33.** Components required for NCS determination

Reference	Component	Characteristic	Filling materials
R	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. 3. Copper Oxide Electrolytic Copper
C	Crucible	<i>Material:</i> Quartz	---
F	Adsorption filter	<i>Material:</i> Glass	1. Quartz wool 4, Magnesium Perchlorate (Anhydron)
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meters <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



**IMPORTANT** If the sample to be analyzed presents particular characteristics (high presence of inorganic material) we suggest to insert the quartz crucible into the oxidation/reduction reactor. In this case it is necessary to reduce the Quartz Wool between Copper Oxide and Electrolytic Copper up to obtain a thin layer and to reduce the Copper Oxide proportionally as shown in the previous figure.

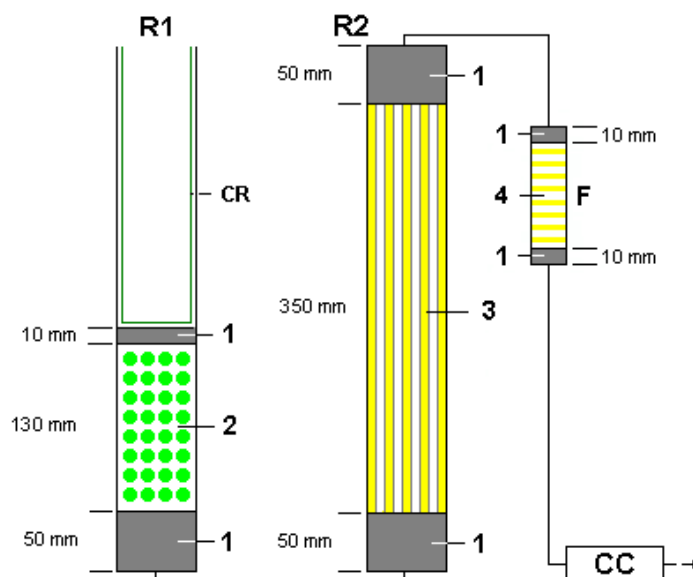
## NC-Soils, NC-Sediments, NC-Filters Configurations

The following [Table 34](#) reports the characteristics of the components required for **NC-Soils**, **NC-Sediments** and **NC-Filters determinations**, and the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filter.

**Table 34.** Components required for NC-Soils, NC-Sediments and NC-Filters Determinations

Reference	Component	Characteristic	Filling materials
R1	Reactor (See note below)	<i>Material:</i> Special Steel	1. Quartz Wool 2. Oxidation Catalyst
R2	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 3. Copper Reduced
F	Adsorption filter	<i>Material:</i> Glass	1. Quartz Wool 4. Magnesium Perchlorate (Anhydron)
CC	Gas chromatographic column	<i>Material:</i> Steel <i>Length:</i> 2 meters <i>Diameter:</i> 6 x 5 mm	---
CR	Crucible	<i>Material:</i> HPAR	---

### Size of the Filling Material



**Note** The R1 combustion reactor requires the use of a crucible CR.



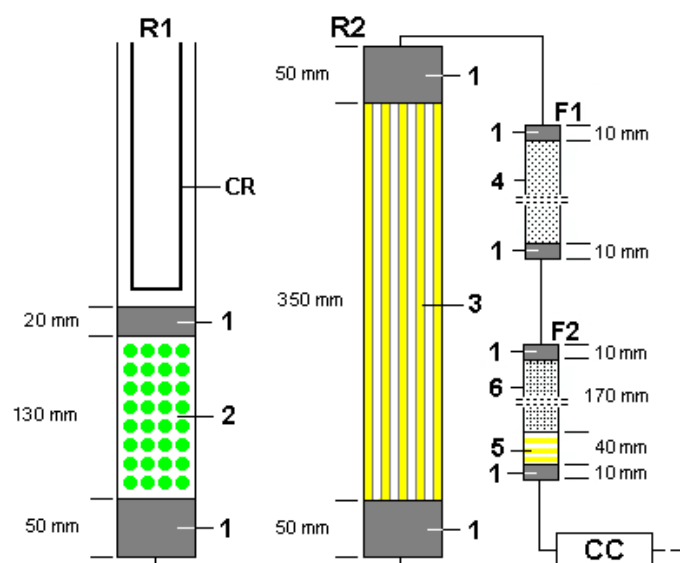
## N/Protein and N-Brew Configurations

The following [Table 35](#) reports the characteristics of the components required for **N/Protein** and **N-Brew determinations**, and the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filters.

**Table 35.** Components required for N/Protein and N-Brew Determinations

Reference	Component	Characteristic	Filling materials
R1	Reactor (See note below)	<i>Material:</i> Special Steel	1. Quartz Wool 2. Oxidation Catalyst
R2	Reactor	<i>Material:</i> Special Steel	1. Quartz Wool 3. Copper Reduced
F1	Adsorption filter	<i>Material:</i> Plexiglas	1. Quartz Wool 4. Soda Lime
F2	Adsorption filter	<i>Material:</i> Plexiglas	1. Quartz Wool 5. Molecular Sieves 6. Silica Gel
CC	Gas chromatographic column	<i>Material:</i> PTFE <i>Length:</i> 50 cm <i>Diameter:</i> 8 x 6 mm	<b>N/Protein</b>
		<i>Material:</i> PTFE <i>Length:</i> 100 cm <i>Diameter:</i> 8 x 6 mm	<b>N-Brew</b>

### Size of the Filling Material



**Note** The R1 combustion reactor requires the use of a crucible CR.

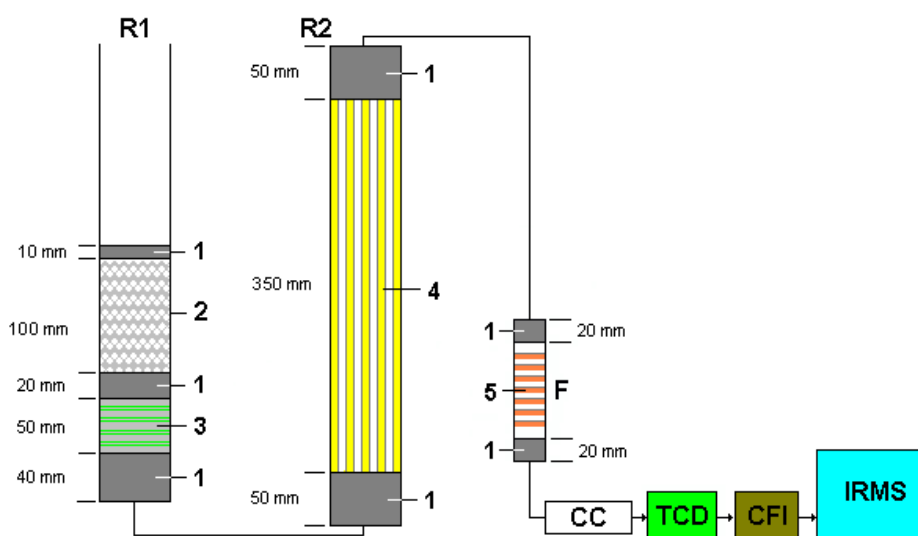
## NC Configuration with the Flash IRMS

The following [Table 36](#) reports the characteristics of the components required for **NC determinations** with the Flash IRMS, and the type and size of the filling materials to be used for a proper preparation of reactors and adsorption filter.

**Table 36.** Components required for NC Determination with the Flash IRMS

Reference	Component	Characteristic	Filling materials
R1	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Chromium Oxide 3. Silvered Cobaltous-Cobaltic Oxide
R2	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 4. Copper Reduced
F	Absorption Filter	<i>Material:</i> Plexiglas	---
CC	Gas chromatographic Column	<i>Material:</i> Stainless Steel <i>Length:</i> 3 meters <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



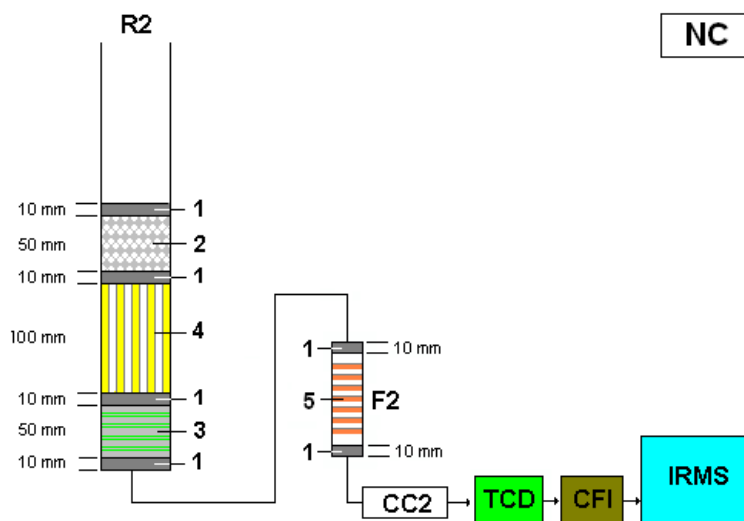
## NC Configuration with the Flash HT

The following [Table 37](#) report the characteristics of the components required for **NC determinations** with the Flash HT, and the type and size of the filling materials to be used for a proper preparation of reactor and adsorption filter.

**Table 37.** Components required for NC Determination with the Flash IRMS

Reference	Component	Characteristic	Filling materials
R2	Reactor	<i>Material:</i> Quartz	1. Quartz Wool 2. Chromium Oxide 3. Silvered Cobaltous-Cobaltic Oxide
F2	Absorption Filter	<i>Material:</i> Plexiglas	1. Quartz Wool 4. Magnesium Perchlorate (Anhydron)
CC2	Gas chromatographic Column	<i>Material:</i> Stainless Steel <i>Length:</i> 3 meters <i>Diameter:</i> 6 x 5 mm	---

### Size of the Filling Material



## 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 paragraph “[Introduction to the Preparation of Reactors and Filters](#)” on [page 57](#).

Please remember that:

- all reactors have a conical bottom end.
- special steel reactors have the upper end provided with two through-holes.



**WARNING** Before using the filling materials required for this operation, please read the hazard warnings and information reported in the Safety Data Sheets 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.

### ❖ To Fill the Quartz Reactor

The following procedure provides instructions for filling a quartz reactor.

#### Material Required

Quartz reactor

Compression rod

Filling material

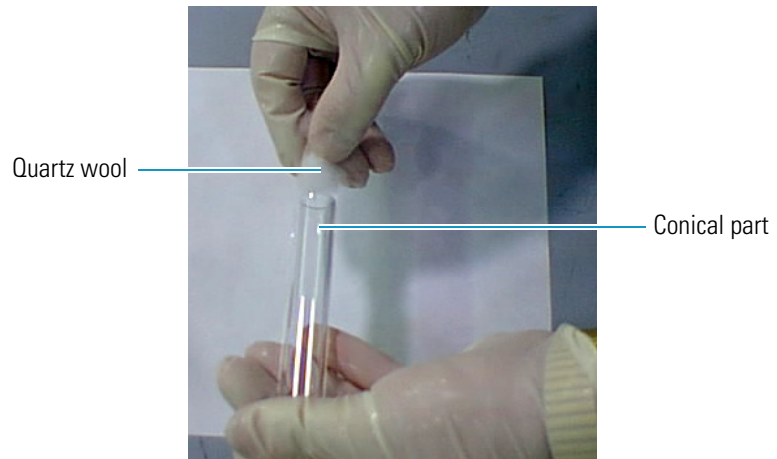
Use the following procedure to fill the quartz reactor:

1. Starting from the reactor bottom (conical end), introduce a sufficient amount of quartz wool to form the required layer, as shown in [Figure 25](#).

**Note** In the CHNS, NCS and S Configuration, Electrolytic Copper is used. The copper wires (about 14 cm long) must be inserted in the reactor through the conical part and pulled inside avoiding their braiding.

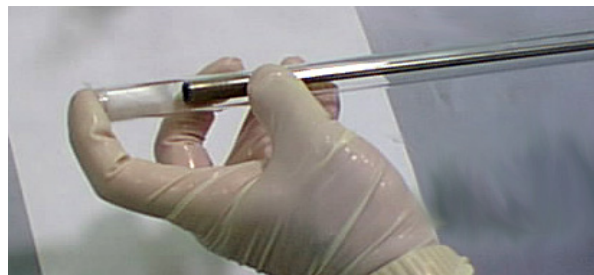
I

**Figure 25.** Introduction of quartz wool into the reactor conical end

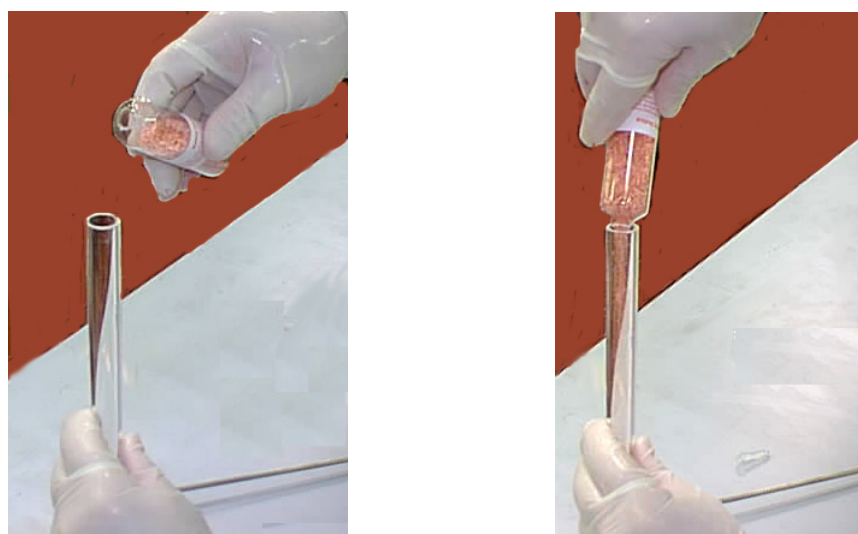


2. Plug with your finger the mouth of the reactor conical end. Gently press the quartz wool using the rod provided, as shown in [Figure 26](#).

**Figure 26.** Compression of quartz wool into the quartz reactor



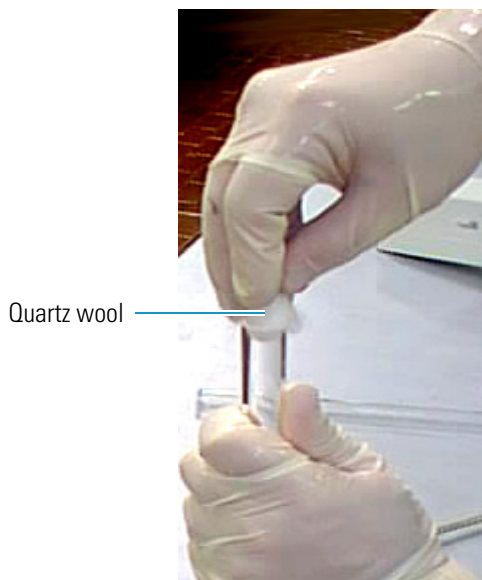
**Figure 27.** Filling of the quartz reactor



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 27](#), 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 28](#).

**Figure 28.** Introduction of quartz wool as last layer of the sequence



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

#### ❖ To Fill the Special Steel Reactor

The following operating procedure provides instructions for filling a steel reactor.

##### **Material required**

Special steel reactor

Compression rod

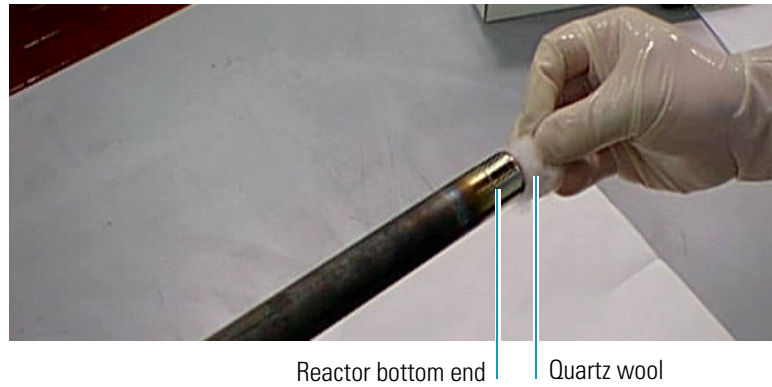
Filling materials

**Note** To measure the different layers, we recommend the use of the compression rod marking each time the measure on the reactor.

Use the following procedure to fill the steel reactor:

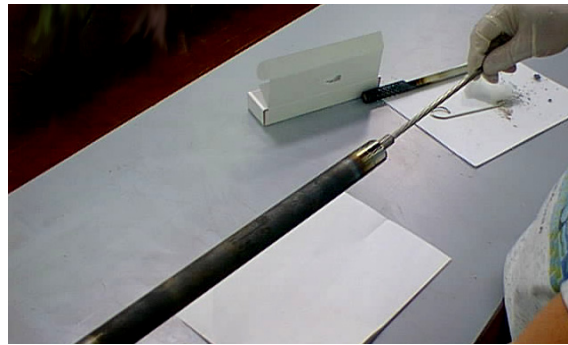
1. Introduce into the bottom end of the reactor a sufficient amount of quartz wool to form the required layer, as shown in [Figure 29](#).

**Figure 29.** Introduction of quartz wool into the bottom end of the reactor



2. Plug with your finger the mouth of the reactor bottom end. Gently press the quartz wool using the rod provided, as shown in [Figure 30](#).

**Figure 30.** Compression of quartz wool into the special steel reactor



3. Turn the bottom end of the reactor downward and delicately rest it onto the workbench.

**Figure 31.** Filling of the special steel reactor and compression of materials



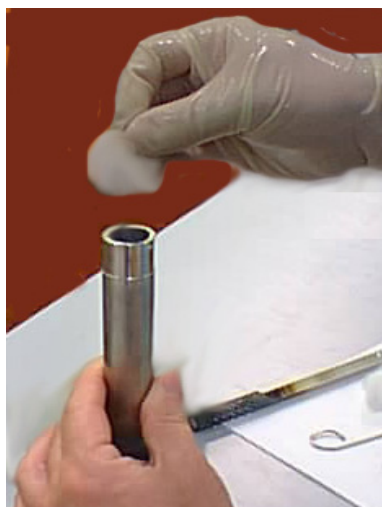
4. Pour sequentially the required filling materials into the reactor, ensuring that each layer has the indicated size.

**Note** When the oxidation catalyst is used (refer to [Table 23](#) on [page 57](#)), it must be introduced into the reactor homogeneously.

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 32](#).

**Figure 32.** Introduction of quartz wool as last layer of the sequence



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

#### ❖ To Prepare the Crucible

The following operating procedure provides instructions for the preparation of the crucible you will use with the special steel reactors required for combustion.

#### Material required

Quartz wool

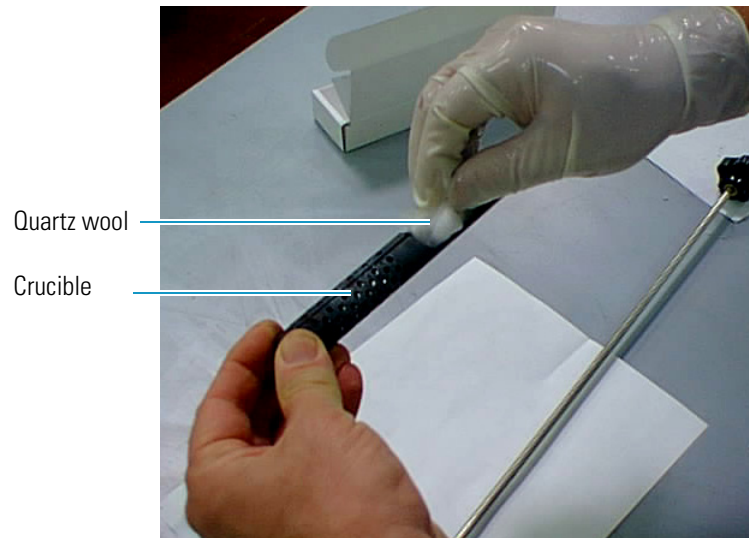
Compression rod

Use the following procedure to prepare the crucible:

1. Hold the crucible as shown in [Figure 33](#). Introduce into the bottom end of the crucible a sufficient quantity of quartz wool to form a 2 cm layer.

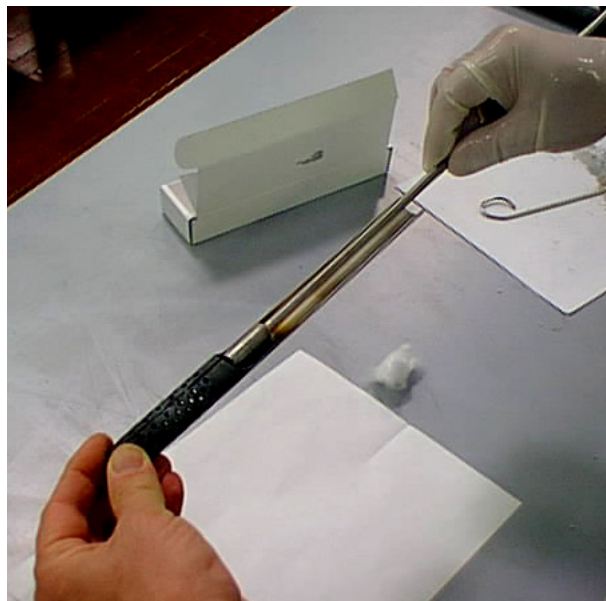


**Figure 33.** Introduction of quartz wool into the crucible



2. Gently press the quartz wool using the rod provided, as shown in [Figure 34](#).

**Figure 34.** Compression of quartz wool into the crucible



## Preparing the Adsorption Filters

According to the instrument configurations, 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 paragraph [“Introduction to the Preparation of Reactors and Filters”](#) on [page 57](#).

According to the analytical configuration required, the following adsorption filters can be used:

- large filter (Plexiglas)
- small filter (Glass)



**WARNING** Before using the filling materials required for this operation, please read the hazard warnings and information reported in the Safety Data Sheets 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.

#### ❖ To Fill the Adsorption Filter

The following procedure provides instructions for filling an adsorption filter.

##### Material required

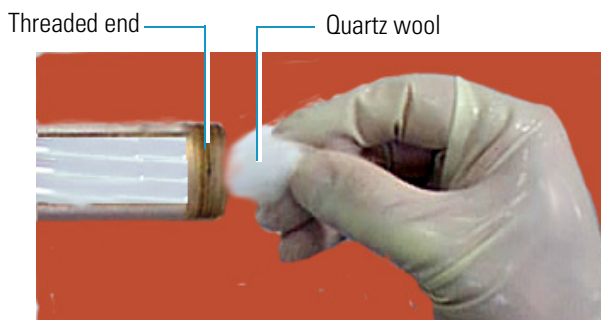
Glass or Plexiglas filter according to the instrument configuration

Compression rod

Filling materials

Introduce into either of the tube ends a sufficient amount of quartz wool to form the required layer as shown in [Figure 35](#).

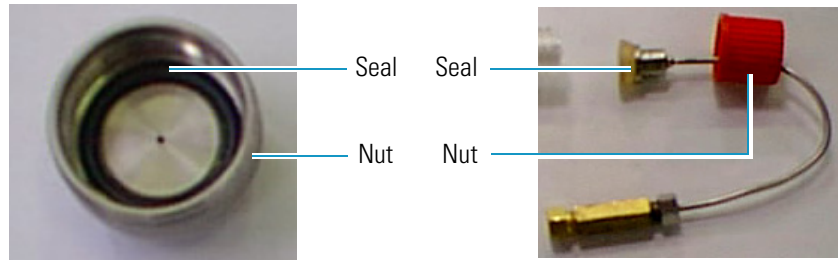
**Figure 35.** Introduction of quartz wool into the tube



3. While plugging the tube mouth with your hand, press gently the quartz wool using the rod provided.

4. Screw the nut complete with its seal onto the threaded mouth, as shown in [Figure 36](#).

**Figure 36.** Nuts and seals for adsorption filters

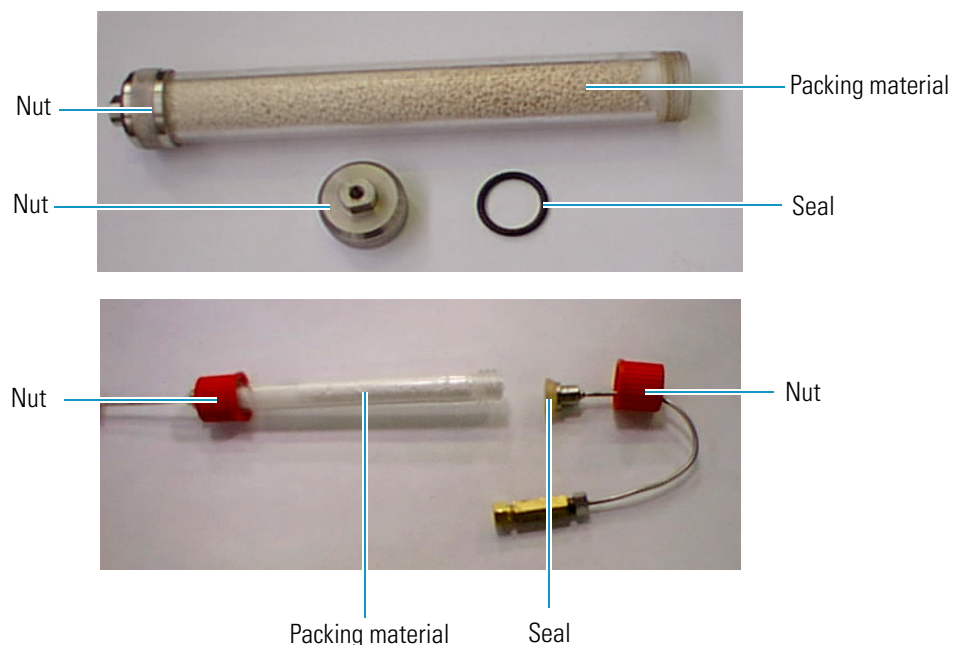


5. Pour sequentially the required filling materials into the adsorption filter, ensuring that each layer has the indicated size. At each step gently press the quartz wool using the rod provided.

**CAUTION** Soda Lime must be wetted before using. Pour 0.5 ml of water on the Soda Lime surface on the side that will be connected to the reduction reactor

6. Do the last layer using a sufficient quantity of quartz wool to form the required layer.
7. Complete the procedure by screwing on the second nut complete with its seal, as shown in [Figure 37](#).

**Figure 37.** Preparation of the adsorption filters



[Figure 38](#) shows the result of the filling operation of an adsorption filter.

## 5 Preparation of Reactors and Adsorption Filters

Preparing the Adsorption Filters

**Figure 38.** Result of the filling of a large adsorption filter and of a small one



## Connecting Reactors and Adsorption Filters

This chapter contains the instructions to install reactors and adsorption filters into the elemental analyzer, and it also provides information on how to remove them.

### Contents

- [Installing the Reactors into the Furnaces](#)
- [Installing the Adsorption Filters](#)
- [Removing the Reactors](#)
- [Removing the Adsorption Filters](#)

### Installing the Reactors into the Furnaces

The following [Table 38](#) summarizes the type of reactor to be used and the furnace where it must be installed according to your instrument configuration.

Refer to paragraph “[Introduction to the Preparation of Reactors and Filters](#)” on [page 57](#).

**Note** The reactors of special steel used for combustion require the use of a crucible.

**Table 38.** Reactors and furnaces

Determination	Left furnace	Right furnace
CHN	Quartz reactor	---
CHN-O	Quartz reactor	Quartz reactor
CHNS	Quartz reactor	---
CHNS-O	Quartz reactor	Quartz reactor
S (Sulphur)	Quartz reactor	---
O (Oxygen)	Quartz reactor	---
N (Nitrogen)	Quartz reactor	Quartz reactor
N Lubricant	Special steel reactor + SS crucible	Special steel reactor
NC	Quartz reactor	Quartz reactor
NCS	Special steel reactor	---

**Table 38.** Reactors and furnaces, continued

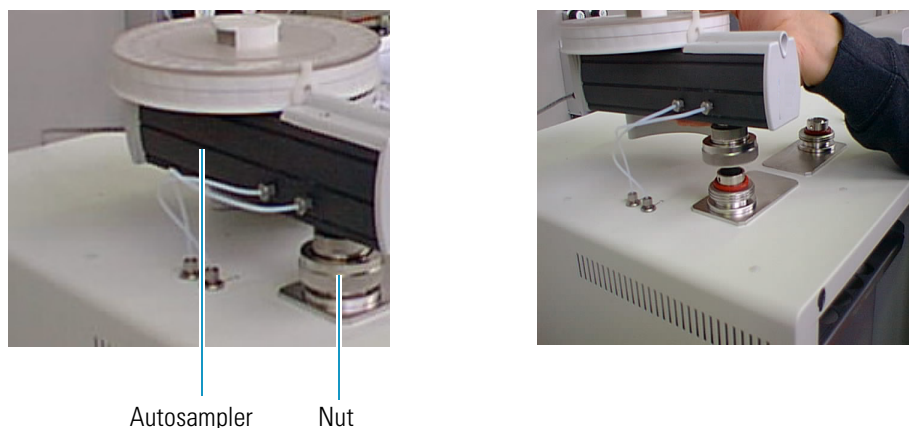
Determination	Left furnace	Right furnace
NC-Soils	Special steel reactor + SS crucible	Quartz reactor
NC-Sediments	Special steel reactor + SS crucible	Quartz reactor
NC-Filters	Special steel reactor + SS crucible	Quartz reactor
N/Protein	Special steel reactor + SS crucible	Special steel reactor
N-Brew	Special steel reactor + SS crucible	Special steel reactor
IRMS (NC)	Quartz reactor	---
HT (NC)		Quartz Reactor
HT (O/H)	Special reactor	---

## Preliminary Operations

Before installing the reactors do the following:

1. Check that the furnaces are at room temperature.
2. Open the furnaces compartment by lifting the cover and removing the protecting plate. Refer to paragraph “Furnaces Compartment” on page 28.
3. Remove the autosampler, if installed, by manually undoing the fixing nut, as shown in Figure 39.

**Figure 39.** Removing the autosampler



### ❖ To Install the Quartz Reactors into the Furnaces

The following procedure contains the instructions to install the quartz reactors.

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



**WARNING** The reactors must be installed with the furnaces at room temperature.

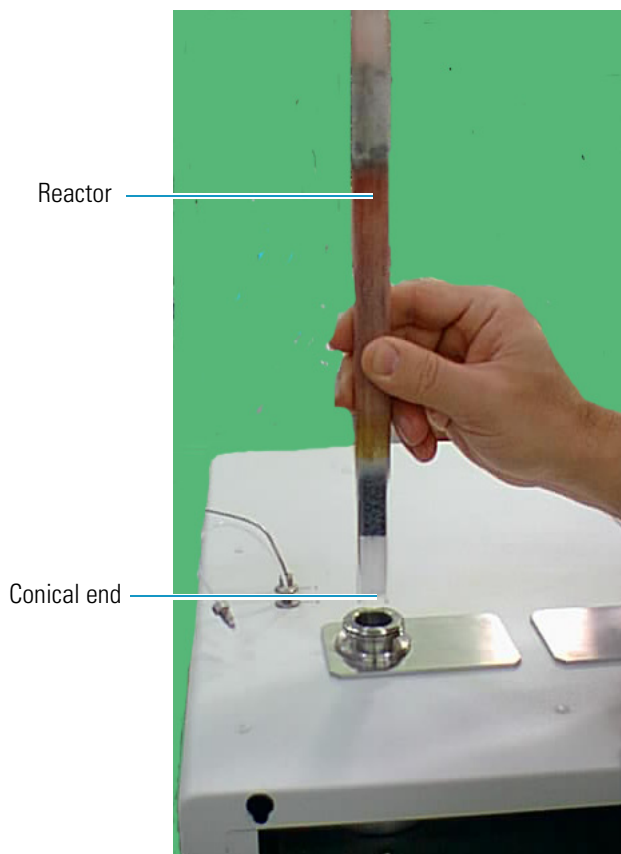
**Material required**

O-ring

**CAUTION** Do not use mechanical tools to screw or unscrew the fixing nut.

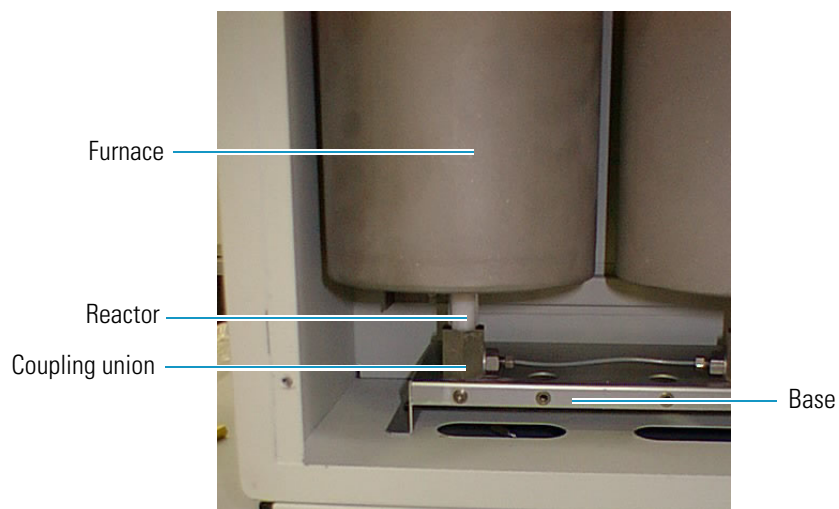
1. Remove the fixing nut.
2. Delicately introduce the reactor into the furnace ensuring that the tube conical end is turned downward, as shown in [Figure 40](#).

**Figure 40.** Introduction of the quartz reactor into the furnace



3. Guide the reactor inside the furnace. The reactor conical end must fit into the coupling union located on the base of the furnaces compartment, as shown in [Figure 41](#).

**Figure 41.** Driving the reactor into the furnace



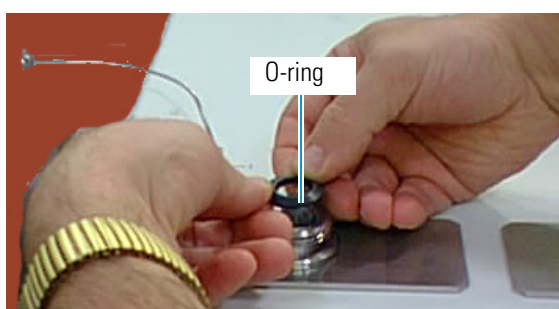
4. Gently press the edge of the reactor until introduction is complete, as shown in [Figure 42](#).

**Figure 42.** Complete the introduction of the reactor



5. Slip on the O-ring with its conical section turned upwards as shown in [Figure 43](#).

**Figure 43.** O-ring



6. Manually screw the autosampler fixing nut.

**Note** If required by your instrument configuration, install the reactor into the right furnace following the same instructions reported in this operating sequence.

The autosampler installed on the right channel is used only for CHNS-O and CHN-O configurations



7. To complete the operation manually screw the fixing nut or the autosampler nut if installed.
8. Put on again the protecting plate and the cover of the furnaces compartment.

❖ **To Install the Special Steel Reactors into the Furnaces**

The following procedure contains the instructions to install the special steel reactors into the left and right furnaces.

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



**WARNING** The reactors must be installed with the furnaces at room temperature.

**Material required**

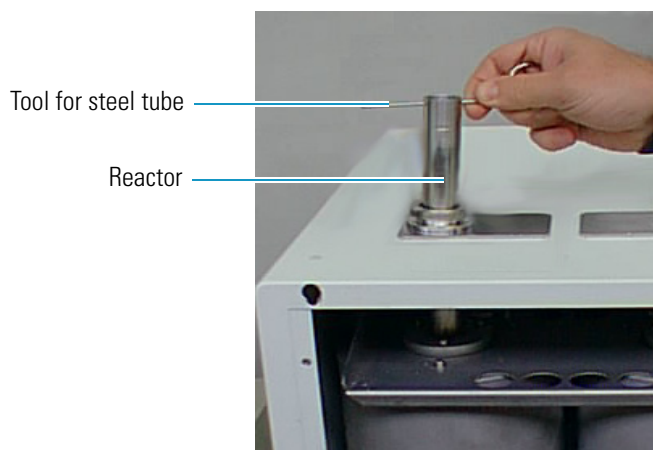
Tool for steel tubes

O-ring

1. Remove the fixing nut.

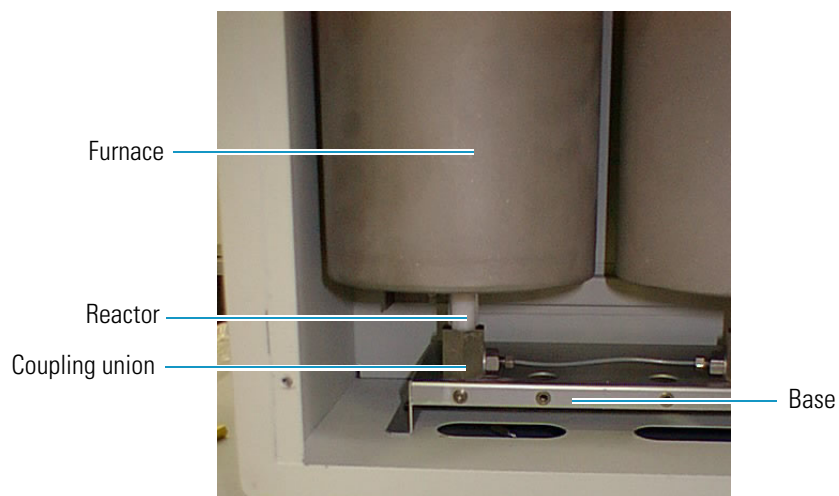
**CAUTION** Do not use mechanical tools to screw or unscrew the fixing nut.

**Figure 44.** Introducing the special steel reactor into the furnace



2. Introduce the tool, provided in the standard outfit, into the holes located on the top end of the reactor.
3. Guide the reactor into the furnace. The conical part should slide into the coupling union located on the base of the furnaces compartment, as shown in [Figure 45](#).

**Figure 45.** Driving the reactor into the furnace



4. Turn the reactor clockwise and push until completely in place, as shown in [Figure 46](#).

**Figure 46.** The reactor in place



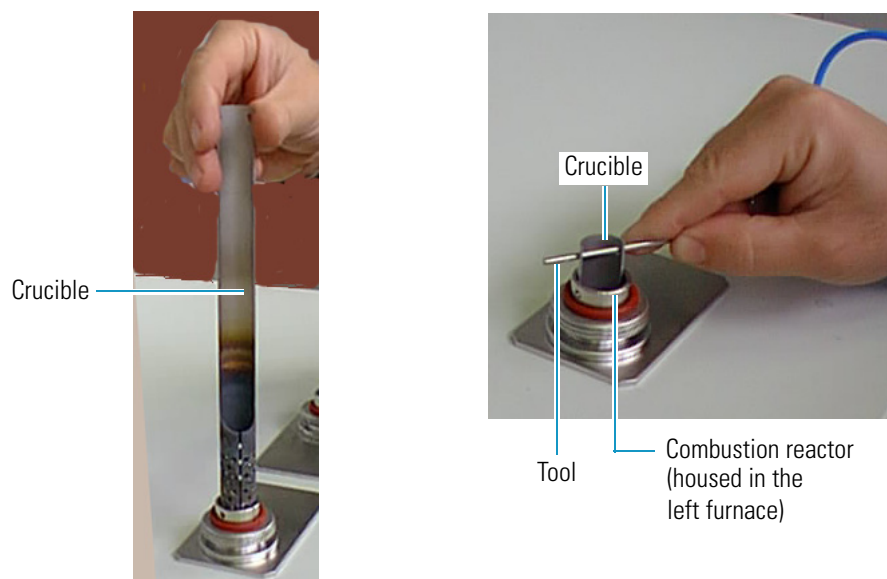
5. Slip on the O-ring as shown in [Figure 47](#).

**Figure 47.** O-ring



6. By using the tool for special steel reactors, introduce the crucible into the combustion reactor, which is in the left furnace. See [Figure 48](#).

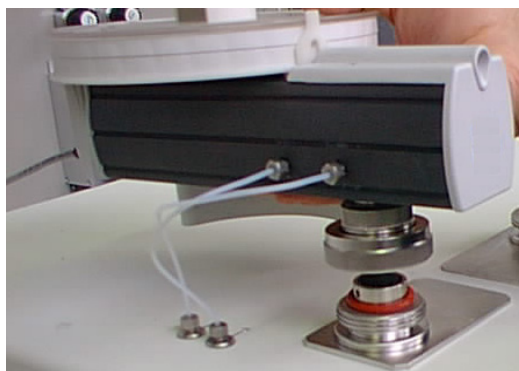
**Figure 48.** Introduction of the crucible into the combustion reactor



7. Manually screw the autosampler nut. See [Figure 49](#).

**CAUTION** Do not use mechanical tools to screw or unscrew the fixing nut.

**Figure 49.** Mounting the autosampler on the left furnace



8. Install the reduction reactor into the right furnace: in case of steel tubes following the instructions of previous operating sequence. In case of quartz tubes refer to the operating sequence “[To Install the Quartz Reactors into the Furnaces](#)” on [page 84](#).

9. At the end of the operation, manually screw the fixing nut or the autosampler nut if installed. See [Figure 50](#).

**Note** The autosampler installed on the right channel is used only for CHNS-O and CHN-O configurations.

**Figure 50.** Mounting the fixing nut on the right furnace



10. Put on again the protecting plate and the cover of the furnaces compartment.

## Installing the Adsorption Filters

The following [Table 39](#) summarizes the type of adsorption filter required and the channel to which it should be connected according to instrument configuration. Refer to paragraph [“Introduction to the Preparation of Reactors and Filters”](#) on [page 57](#).

**Table 39.** Adsorption filters

Determination	Left furnace	Right furnace
CHN	---	---
CHN-O	---	Glass filter
CHNS	---	---
CHNS-O	---	Glass filter
S (Sulphur)	Glass filter	---
O (Oxygen)	Glass filter	---
N (Nitrogen)	---	Two Plexiglas filters in series
N Lubricant	---	Two Plexiglas filters in series
NC	---	Glass filter
NCS	Glass filter	---
NC-Soils	---	Glass filter
NC-Sediments	---	Glass filter
NC-Filters	---	Glass filter
N/Protein	---	Two Plexiglas filters in series
N-Brew	---	Two Plexiglas filters in series
IRMS (NC)	Plexiglas filter	---
HT (NC)	---	Plexiglas filter
HT (O/H)	Plexiglas filter	---

## Preliminary Operations

The following preliminary operations are required to install the adsorption filters.

1. Have access to the detector compartment by opening the right side door of the instrument. Refer to paragraph “[Detector Compartment](#)” on [page 30](#). [Figure 51](#) shows the detector compartment.

**Figure 51.** Detector compartment



### ❖ To Connect and Install the Adsorption Filters

The following operating procedure contains the instructions to install the adsorption filters. [Figure 52](#) shows the result of the installation of two adsorption filters connected in series.

**Figure 52.** Adsorption filters installed in the detector compartment



Single filter



Two filters in series

According to your instrument configuration, do the installation and connection of the adsorption filters following the instructions of next paragraphs [Single Filter](#) or [Two Filters in series](#).

## Single Filter

To connect the filter do the following:

1. Connect the filter inlet to the connection coming from the reactor.
2. Connect the filter outlet to the connection coming from the gas chromatographic column.
3. Secure the filter by means of the appropriate clips.

## Two Filters in series

To connect two filters in series do the following:

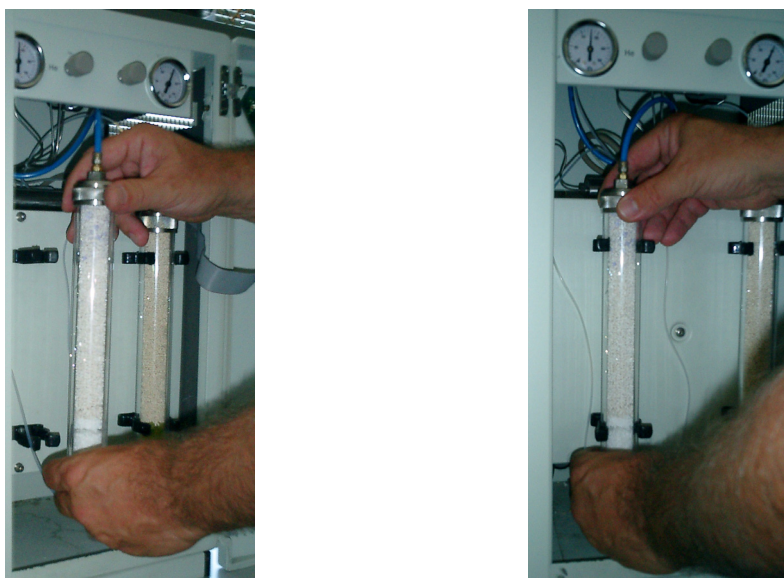
1. Connect the filters to one another, as shown in [Figure 53](#), and then to the circuit as per relevant diagram. Refer to “[Introduction to the Preparation of Reactors and Filters](#)” on [page 57](#).

**Figure 53.** Connection of two filters in series



2. Introduce the filter into the securing clips, as shown in [Figure](#) on [page 93](#).

**Figure 54.** Installation of the filters into the detector compartment



## Removing the Reactors

Before starting this operation:

- Check that the furnaces are at room temperature.
- Open the furnaces compartment by lifting the cover and removing the protecting plate. Refer to paragraph “Furnaces Compartment” on page 28.
- Undo the nuts securing the reactors. If the autosampler is installed, manually unscrew the fixing nut to remove it as shown in Figure 39 page 84.

### ❖ To Remove the Quartz Reactors from the Furnaces

The following operating procedure contains the instructions to remove the quartz reactors from the left and right furnaces.

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



**WARNING** The reactors must be installed with the furnaces at room temperature.

1. Remove the O-ring from the top of the reactor as shown in the left image of Figure 55.
2. Using both hands turn the tube counterclockwise and simultaneously pull it upward as shown in the right image of Figure 55.

**Figure 55.** O-ring and removal of the reactor from the furnace



❖ **To Remove Special Steel Reactors from the Furnaces**

The following operating procedure contains the instructions to remove the special steel reactors from the left and right furnaces.



**WARNING** The reactors must be installed with the furnaces at room temperature.

**Material required**

Tool for special steel reactor

Tool for crucible

1. Remove the O-ring from the top of the reactor as shown in [Figure 56](#).

**Figure 56.** Removal of the steel reactor o-ring



2. Remove the crucible from the combustion reactor (left furnace) using the appropriate tool as shown in [Figure 57](#).

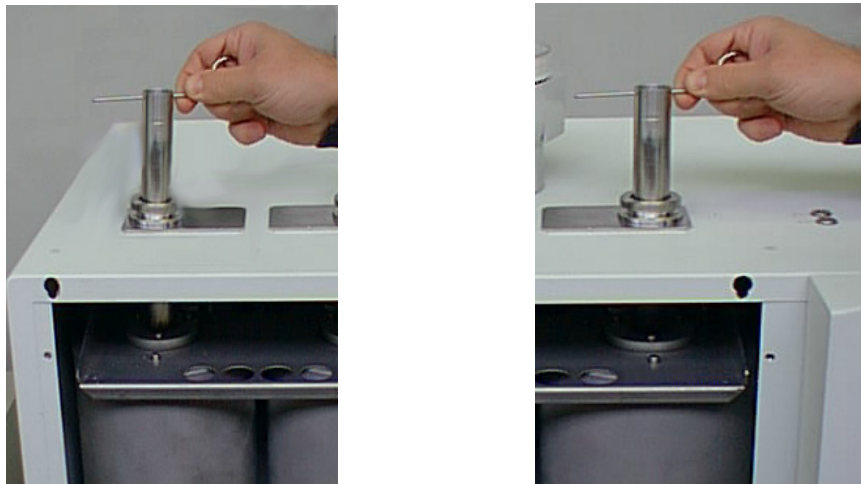
**Figure 57.** Removal of the crucible





3. By using the proper tool, remove the reactor turning it counterclockwise and simultaneously pulling it upwards, as shown in [Figure 58](#).

**Figure 58.** Removal of the reactor



## Removing the Adsorption Filters

Before starting this operation:

1. Open the right side door of the instrument to have access to the detector compartment. Refer to paragraph “[Detector Compartment](#)” on [page 30](#).

### ❖ To Remove the Adsorption Filter

The following operating procedure contains the instructions to remove the adsorption filter from the system.

1. Remove the filter from the securing clips.
2. Disconnect the filter inlet and then its outlet from the relevant connections.

# Preparing the Sample

This chapter describes some techniques for the sample preparation, also it provides basic instructions to homogenize and weigh the sample.

## Contents

- [Introduction](#)
- [Homogenizing the Sample](#)
- [Sample Weighing Technique](#)

## Introduction



**WARNING** Be very careful in preparing samples, because the substances to be analyzed may be dangerous. Read the Safety Data Sheets referring to the different chemicals and handle them in the appropriate environment (e.g. under a fumes hood), strictly obeying the company safety regulations.

## Homogenizing the Sample

Before the analysis the sample must be properly homogenized. This paragraph gives you basic information on how to prepare the most currently analyzed materials.

[Table 40](#) on [page 98](#) gives you indications to weigh the sample according to your instrument configuration.

**Table 40.** Information on sample weighing

	Sample	Information
<b>Sulfur</b>	Soils	In soils, sulfur is often present as ion sulfate, and therefore it is necessary to add 5-10 mg of vanadium pentoxide ( $V_2O_5$ ) every 10-20 mg of soil to ensure complete conversion of inorganic sulfur into sulfur dioxide.
	Minerals	The sulfur content in minerals can vary from a few percent units (e.g. bauxite) to definitely higher values (e.g. pyrite). If the mineral to be analyzed is unknown, a pre-analysis is recommended. Once the sulfur content is defined, the required analyses with proper sample amounts can be performed:  <i>Example:</i> For minerals rich in sulfur, samples of 2-5 mg are prepared. For minerals with sulfur traces, samples of 10-20 mg are prepared.
	Plants	Plants are rich in Nitrogen, Carbon and Hydrogen, but relatively poor in Sulfur. Therefore, after the first analysis, check that the peak of sulfur dioxide is correctly integrated.
<b>Nitrogen</b>	Soils	The Nitrogen content in such samples is generally very low (0.1%).
	Sediments	Set a very high sensitivity of integration and use Oxygen of maximum purity grade.

## Soils, Sediments and Minerals

Before the analysis, samples of such nature and origin require homogenizing, which can be performed by means of proper mills allowing the simultaneous preparation of several samples. A first coarse homogenizing on sample amounts of a few hundreds of grams is followed by finer homogenizing on a few dozens of grams, until optimum granularity (100-200  $\mu\text{m}$ ) is reached. The resulting sample is dried in an oven.

## Carbons

The technique to homogenize carbons is the same as that used for the preparation of soils, sediments and minerals, but the sample drying requires specific operations: The samples are dried in an oven for one hour at 105°C, left in the air for the same time to let them acquire again their natural moisture and then stored in airtight containers. Finally they are put into driers.

## Metals

The sample preparation technique is a function of the metal hardness. Special machines can be used as drills, mills or lathes. In case of particularly hard materials, use a diamond file.

You should obtain metal chips as small and light as possible. The homogenizing degree depends on the particle size.

The quantity of sample for the analysis is a function of the alloy composition.

- For cast irons, prepare samples of 10-20 mg.
- For steels and other metal alloys, weigh 40-50 mg.

## Plastics

Polymers are generally available as pellets, or only rarely as powders.

If you don't want or cannot homogenize the sample, you can cut the pellets into small pieces and analyze 2-3 mg.

The same process is used for synthetic and natural rubbers.

## Vegetal

To prepare samples of vegetal products, two types of mills are normally used:

- **Blade mills** to homogenize cereals, leaves, forage and wood. In these mills, devices with 1 mm mesh sieves are used for N/Protein determination.
- **Ball mills** to homogenize samples of fruit and vegetables after lyophilization. These mills use devices for finer granulometry.

The sample amount to be analyzed depends on the type of determination and on the homogenizing degree.

## Liquids

Liquid samples are prepared according to a procedure that depends on the sample volatility.

Liquid samples with limited volatility are weighed in traditional tin containers.

However, to avoid sample losses, we suggest to use two containers for each sample.

If the sample is characterized by high viscosity, it should be properly mixed before being drawn for injection.

Samples injectable by micro syringes can be introduced manually using the optional manual injection device, or automatically using the autosampler for liquids.

## Sample Weighing Technique

To define the weighing range, you should know the nature (organic, inorganic, metal-organic) and origin (pure chemical, natural product) of the substance to be analyzed.

For weighing samples, the following materials are required:

- Balance
- Tools to seal the container containing the sample.

Samples of different nature require specific weighing techniques.

**Note** The technique of two containers is suggested to prevent sample losses due to defective sealing of the container and consequently prevent the autosampler contamination.

The weighing procedure requires a series of operations according to the sample nature.

## Solid Samples

Solid samples are introduced directly into the tin container using a spatula.

According to the sample quantity to be analyzed, refer to the following operating procedures:

- “Weighing Technique for Large Quantities of Solid Samples” on page 101.
- “Weighing Technique for Small Quantities of Solid Samples” on page 104.

## Liquid Samples

The weighing procedure changes according to the sample type.

### Samples Characterized by Limited Volatility

- *If the sample density is 1 or close to 1*  
Introduce the sample directly into the tin container for liquids using a syringe of 10 or 100 µl capacity according to the instrument configuration.
- *If the sample density varies significantly, even in samples of the same nature (milk is a typical example)*  
Only for N determination, let the sample be adsorbed on a Chromosorb WAW<sup>®</sup> layer previously introduced into the container.

Refer to the following operating procedures:

- “Weighing Technique for Liquid Samples” on page 105.
- “Weighing Technique for Liquid Samples Deposited on Adsorbent Material” on page 107.

### Samples Characterized by High Viscosity

They must be properly mixed before being drawn.

To introduce the sample, take some using a spatula and let it slide along the container walls as shown in [Figure 59](#) on page 101.

**Note** Only for N Determination, according to the sample viscosity, it may be necessary to adsorb the sample on a Chromosorb WAW<sup>®</sup> (only for N, N-Brew and N/Protein Configurations) layer previously introduced into the tin container.

Refer to the following operating procedure:

- “Weighing Technique for Viscous Samples” on page 108.

## Samples Available in Liquid Phase

They can be injected directly into the reactor as follows:

- *Manually*, through the manual injection device using a micro syringe.
- *Automatically*, using the autosampler for liquids.

### ❖ Weighing Technique for Large Quantities of Solid Samples

The following procedure contains the instructions to weigh large quantities of solid samples.

#### Materials required

Balance

Tin disks

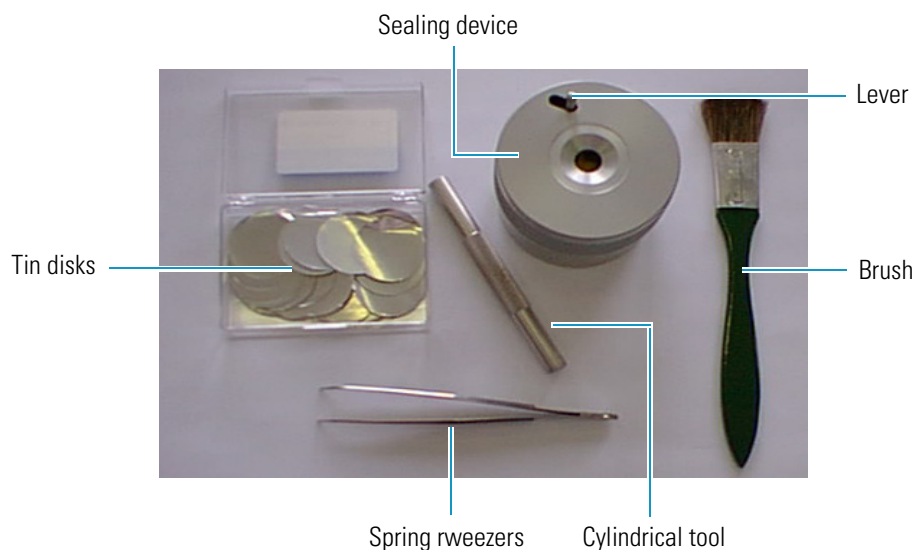
Spring tweezers

Sealing device and cylindrical tool

Spatula

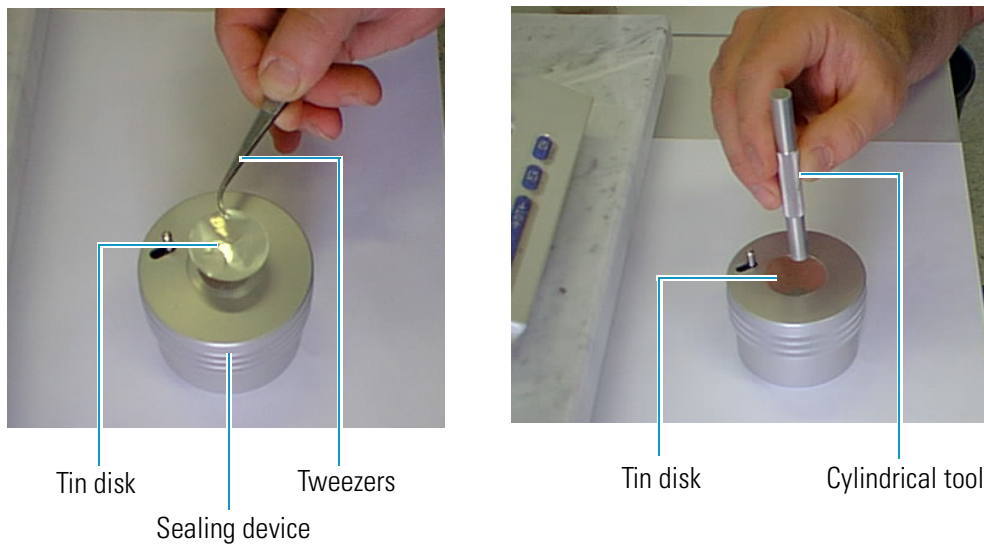
Brush

**Figure 59.** Accessories required to weigh solid samples



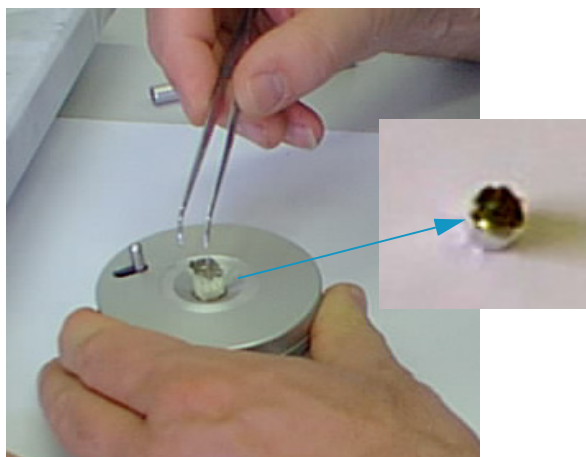
1. By using the tweezers, take a tin disk and rest it on the cavity of the sealing device, as shown in [Figure 60](#).
2. By using the cylindrical tool, press the tin disk and make it enter the cavity of the sealing device, as shown in [Figure 60](#).

**Figure 60.** Preparation of the tin container (1)



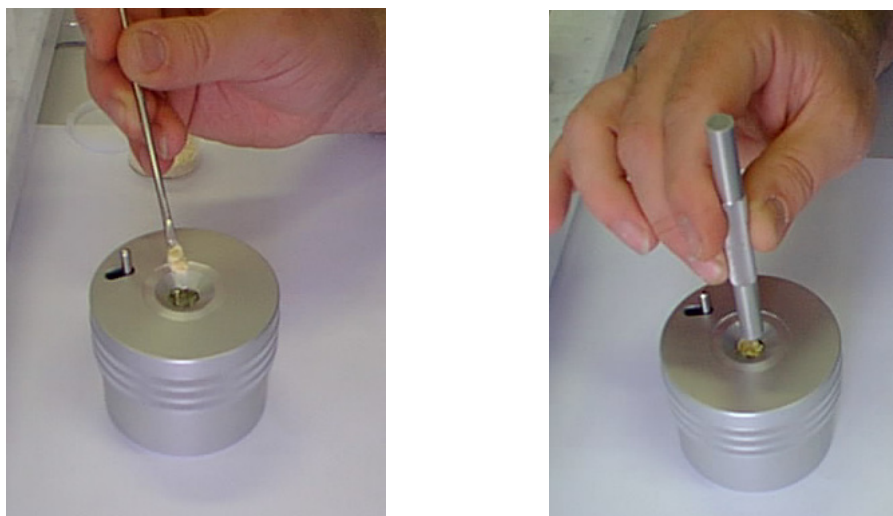
3. Press the top of the sealing device downwards to have the container come out of the cavity.
4. Take out the container using the spring tweezers, as shown in [Figure 61](#).
5. Put the prepared container on a clean surface.

**Figure 61.** Preparation of the tin container (2)



6. Using a spatula, introduce some sample into the tin container until sufficiently filled, then delicately press the sample using the cylindrical tool, as shown in [Figure 62](#).

**Figure 62.** Introduction and compression of the sample



7. Close the container using the lever located on the top surface of the sealing device.
8. Press the top of the sealing device downwards to have the container come out of the cavity.
9. Remove the container using the spring tweezers, as shown in [Figure 63](#).

**Figure 63.** Removal of the closed container and cleaning of the contact surface



10. Weigh the container obtained and take note of the value.

We suggest the following weighing procedure to prevent sample losses due to defective sealing.

- a. Using the tweezers, take a tin disk and rest it on the cavity of the sealing device, as shown in [Figure 60](#).
- b. Using the cylindrical tool, press the tin disk and make it enter the cavity of the sealing device, as shown in [Figure 60](#).
- c. Press the top of the sealing device downwards to have the container come out of the cavity.



- d. Take out the container using the spring tweezers, as shown in [Figure 61](#).
- e. Put the prepared container on a clean surface.
- f. Prepare a second container placing another tin disk on the cavity of the sealing device.
- g. Using the cylindrical tool, gently press the disk to obtain a half-open container.
- h. Place both containers on the balance pan and do the tare.
- i. Take the first container and put it into the cavity of the sealing device.
- j. Using a spatula, introduce some sample into the tin container until sufficiently filled, then delicately press the sample using the cylindrical tool, as shown in [Figure 62](#).
- k. Close the container using the lever located on the top surface of the sealing device.
- l. Press the top of the sealing device downwards to have the container come out of the cavity.
- m. Remove the container using the spring tweezers, as shown in [Figure 63](#), and rest it on a clean surface.
- n. Clean the contact surfaces using the brush.
- o. Put the half-open container on the cavity of the sealing device and place thereon the container containing the sample,
- p. Using the cylindrical tool, introduce the containers into the sealing device and then repeat steps **k**, **l**, **m** and **n** of this procedure.
- q. Weigh the container obtained and take note of the value.

#### ❖ **Weighing Technique for Small Quantities of Solid Samples**

We suggest the following weighing procedure to prevent sample losses due to defective sealing.

The following procedure contains the instructions to weigh small quantities of solid samples.

##### **Materials required**

Electronic micro balance

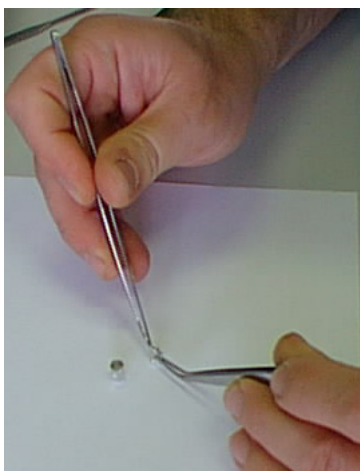
Tin containers for small weighings

Two spring tweezers

Spatula

1. Take two containers for small weighings, put them onto the balance pan and do the tare.
2. Remove one of the containers from the balance pan and put it onto a clean surface. Using a spatula, introduce into the container the sample quantity required for the analysis.
3. Weigh the container with sample and read the value. If the weight is correct for the analysis to be run, remove the two containers from the balance pan and rest them on a clean surface.
4. Close the container containing the sample using two spring tweezers, as shown in [Figure 64](#), to obtain a pellet.

**Figure 64.** Preparation of the container for small sample quantities



- 
5. Introduce the pellet into the second container and close the latter in the same way.
  6. Put the container obtained onto the balance pan, weigh it and take note of the value.

#### ❖ **Weighing Technique for Liquid Samples**

The following operating procedure contains the instructions to properly weigh liquid samples.

##### **Materials required**

Electronic micro balance

Tin container for liquid samples

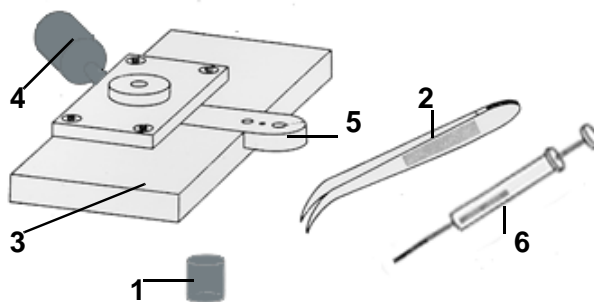
Spring tweezers

Sealing device (optional)

Spatula

10 or 100  $\mu$ l syringe according to the instrument configuration

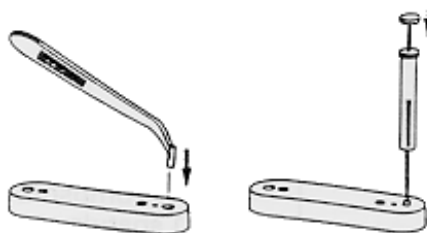
**Figure 65.** Accessories to weigh liquid samples



- |                         |                    |
|-------------------------|--------------------|
| 1. Container for liquid | 2. Spring tweezers |
| 3. Sealing device       | 4. Lever           |
| 5. Sample-holding slide | 6. Syringe         |

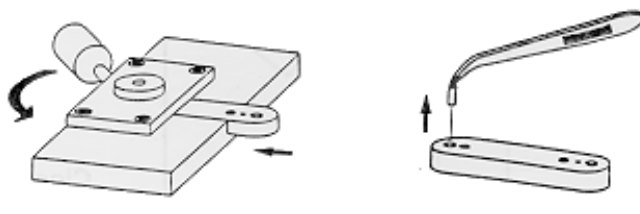
1. Take a tin container for liquid samples and put it onto the micro balance pan. Do the tare.
2. Place the tin container into the appropriate position in the slide of the sealing device, then inject the sample using a syringe, as shown in [Figure 66](#).

**Figure 66.** Housing for the container and sample injection



3. Put the slide with the container into the sealing device and tighten the container using the appropriate lever, as shown in [Figure 67](#).

**Figure 67.** Closing and removing the container



4. Remove the slide from the sealing device and then the container with the sample from the slide, as shown in [Figure 67](#).
5. Put the container onto the micro balance pan, weigh it and take note of the weight value.

❖ **Weighing Technique for Liquid Samples Deposited on Adsorbent Material**

The following operating procedure contains the instructions to properly weigh liquid samples previously deposited on adsorbent material.

**Materials required**

Balance

Tin containers for liquid samples

Two spring tweezers

Spatula

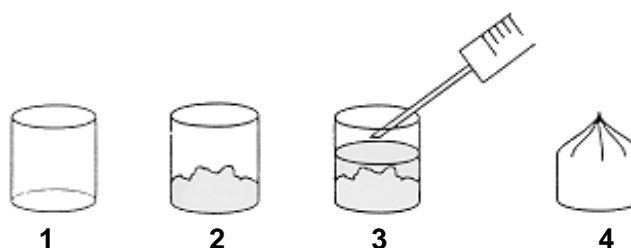
100  $\mu$ l syringeChromosorb WAW<sup>®</sup> (Only for N, N/Protein and N-Brew Determinations)

**Note** Chromosorb WAW<sup>®</sup> is an extremely porous inert inorganic material, made of silicates and completely free from Nitrogen. When Chromosorb is free from contamination, its contribution to the blank value is negligible.

1. Introduce one or two spatula tips of Chromosorb WAW<sup>®</sup> into the tin container.
2. Put the container with the adsorbent material and an empty container onto the balance pan and do the tare.
3. Remove the containers from the balance pan and rest them onto a clean surface.
4. Using the syringe, introduce the liquid sample depositing it on the layer of adsorbent material contained in the container, as shown in [Figure 68](#).

**CAUTION** Do not wet the container walls during sample introduction.

**Figure 68.** Weighing of a liquid sample deposited on adsorbent material



1. Empty container

2. Layer of adsorbent material

3. Introduction of the sample

4. Closed container

5. Close the container using two spring tweezers or the sealing device.
6. Introduce the container with the sample into the second container and close the latter in the same way.
7. Weigh the container and take note of the weight value.

❖ **Weighing Technique for Viscous Samples**

The following operating procedure contains the instructions to properly weigh viscous samples.

According to its viscosity, a sample can be weighed as described in either of the following operating sequences:

- “Weighing Technique for Liquid Samples” on page 105.
- “Weighing Technique for Liquid Samples Deposited on Adsorbent Material” on page 107.

**CAUTION** When a liquid sample is too viscous to be drawn by means of a syringe, use the spatula provided in the instrument standard outfit, as shown in [Figure 69](#).

**Figure 69.** Introduction of a viscous sample into the container



# Analytical Methods

This chapter describes the analytical methods used for all configurations of the Flash 2000 elemental analyzer.

## Contents

- Introduction
- Analytical Method for CHN Configuration
- Analytical Method for CHN-O Configuration
- Analytical Method for CHNS Configuration
- Analytical Method for CHNS-O Configuration
- Analytical Method for S (Sulfur) Configuration
- Analytical Method for O (Oxygen) Configuration
- Analytical Method for N (Nitrogen) Configuration
- Analytical Method for NC Configuration
- Analytical Method for NCS Configuration
- Analytical Method for NC-Soils, NC-Sediments and NC-Filters Configurations
- Analytical Method for N Lubricant, N/Protein and N-Brew Configurations
- Analytical Method for NC-IRMS Configuration
- Analytical Method for NC-HT Configuration

## Introduction

Each configuration of the Flash 2000 elemental analyzer has its own dedicated analytical method.

The description of the analytical method is illustrated by diagrams referring to the concerned instrument configuration.

When necessary, also refer to Chapters 1, 4 and 5 for more information concerning the components of the instrument and their functions. The analytical method used in each instrument configuration includes different subsequent steps leading to determine the percent composition of the components of interest through the transformation of the solid or liquid sample into gas.



**Note** To develop and perform the analytical cycle, refer to the relevant chapter contained in **Section III**.

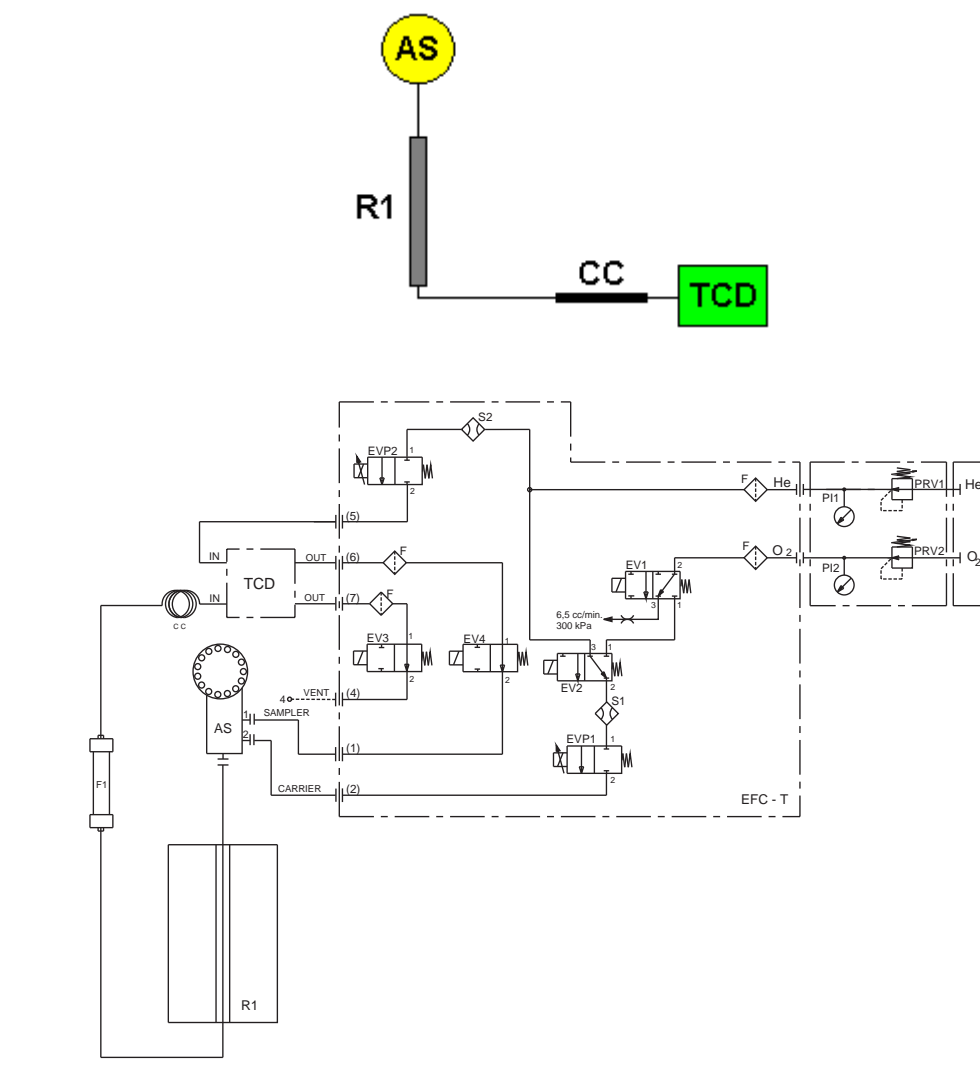
For correct sample analyses all pneumatic lines must be leak-free. Therefore, a preliminary leak check is recommended before starting analytical cycles.

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

## Analytical Method for CHN Configuration

An autosampler **AS** is connected to a quartz reactor **R1** housed in an furnace at a temperature of 900 °C. This reactor is connected to the analytical column **CC**, which on its turn is connected to a channel of the thermal conductivity detector **TCD**.

**Figure 70.** Instrument parts diagram and pneumatic diagram for CHN determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow through the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.



The proportional valve **EVP2**, connected to the detector reference channel **RF**, controls the Helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed.

The Oxygen line **O2** is connected to the solenoid valve **EV1**.

This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off the Oxygen flow, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor. Tin, coming in contact with the extremely oxidizing environment, triggers a strong exothermic reaction.

Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, valves **EV1** and **EV2** return to their original position restoring Helium flow.

The combustion products are conveyed across the reactor **R1**, where oxidation is completed.

Nitrogen oxides possibly formed are reduced to elemental nitrogen and Oxygen excess is retained.

**Note** Sulfur and halogenated compounds (Chlorine, Bromine, etc.), possibly present in the sample, do not affect the analysis, since the silvered cobaltous/cobaltic oxide catalyst holds back both SO<sub>2</sub> and halogens.

Then the gas mixture (N<sub>2</sub>, CO<sub>2</sub> and H<sub>2</sub>O) flows into the chromatographic column **CC1**, where separation takes place.

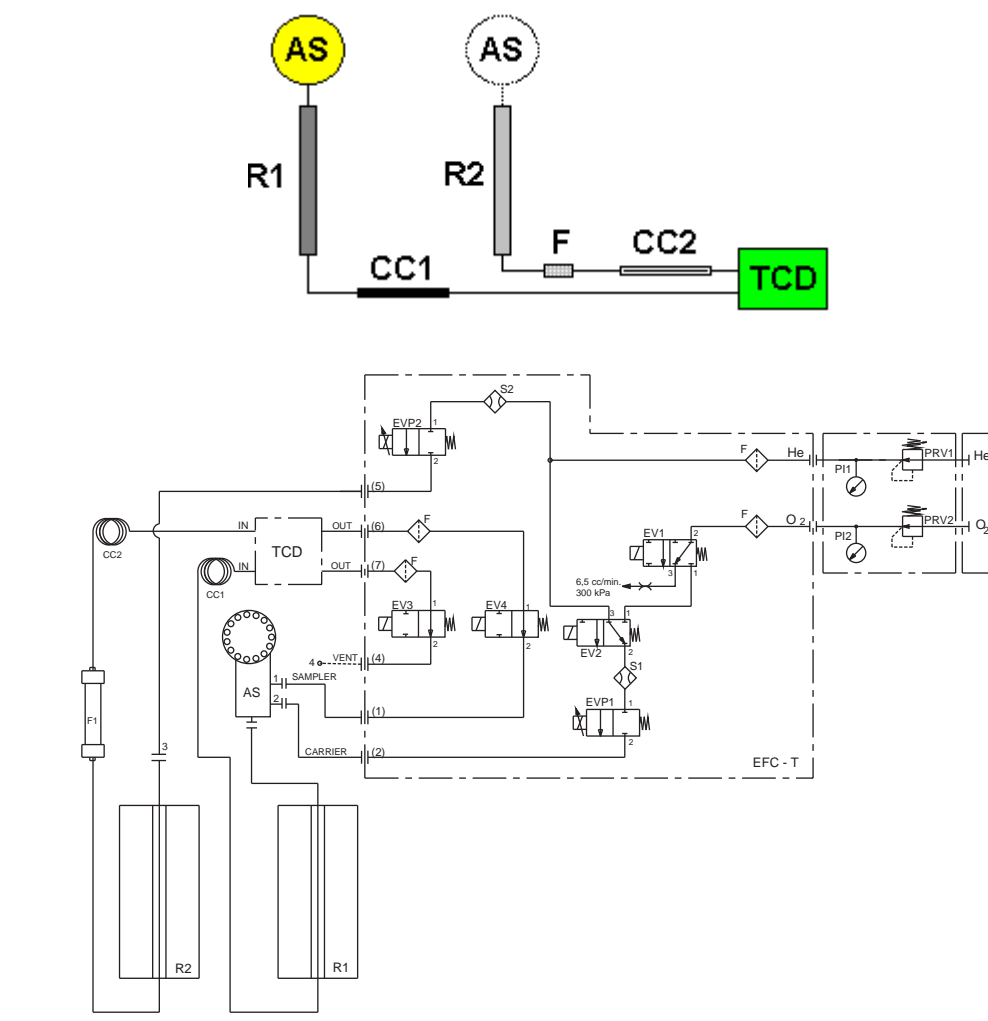
The eluted gases are conveyed to the thermal conductivity detector **TCD** that generates electrical signals, which properly processed by the Eager Xperience software provide Nitrogen, Carbon and Hydrogen percentages.

## Analytical Method for CHN-O Configuration

**CHN Determination:** An autosampler **AS** is connected to a quartz reactor **R1** placed in a furnace at a temperature of 900 °C. This reactor is connected to the analytical column **CC1**, on its turn connected to a channel of the thermal conductivity detector **TCD**.

**Oxygen Determination:** A second autosampler **AS** is connected to a reactor **R2** placed in a furnace at a temperature of 1060 °C. An adsorption filter **F** is connected to the reactor outlet. The **F** outlet is connected to the analytical column **CC2**, on its turn connected to the other channel of the thermal conductivity detector **TCD**.

**Figure 71.** Instrument parts diagram and pneumatic diagram for CHN-O determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow through the whole pneumatic circuit as far as the solenoid valve **EV3**.

This valve, normally open, exhausts Helium to the atmosphere through Vent 4. The proportional valve **EVP2** controls Helium flowing to the circuit comprising **R2**, **F** and **CC2** as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the area where the sample is housed.

**Note** When two autosamplers are installed on the elemental analyzer, the point 1 (purge) must be connected to the autosampler that you intend to use for the analysis.

When a single autosampler is installed, to pass from the CHN configuration to the O Configuration, or vice-versa, change the position of the autosampler from R1 to R2 or voice-overs. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

### CHN Determination

During pre-analysis, the solenoid valve **EV1** shuts off the Oxygen flow, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion. At the end of the time set for Oxygen introduction, valves **EV1** and **EV2** return to their original position restoring Helium flow.

The combustion products are conveyed across the reactor **R1**, where oxidation is completed. Nitrogen oxides possibly formed are reduced to elemental nitrogen, and Oxygen excess is retained.

**Note** Sulfur and halogenated compounds (Chlorine, Bromine, etc.), possibly present in the sample, do not affect the analysis, since the silvered cobaltous/cobaltic oxide catalyst holds back both SO<sub>2</sub> and halogens.

Then the gas mixture (N<sub>2</sub>, CO<sub>2</sub> and H<sub>2</sub>O) flows into the chromatographic column **CC1**, where separation takes place. The eluted gases are conveyed to the thermal conductivity detector **TCD** that generates electrical signals, which properly processed by the Eager 300 software provide Nitrogen, Carbon and Hydrogen percentages.

### Oxygen Determination

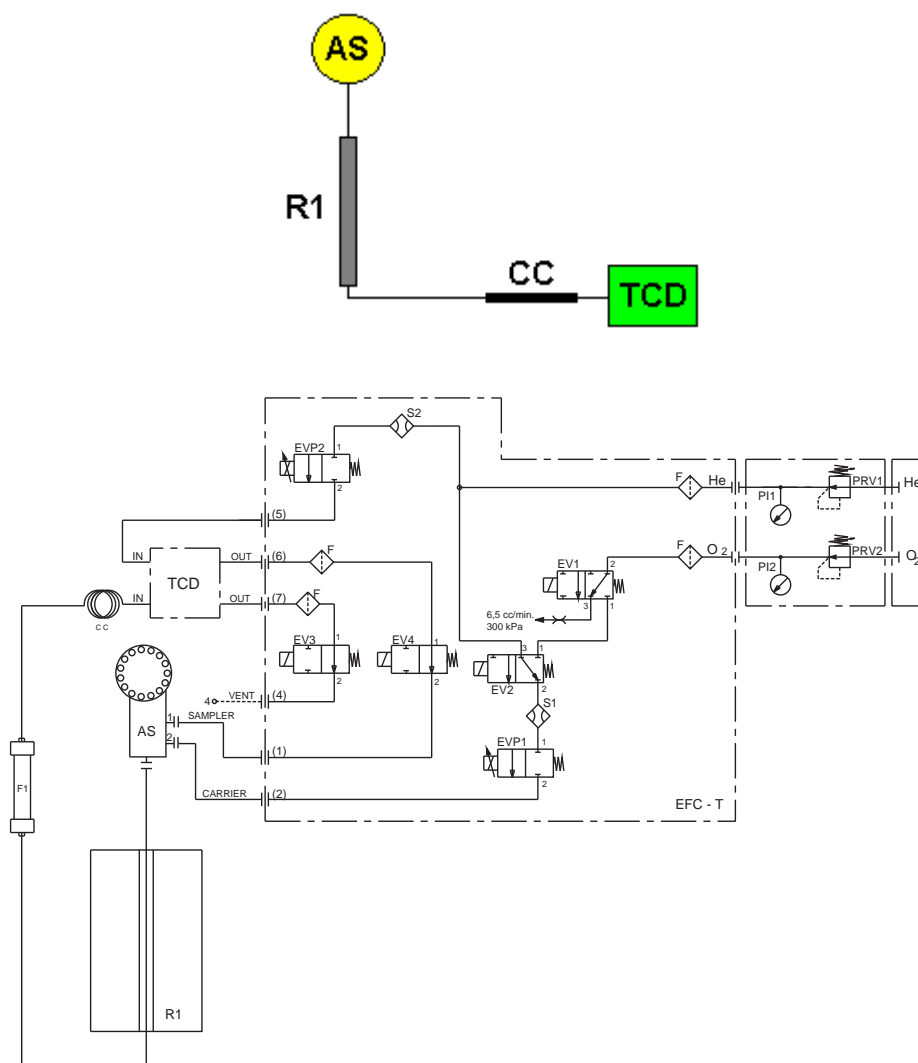
No switching of valves. When *Start Analysis* is pressed, the sample, weighed in a silver container and stored in the autosampler, is dropped into the reactor **R2** where it undergoes instant pyrolysis. During pyrolysis, N<sub>2</sub>, CO and H<sub>2</sub> form. The pyrolysis products cross the adsorption filter **F** where halogenated compounds (Chlorine, Bromine, etc.) are retained. The gas mixture flows into the chromatographic column **CC2**, where carbon monoxide is separated from other gases.

Then the eluted gases are conveyed to the thermal conductivity detector **TCD** that generates an electrical signal, which properly processed by the Eager Xperience software provides Oxygen percentage.

## Analytical Method for CHNS Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at a temperature of 900 °C. This reactor is connected to the analytical column **CC**, on its turn connected to a channel of the thermal conductivity detector **TCD**.

**Figure 72.** Instrument parts diagram and pneumatic diagram for CHNS determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the helium flow through the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent 4.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the

Helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis, the solenoid valve **EV1** shuts off the Oxygen flow, whereas the solenoid valve **EV2** allows Helium to flow in the circuit.

When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds the sample, weighed in a tin container and placed in the autosampler, is dropped into the combustion reactor. Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction.

Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow. The combustion products are conveyed across the reactor **R1** where oxidation is completed. Nitrogen oxides and sulfur trioxide, possibly formed, are reduced to elemental nitrogen and sulfur dioxide, and the Oxygen excess is retained.

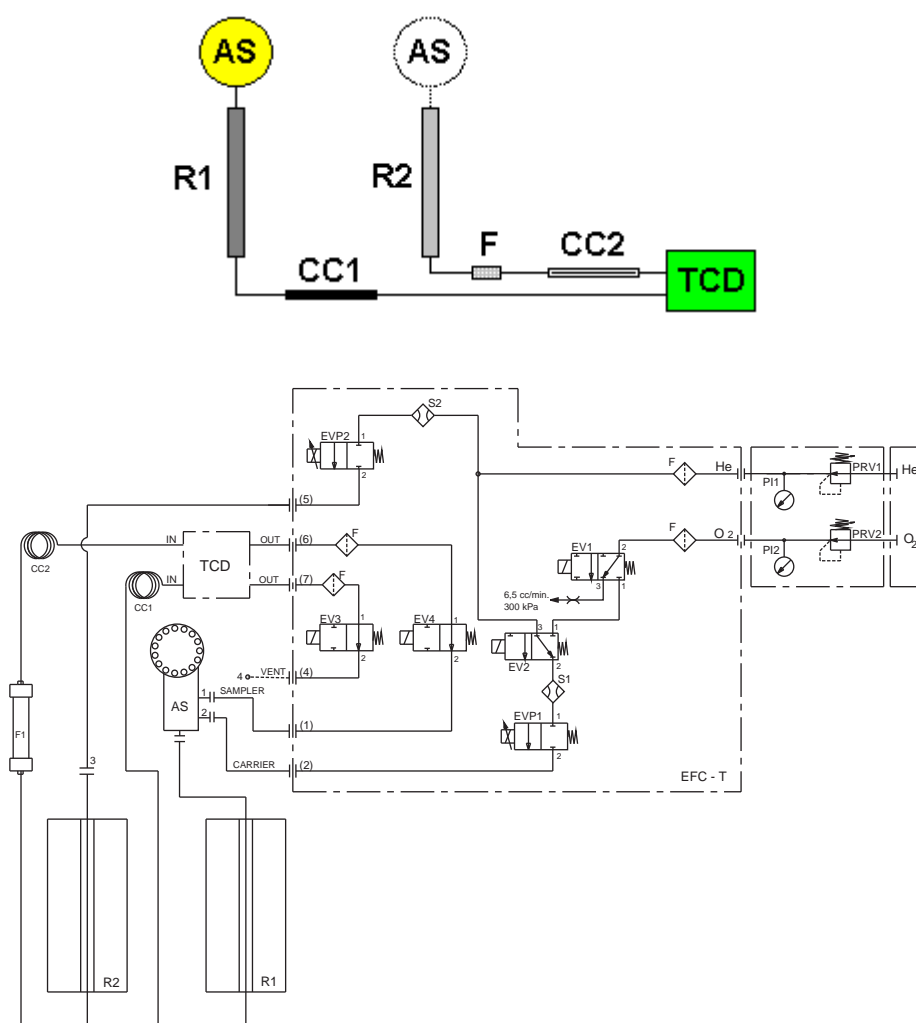
Then the gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O e SO<sub>2</sub>) flows into the chromatographic column **CC1** where separation takes place. The eluted gases are sent to the thermal conductivity detector **TCD** that generates electrical signals, which, properly processed by the Eager Xperience software, provide the percentages of Nitrogen, Carbon, Hydrogen and Sulfur contained in the sample.

## Analytical Method for CHNS-O Configuration

**CHNS Determination:** An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of 900 °C. This reactor is connected to the analytical column **CC1**, on its turn connected to a channel of the thermal conductivity detector **TCD**.

**Oxygen Determination:** A second autosampler **AS** is connected to a reactor **R2** placed in an furnace at the temperature of 1060 °C. To the reactor outlet an adsorption filter **F** is connected. The **F** outlet is connected to the analytical column **CC2**, on its turn connected to the other channel of the thermal conductivity detector **TCD**.

**Figure 73.** Instrument parts diagram and pneumatic diagram for CHNS-O determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow

through the pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.

The proportional valve **EVP2** controls the Helium flow in the circuit comprising **R2**, **F** and **CC2** as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the area where the sample is housed.

**Note** When two autosamplers are installed on the elemental analyzer, the point 1 (purge) must be connected to the autosampler that you intend to use for the analysis.

When a single autosampler is installed, to pass from the CHNS configuration to the O Configuration, or vice-versa, change the position of the autosampler from R1 to R2 or vice-versa. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

### CHNS Determination

During pre-analysis the solenoid valve **EV1** shuts off the Oxygen flow, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature reaches approximately 1800 °C instantly causing the sample combustion. At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow.

The combustion products are conveyed across the reactor **R1** where oxidation is completed. Nitrogen oxides and sulfur trioxide possibly formed are reduced to elemental nitrogen and sulfur dioxide, and Oxygen excess is retained.

Then the gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O e SO<sub>2</sub>) flows into the gas chromatographic column **CC1** where separation occurs.

The eluted gases are conveyed to the thermal conductivity detector **TCD** that generates electrical signals, which, properly processed by the Eager 300 software, provide the Nitrogen, Carbon, Hydrogen and Sulfur percentages contained in the sample.

### Oxygen Determination

No switching of valves. When *Start Analysis* is pressed, the sample, weighed in a silver container and stored in the autosampler, is dropped into the reactor **R2** where it undergoes instant pyrolysis.

During pyrolysis, N<sub>2</sub>, CO and H<sub>2</sub> form. The pyrolysis products cross the adsorption filter **F** where halogenated compounds (Chlorine, Bromine, etc.) are retained.

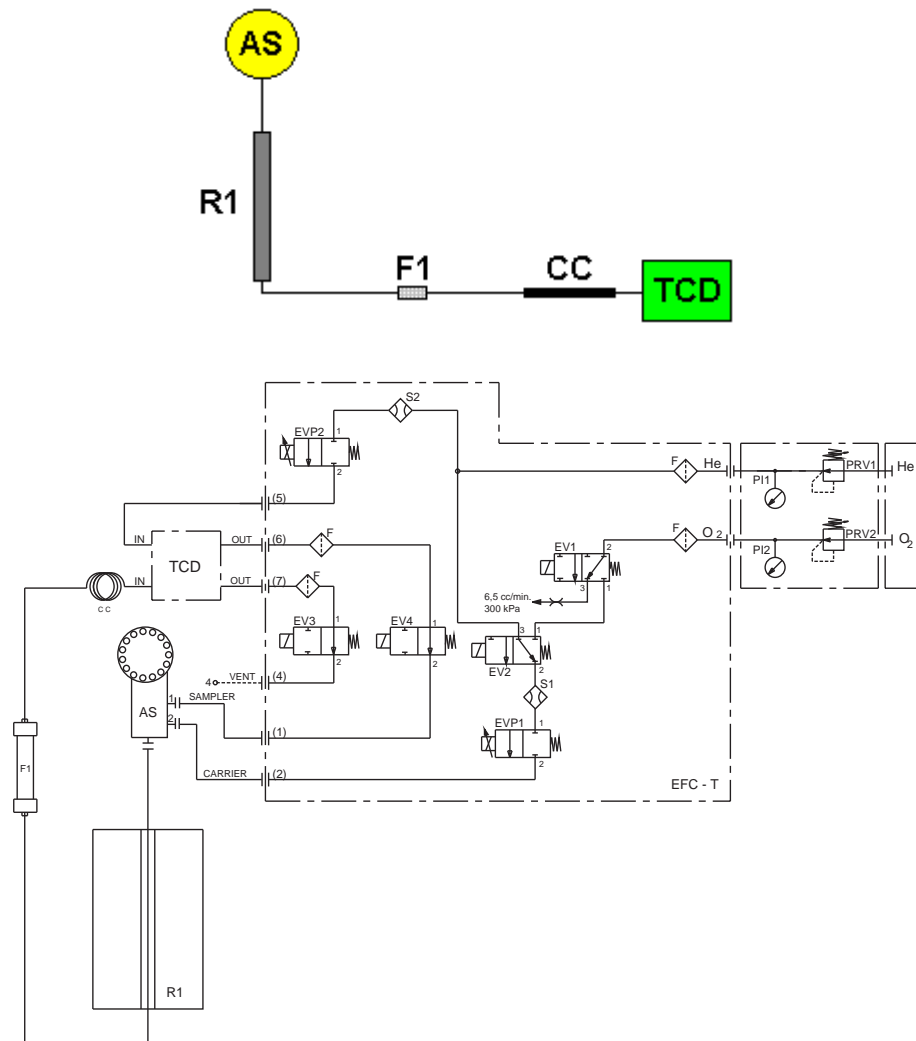
The gas mixture flows into the chromatographic columns **CC2** where carbon monoxide is separated from the other gases.

Then the eluted gases are conveyed to the thermal conductivity detector **TCD** that generates an electrical signal, which, properly processed by the Eager Xperience software, provides the Oxygen percentage.

## Analytical Method for S (Sulfur) Configuration

An autosampler **AS** is connected to a reactor **R1** placed in a furnace at the temperature of 900°C. To the reactor outlet an adsorption filter **F1** is connected. The **F1** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 74.** Instrument parts diagram and pneumatic diagram for S (Sulfur) determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to the relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the Helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed.



The Oxygen line **O2** is connected to the solenoid valve **EV1**.  
This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen flow, whereas the solenoid valve **EV2** allows Helium to flow in the circuit.

When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

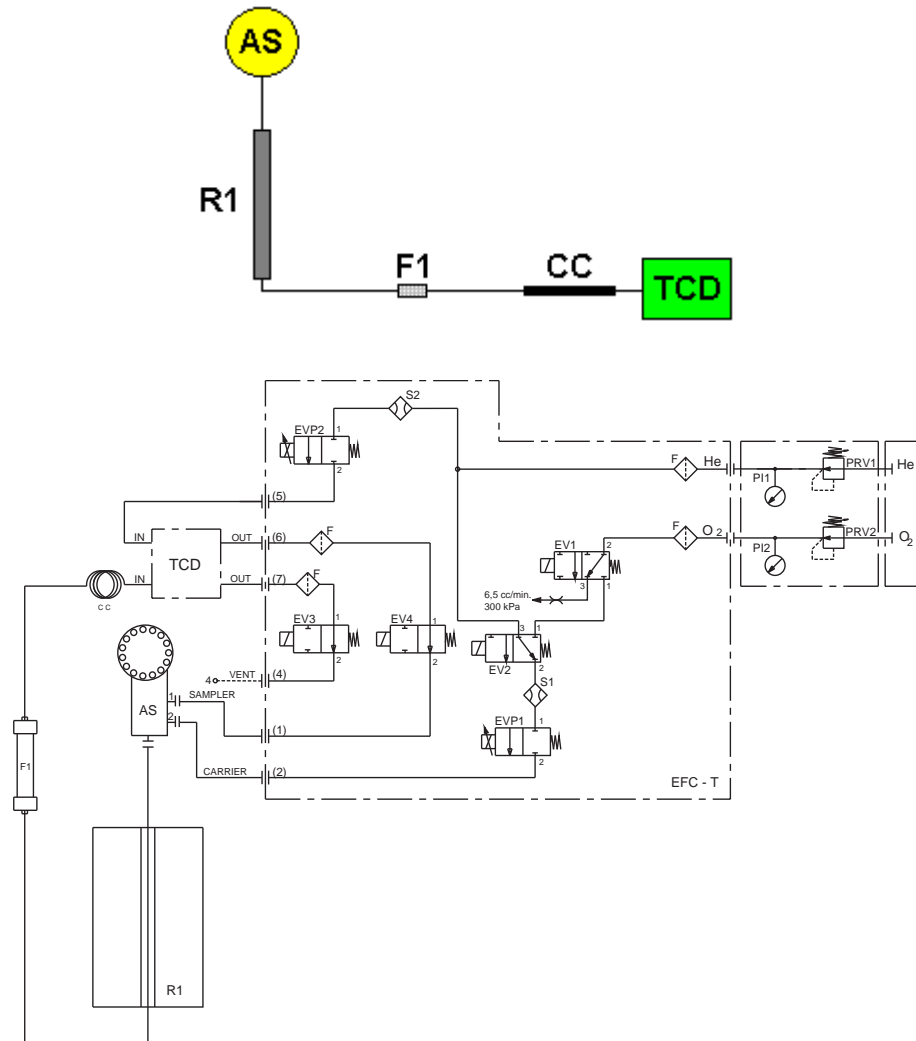
Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow. The combustion products are conveyed across the reactor **R1** where oxidation is completed. Nitrogen oxides and sulfur trioxide possibly formed are reduced to elemental nitrogen and sulfur dioxide, and the Oxygen excess is retained. Then the gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) flows through the adsorption filter **F1**, which retains water, then into the chromatographic column **CC1** where separation takes place. The eluted gases are conveyed to the thermal conductivity detector **TCD** that generates an electrical signal, which, properly processed by the Eager Xperience software, provides the Sulfur percentage.

## Analytical Method for O (Oxygen) Configuration

An autosampler **AS** is connected to a reactor **R1** placed in an furnace at the temperature of 1060 °C. To the reactor outlet an adsorption filter **F1** is connected. The filter **F1** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 75.** Instrument parts diagram and pneumatic diagram for O (Oxygen) determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to the relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**. The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the Helium flow as far as the solenoid valve **EV4**.

This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed.

The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

No switching of valves. When *Start Analysis* is pressed, the sample, weighed in a tin container and stored in the autosampler, is dropped into the reactor **R1** where it undergoes instant pyrolysis.

During pyrolysis,  $N_2$ , CO and  $H_2$  form. The pyrolysis products cross the adsorption filter **F** where halogenated compounds (Chlorine, Bromine, etc.) are retained.

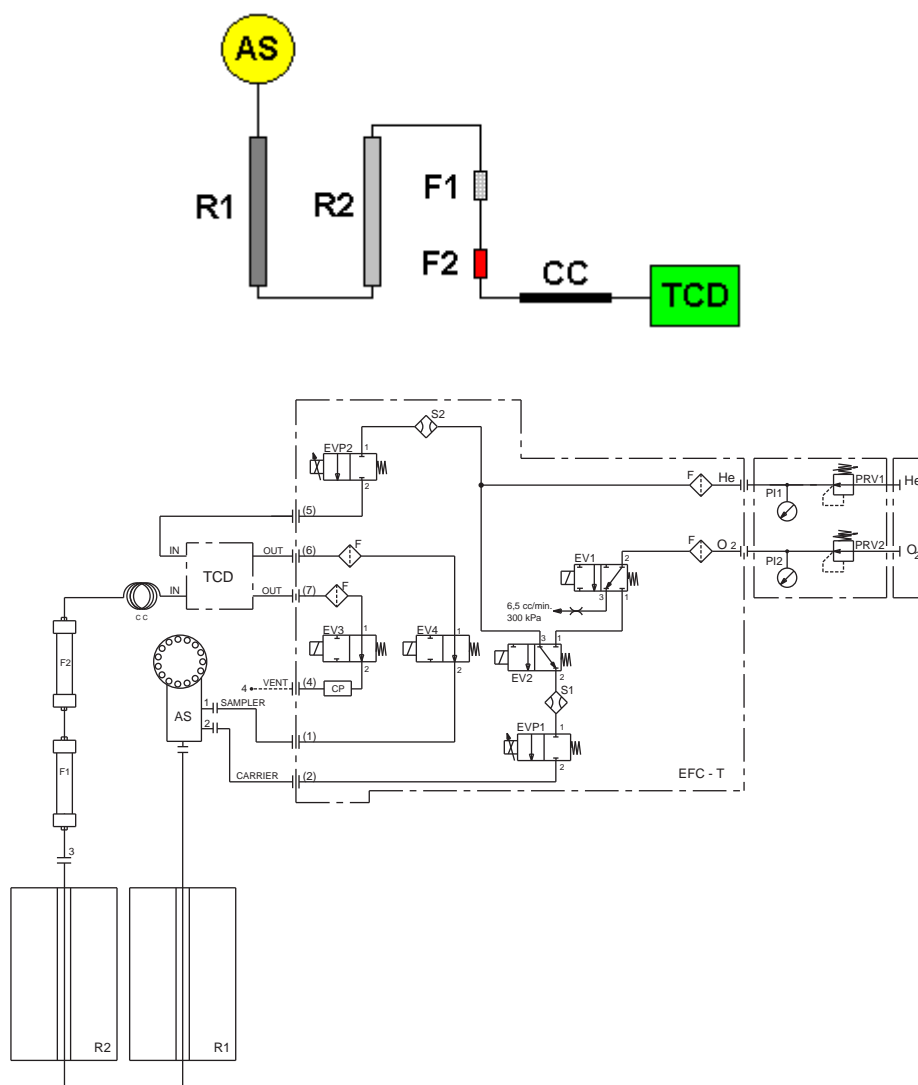
The gas mixture flows into the chromatographic column **CC** where carbon monoxide is separated from the other gases.

Then the eluted gases are conveyed to the thermal conductivity detector **TCD** that generates an electrical signal, which, properly processed by the Eager Xperience software, provides the Oxygen percentage.

## Analytical Method for N (Nitrogen) Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of 900 °C. This reactor, on its turn, is connected in series to a second reactor **R2** placed in an furnace at the temperature of 680 °C. To the reactor **R2** outlet two filters **F1** e **F2** are connected in series. The filter **F2** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 76.** Instrument parts diagram and pneumatic diagram for N (Nitrogen) determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to the relevant proportional valves **EVP1** and **EVP2**. The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the helium flow as far as the solenoid valve **EV4**.

This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit.

When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time. After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor. tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction.

Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow.

The gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) generated by combustion is conveyed across the reactor **R1** where the oxidation of components is completed. Then, the mixture crosses the reactor **R2** where nitrogen oxides possibly formed are converted into elemental nitrogen, and the Oxygen excess is retained.

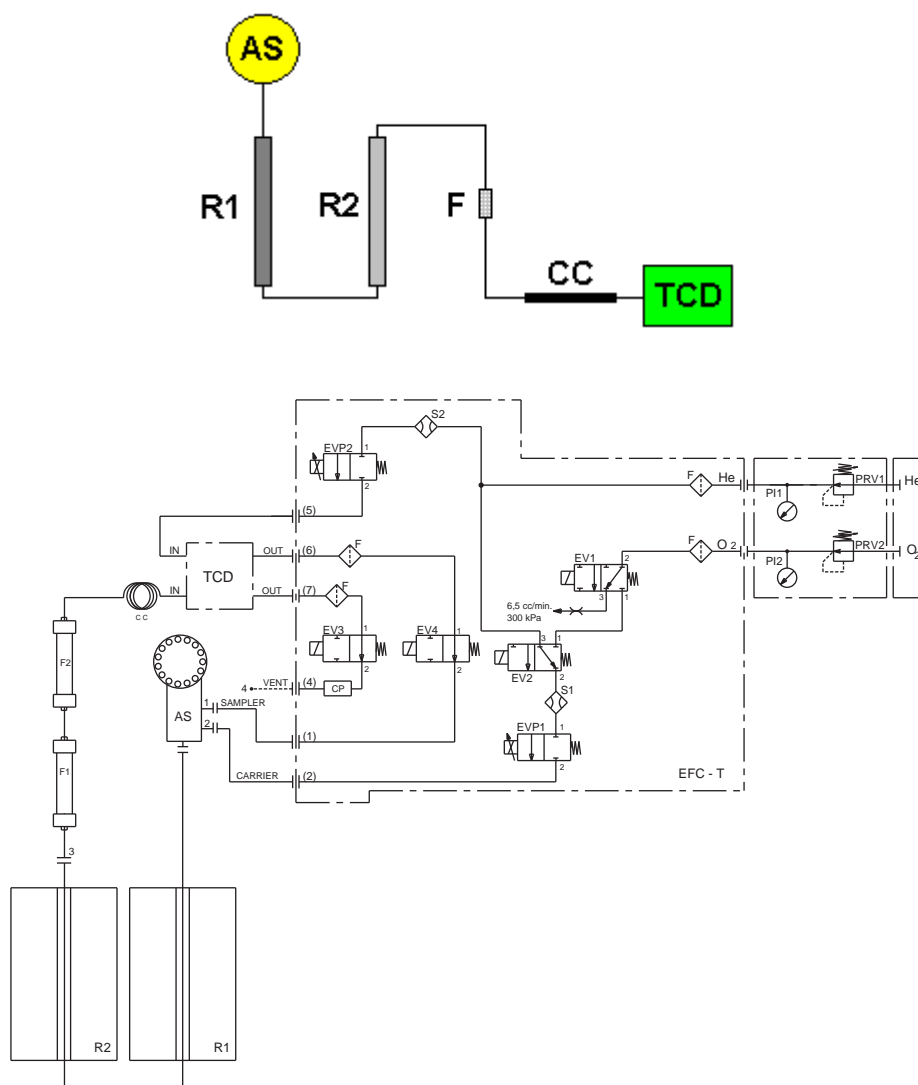
Then the gas mixture passes across the two adsorption filters **F1** e **F2** connected in series. The first filter holds back carbon and sulfur dioxides, whereas the second filter retains water.

Nitrogen is then eluted in the chromatographic column **CC** and conveyed to the thermal conductivity detector **TCD** that generates an electrical signal, which, properly processed by the Eager Xperience software, provides the Nitrogen percentage.

## Analytical Method for NC Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of 900 °C. This reactor, on its turn, is connected in series to a second reactor **R2** placed in an furnace at the temperature of 680 °C. To the **R2** outlet an adsorption filter **F1** is connected. The filter **F1** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 77.** Instrument parts diagram and pneumatic diagram for NC determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**. The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent 4.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the Helium flow as far as the solenoid valve **EV4**.

This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow.

The gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) generated by combustion is conveyed across the reactor **R1** where the oxidation of components is completed. Then, the mixture crosses the reactor **R2** where nitrogen oxides possibly formed are converted into elemental nitrogen, and the Oxygen excess is retained.

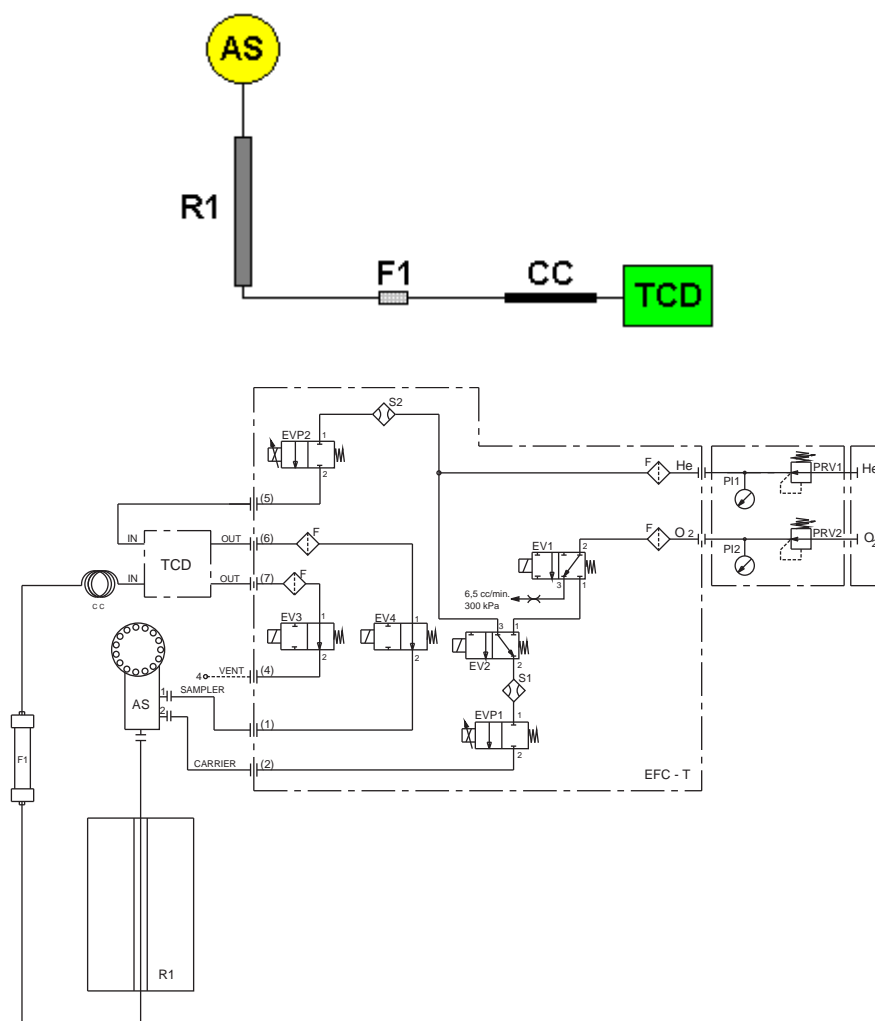
**Note** Sulfur and halogenated compounds (Chlorine, Bromine, etc.), possibly present in the sample, do not affect analysis, since the silver-plated cobalt oxide catalyst holds back both SO<sub>2</sub> and halogens.

The gas mixture then crosses the adsorption filter **F1** that retains water. Nitrogen and carbon are eluted in the chromatographic column **CC** and then conveyed to the thermal conductivity detector **TCD** that generates electrical signals, which, properly processed by the Eager Xperience software, provide the Nitrogen and Carbon percentages.

## Analytical Method for NCS Configuration

An autosampler **AS** is connected to a reactor **R1** placed in an furnace at the temperature of 900 °C. To the reactor outlet an adsorption filter **F1** is connected. The filter **F1** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 78.** Instrument parts diagram and pneumatic diagram for NCS determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the Helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed.



The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow. The gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) generated by combustion is conveyed across the reactor **R1** where oxidation is completed. Nitrogen oxides and sulfur trioxide possibly formed are converted into elemental nitrogen and sulfur dioxide, and the Oxygen excess is retained.

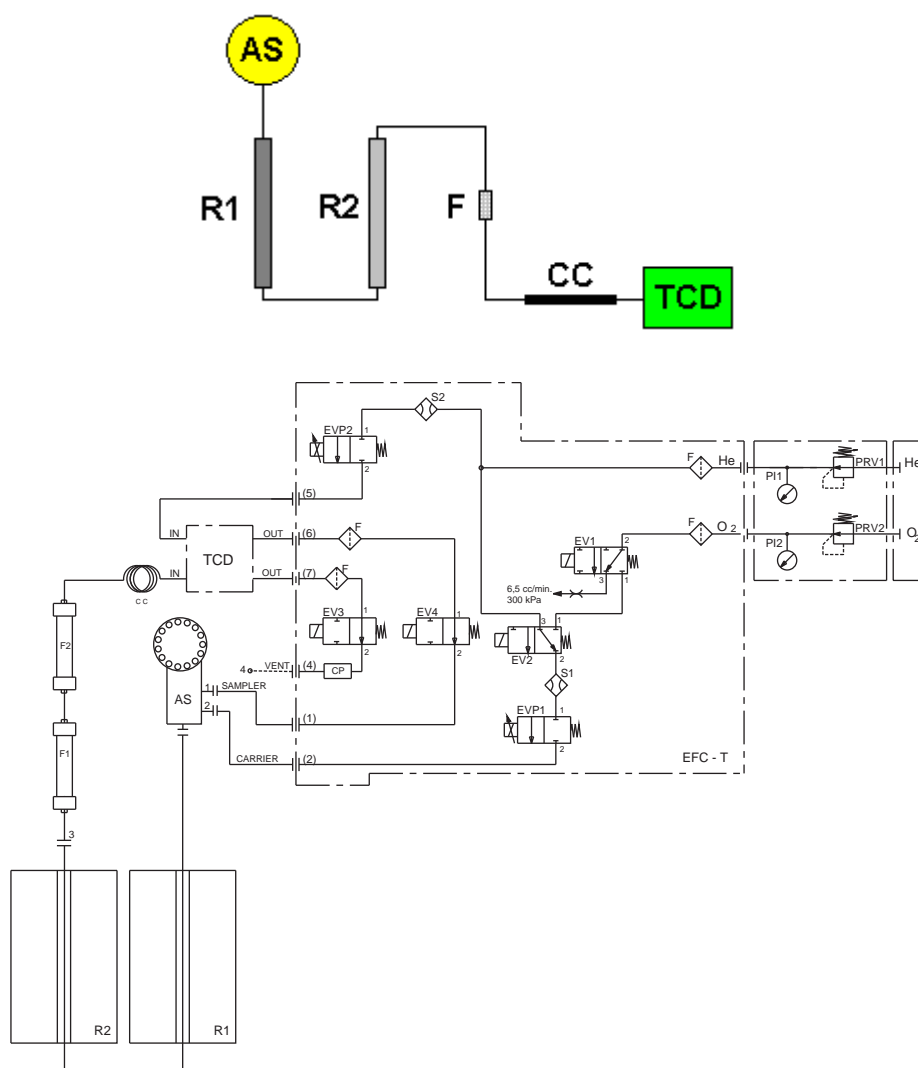
Then the gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) crosses the adsorption filter **F1** that retains water, and flows to the chromatographic column **CC1** where separation occurs.

The eluted gases are conveyed to the thermal conductivity detector **TCD** that generates electrical signals, which, properly processed by the Eager Xperience software, provide Nitrogen, Carbon and Sulfur percentages.

## Analytical Method for NC-Soils, NC-Sediments and NC-Filters Configurations

An autosampler **AS** is connected to a steel reactor **R1** placed in an furnace at the temperature of 900 °C. This reactor on its turn is connected to a second reactor **R2** placed in an furnace at the temperature of 680 °C. To the **R2** outlet an adsorption filter **F1** is connected. The filter **F1** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 79.** Instrument parts diagram and pneumatic diagram for NC-Soils, Sediments and Filters determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**. The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow.

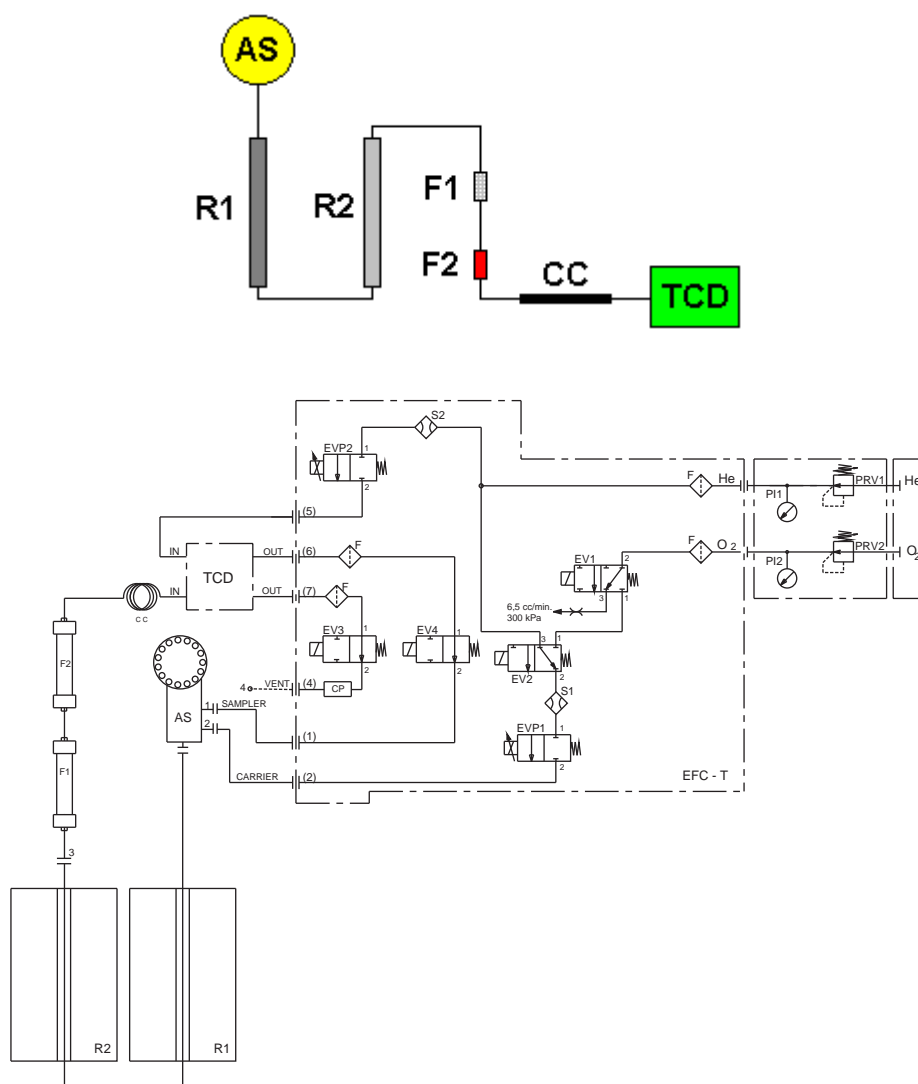
The gas mixture ( $N_2$ ,  $CO_2$ ,  $H_2O$  e  $SO_2$ ) generated by combustion is conveyed across the reactor **R1** where oxidation is completed. Then the mixture crosses the reactor **R2** where nitrogen oxides possibly formed are converted into elemental nitrogen, and the Oxygen excess is retained. Then the gas mixture crosses the adsorption filter **F1** which retains water.

Nitrogen and Carbon are then eluted in the chromatographic column **CC** and conveyed to the thermal conductivity detector **TCD** that generates electrical signals, which, properly processed by the Eager Xperience software, provide the Nitrogen and carbon percentages contained in the sample.

## Analytical Method for N Lubricant, N/Protein and N-Brew Configurations

An autosampler **AS** is connected to a steel reactor **R1** placed in an furnace at the temperature of 950 °C. This reactor on its turn is connected to a second reactor **R2** placed in an furnace at the temperature of 840 °C. To the **R2** outlet two filters **F1** and **F2** are connected in series. The filter **F2** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**.

**Figure 80.** Instrument parts diagram and pneumatic diagram for N-Lubricant, N/Protein and N-Brew determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**.

The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**. The proportional valve **EVP2**, connected to the detector reference channel **RF**, controls the Helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach the point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion.

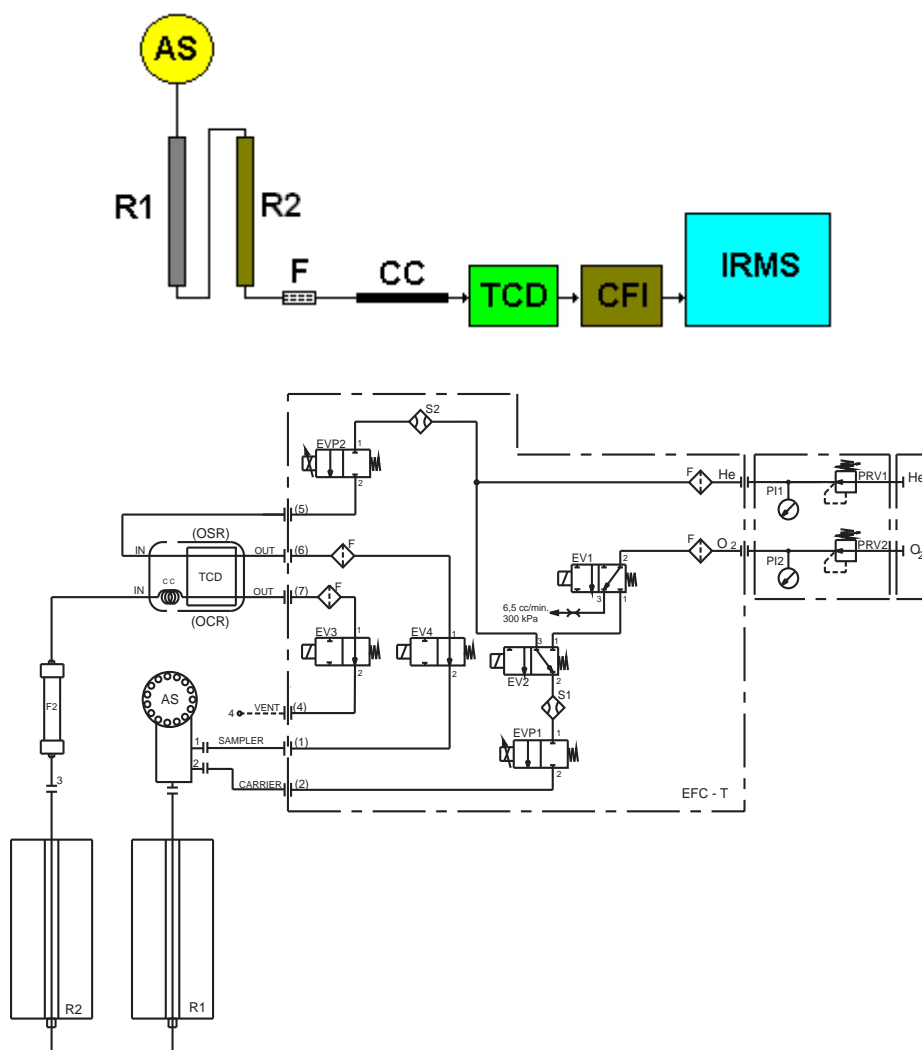
At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow. The gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) generated by combustion is conveyed across the reactor **R1** where oxidation is completed. Then the mixture crosses the reactor **R2** where nitrogen oxides possibly formed are converted into elemental nitrogen, and the Oxygen excess is retained.

Then the gases pass through the two adsorption filters **F1** and **F2** connected in series. The first filter retains carbon and sulfur dioxides, whereas the second filter holds back water. Nitrogen is then eluted in the chromatographic column **CC** and conveyed to the thermal conductivity detector **TCD** that generates an electrical signal, which, properly processed by the Eager Xperience software, provides the nitrogen-protein percentage.

## Analytical Method for NC-IRMS Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of 900 °C. This reactor, on its turn, is connected in series to a second reactor **R2** placed in an furnace at the temperature of 680 °C. To the **R2** outlet an adsorption filter **F1** is connected. The filter **F1** outlet is connected to the analytical column **CC**, on its turn connected to the thermal conductivity detector **TCD**, the continuous flow interface **CFI** and to the isotopic ratio mass spectrometer **IRMS**.

**Figure 81.** Instrument parts diagram and pneumatic diagram for NC-IRMS determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**. The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent **4**.

The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the Helium flow as far as the solenoid valve **EV4**.

This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R1** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor.

Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction. Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow.

The gas mixture (N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O and SO<sub>2</sub>) generated by combustion is conveyed across the reactor **R1** where the oxidation of components is completed. Then, the mixture crosses the reactor **R2** where nitrogen oxides possibly formed are converted into elemental nitrogen, and the Oxygen excess is retained.

**Note** Sulfur and halogenated compounds (Chlorine, Bromine, etc.), possibly present in the sample, do not affect analysis, since the silver-plated cobalt oxide catalyst holds back both SO<sub>2</sub> and halogens.

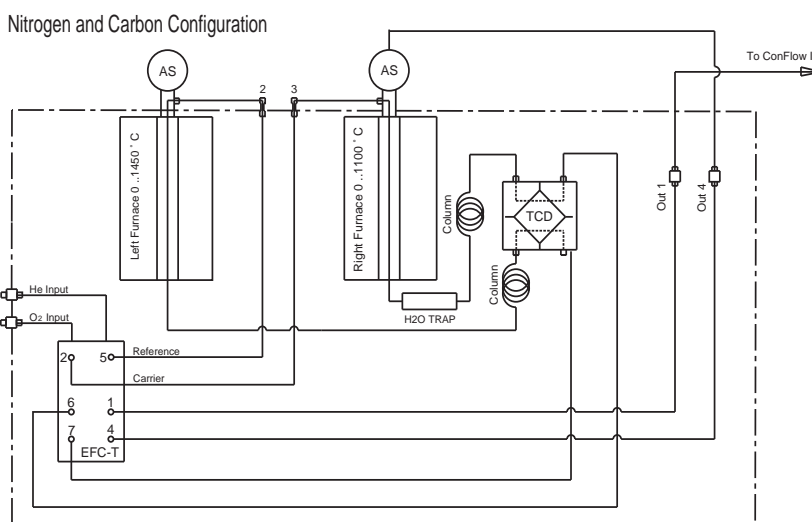
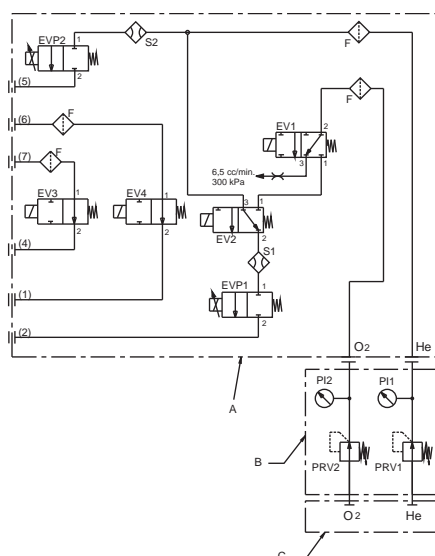
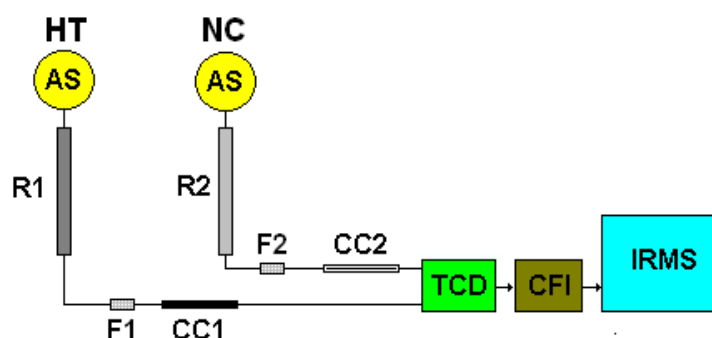
The gas mixture then crosses the adsorption filter **F2** that retains water. Nitrogen and carbon are eluted in the chromatographic column **CC** and then conveyed to the thermal conductivity detector **TCD** (it generates electrical signals which, properly processed by the Eager Xperience software, provide the Nitrogen and Carbon percentages) to the **CFI** device and to the **IRMS** detector.

## Analytical Method for NC-HT Configuration

**IMPORTANT** Only the NC configuration is considered

An autosampler **AS** is connected to a quartz reactor **R2** placed in the right furnace at the temperature of 950 °C. To the **R2** outlet an adsorption filter **F2** is connected. The filter **F2** outlet is connected to the analytical column **CC2**, on its turn connected to the thermal conductivity detector **TCD**, the continuous flow interface **CFI** and to the isotopic ratio mass spectrometer **IRMS**.

**Figure 82.** Instrument parts diagram and pneumatic diagram for NC-HT determination



### Pneumatic Diagram Description

Helium **He** flows to the flow sensor **S1**, through the solenoid valve **EV2**, and directly to the flow sensor **S2**. Both flow sensors are connected to relevant proportional valves **EVP1** and **EVP2**. The proportional valve **EVP1**, connected to the autosampler **AS**, controls the Helium flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts Helium to the atmosphere through Vent 4.



The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the Helium flow as far as the solenoid valve **EV4**. This valve, normally open, allows Helium to reach point 1 of the autosampler and purge the zone where the sample is housed. The Oxygen line **O2** is connected to the solenoid valve **EV1**. This valve controls the Oxygen inlet.

## Sequence of the Method Stages

During pre-analysis the solenoid valve **EV1** shuts off Oxygen, whereas the solenoid valve **EV2** allows Helium to flow in the circuit. When *Start Analysis* is pressed, the valve **EV1** opens, whereas the valve **EV2** switches to allow Oxygen to flow in as far as the combustion reactor **R2** for a preset time.

After a few seconds, the sample, weighed in a tin container and stored in the autosampler, is dropped into the combustion reactor. Tin, coming in contact with an extremely oxidizing environment, triggers a strong exothermic reaction.

Temperature rises to approximately 1800 °C instantly causing the sample combustion.

At the end of the time set for Oxygen introduction, the valves **EV1** and **EV2** return to their original position restoring the Helium flow. The gas mixture generated by combustion is conveyed across the reactor **R2** where the oxidation of components is completed.

**Note** Sulfur and halogenated compounds (Chlorine, Bromine, etc.), possibly present in the sample, do not affect analysis, since the silver-plated cobalt oxide catalyst holds back both SO<sub>2</sub> and halogens.

The gas mixture then crosses the adsorption filter **F2** that retains water. Nitrogen and carbon are eluted in the chromatographic column **CC2** and then conveyed to the thermal conductivity detector **TCD** (it generates electrical signals which, properly processed by the Eager Xperience software, provide the Nitrogen and Carbon percentages) to the **CFI** device and to the **IRMS** detector.

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Part

1

# Analysis

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# Instrument Start-up

## Contents

- Introduction
- Powering on the System
- Installation of Eager Xperience
- Eager Xperience Main Menu
- Analytical Configuration
- Leak Test
- Detector Signal Level

## Introduction

To analyze any type of sample the instrument must be in the correct operating conditions.

Proceed according to the following operating sequences:

- Powering on
- Installation of Eager Xperience software into the PC
- Analytical configuration
- Leak checking
- Adjustment of the detector signal level



**CAUTION** Before starting the operating sequences, make sure that instrument, reactors, adsorption filters, autosampler (or manual injection device for liquids) and any complementary units are properly installed as described in previous chapters.

## Powering on the System



Switch on the instrument lifting the breaker located at the back of the instrument (position I). At the powering on, the indicating LED **Power On** on the synoptic panel light up. Switch on the computer and any complementary units by means of relevant switches.

## Installation of Eager Xperience

### ❖ To Install Eager Xperience

#### Material required

Eager Xperience package


The software *Eager Xperience* can be installed in a system provided with Windows™ 2000/XP/Vista operating systems. The free space on the PC hard disk must be at least 30 MB. *Eager Xperience* is installed by using the CD provided in the standard outfit and operating as follows:

1. When the CD is introduced into the CD driver of the PC, the installation menu shown in [Figure 83](#) is displayed.

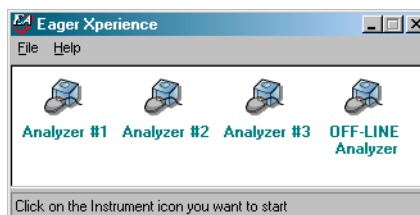
**Figure 83.** Eager Xperience installation menu



**Note** If the installation menu does not automatically appear, start the CD *Autorun* program through the Windows™ Start-Run command.

2. Start installation by clicking the push-button  **Install Eager Xperience for Flash**.
3. Follow the instructions prompted step by step.
4. At the end of installation, in the page **Start-Program Eager Xperience**, double-click the **Eager Xperience for Flash** icon. The window of [Figure 84](#) is displayed.

**Figure 84.** Selection of the instrument



5. 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

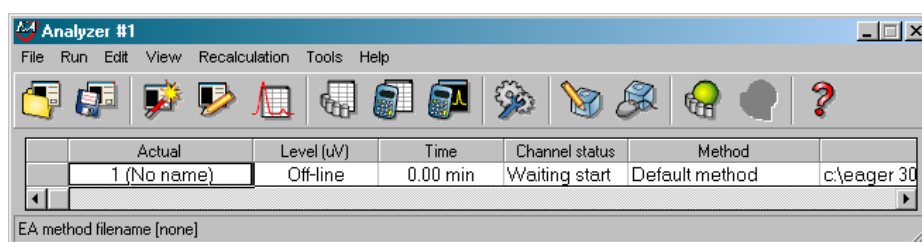
below the icon can be changed. To do this, click on the existing name and overwrite the new one.

6. *Eager Xperience* proceed 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 Xperience* again selecting **Start > Programs > Eager Xperience > Eager Xperience for FlashEA**.
7. Follow the prompted indications. At the end of the installation, the Main Menu is displayed.

## Eager Xperience Main Menu

The Main Menu of *Eager Xperience*, shown in [Figure 85](#), is the starting point to enter all menus and relevant functions. Menus and icons of Main Menu are described in the following [Table 41](#) and [Table 42](#) respectively.

**Figure 85.** Eager Xperience Main Menu




**Table 41.** Main Menu: description of the menus

Menu	Description	Submenus and Options
File	This menu contains functions concerning the instrument operation. It is used during the analyzer installation procedure.	<ul style="list-style-type: none"> <li>• Set language</li> <li>• Colour set</li> <li>• Instrument configuration</li> <li>• System administration</li> <li>• Installation qualification</li> <li>• Load method</li> <li>• Load system defined method</li> <li>• Save method</li> <li>• Copy method from...</li> <li>• Printer setup</li> <li>• Print method</li> <li>• Exit Eager Xperience</li> </ul>













**Table 41.** Main Menu: description of the menus, continued

Menu	Description	Submenus and Options
Run	Use this menu to choose the type of start command to be sent to the analyzer, and also to stop the analytical cycle or abort the current analysis.	<ul style="list-style-type: none"> <li>• Start sequence of samples</li> <li>• Stop running sequence</li> <li>• Start single sample data acquisition</li> <li>• Stop data acquisition</li> <li>• Abort data acquisition</li> <li>• Run macro</li> </ul>
Edit	This menu provides functions related to the instrument setup and analytical parameters.	<ul style="list-style-type: none"> <li>• Edit Method</li> <li>• Component table</li> <li>• Sample table</li> <li>• Wizard method development</li> <li>• Edit Elemental Analyzer parameters</li> </ul>
View	Use this menu to monitor the analysis in real time, read the result of the last sample run, check the calibration curve, compare and overlay chromatograms, check the instrument status and maintenance.	<ul style="list-style-type: none"> <li>• View sample being acquired</li> <li>• Last sample calculated results</li> <li>• View Calibration curve</li> <li>• View Chromatograms</li> <li>• Overlay Chromatograms</li> <li>• Operate on Chromatograms</li> <li>• Compare Chromatograms</li> <li>• View Elemental Analyzer Status</li> <li>• View Maintenance</li> </ul>
Recalculation	Use this menu to cancel the calibration curve and the results of previous analyses. You can recalculate previous results individually or sequentially. It also provides the summary of results.	<ul style="list-style-type: none"> <li>• Reset calibration factor</li> <li>• Recalculation</li> <li>• Summarize results</li> </ul>
Tools	Use this menu when the ashes removal and/or reactor replacement is required as maintenance	<ul style="list-style-type: none"> <li>• Ashes removal</li> <li>• Reactor replacement</li> <li>• Cleaning the MAS piston</li> </ul>
Help	Use this menu to enter Eager Xperience help program. It is subdivided into different modules, each one designed to cover specific issues of the module currently in use.	<ul style="list-style-type: none"> <li>• Help</li> <li>• About Eager Xperience</li> </ul>

**Table 42.** Main Menu: description of the icons

Icon	Function	Description
	Load method	Use this icon to load a previously saved analytical method.

**Table 42.** Main Menu: description of the icons, continued

Icon	Function	Description
	Save method	Use this icon to store new operating methods.
	Wizard method development	Use this icon to develop new operating methods
	Edit method	This icon accesses to the integration and calculation parameters, and to the parameters for printing analytical reports.
	Components Table	This table contains the stored retention times, which allow to identify N, C, H, S and O.
	Sample Table	This table contains all functions related to sample records, and the function allowing communication with the balance.
	Summarize results	This feature contains analytical results, print options and chromatograms.
	Recalculation	This function allows to recalculate previous results.
	View Maintenance	This icon allows to program current maintenance by recording the number of analyses run by each reactor of the analytical circuit.
	Edit Elemental Analyzer Parameters	Use this icon to open the pages containing the commands for the setting of temperatures, flows, times, detector and the analyzer control functions.
	View Elemental Analyzer Status	This function comprises the pages displaying the analyzer conditions. It contains special functions to check the system pneumatic tightness (Leak Test), to check the baseline level, and to program automatically the “Autoready” function.
	Start sequence	Use this icon to start a series of analyses having different current and timed requirements. At the end of the analytical cycle, the instrument can either be put in Standby Mode or the furnace and detectors be switched off, or the gas flows turned off.
	Stop sequence	Use this function to stop in any moment the sequence of analyses only completing the current run.

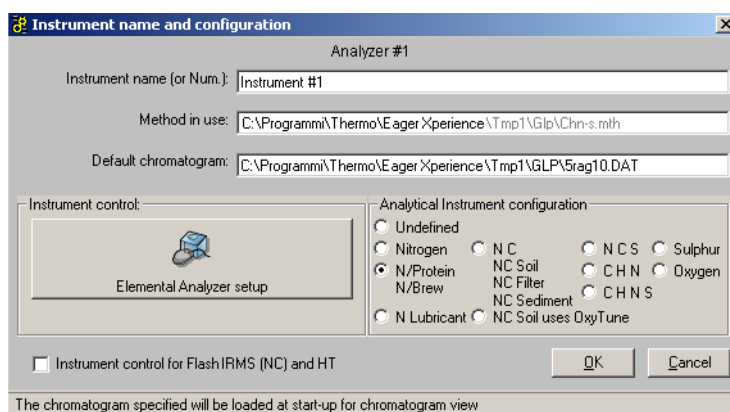
## Analytical Configuration

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

1. In Main Menu of [Figure 85](#) on [page 141](#), choose **File > Instrument Configuration**. The following window is displayed.

**Figure 86.** Instrument name and configuration



- a. In the section **Instrument name**, type the instrument **serial number** (6 digits; for example 991234). See the label located on the instrument rear panel.
- b. In the section **Analytical instrument configuration** select the configuration of your instrument.



**CAUTION** NCS, CHN, CHNS, Sulfur and Oxygen configurations use the LEFT furnace only. Do not set any temperature for the RIGHT furnace.

**Note** The option **Undefined** can be used in case the desired operating conditions are different from those defined for the instrument configuration.

- c. If the instrument is coupled with an IRMS detector, check the function **Simple Instrument control for IRMS**. At the reboot of Eager Xperience, the simplified Main Menu for IRMS is visualized as shown in [Figure 87](#).

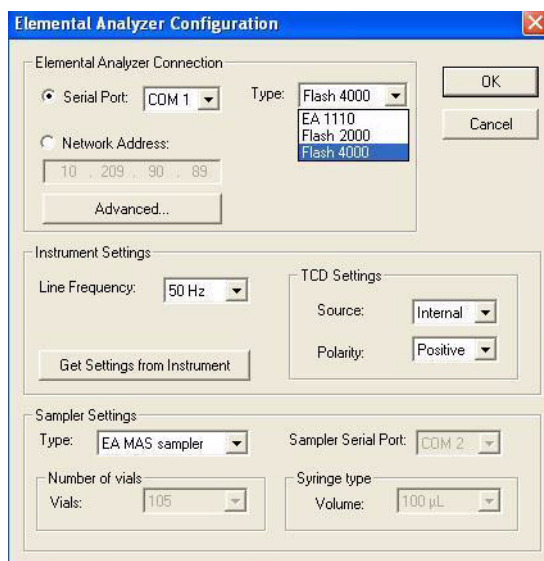
**Figure 87.** Main menu for IRMS



- d. Click **Elemental analyzer setup** to enter the dialog window of [Figure 88](#) where the configuration parameters have to be set.



Figure 88. Configuration dialog window




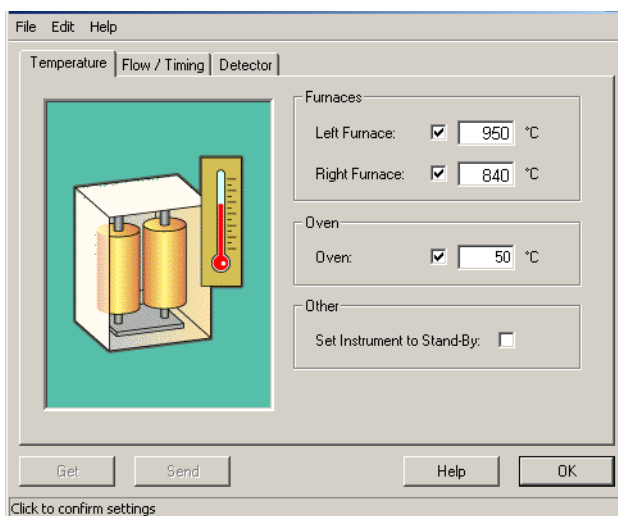
- e. In the section **Elemental Analyzer Connection** select the computer serial port (COM1, COM2 etc.) to which the instrument is connected.
  - f. In the section **Type** verify that the instrument in use is **Flash 2000**.
  - g. In the section **Instrument Settings** choose the following settings:
    - i. Line Frequency = 50 Hz
    - ii. TCD Settings Source = Internal
    - iii. TCD Settings Polarity = Positive
- Note** For the Oxygen determination in CHN-O and CHNS-O configurations, select the negative polarity. If the same configurations two autosamplers are used, refer to the relevant Analytical Method described in Chapter 8.
- h. In the section **Sampler Setting** select the type of autosampler installed on the instrument.
    - i. In the case of autosampler for liquid sample, also specify the computer serial port, to which the autosampler is connected, and the number of vials.
    - ii. Click **Ok** to go back to the window of [Figure 88](#), then click **Ok** to return to Main Menu.
2. In the main menu, select **File** and then the option **Load System Defined Method**. The file name of the loaded method is displayed in the grid **Filename of method in use** of Main Menu.
  3. In Main Menu select **Edit** and then the option **Edit Elemental Analyzer Parameters** or just click the icon . The following window appears where the analyzer operating parameters are displayed.

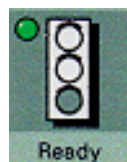
Figure 89. Example of analyzer parameters



- Click **Send** to transfer the operating parameters to the instrument.
- From now on the analyzer is working. The furnaces begin to heat, Helium flows in the circuit and the LEDs Furnace, Oven and TCD lights up on the synoptic panel.



After about 50 minutes the instrument furnaces reach the temperature settings and the LED Ready on the synoptic panel lights up.




The instrument is now ready to run analyses. However, before starting an analytical cycle, a **leak test** must be carried out to check that reactors, filters, if any, and gas chromatographic columns have been properly installed.

## Leak Test

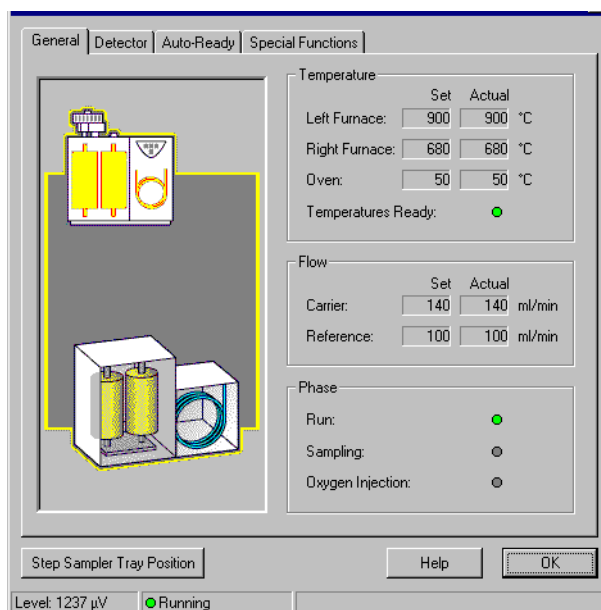
The leak test must be performed any time a component of the pneumatic circuit is replaced. Operate according to the instructions reported in the following operating sequence.

### ❖ To Check the Leaks

- In Main Menu select **View** and then the option **View Elemental Analyzer Status** or just

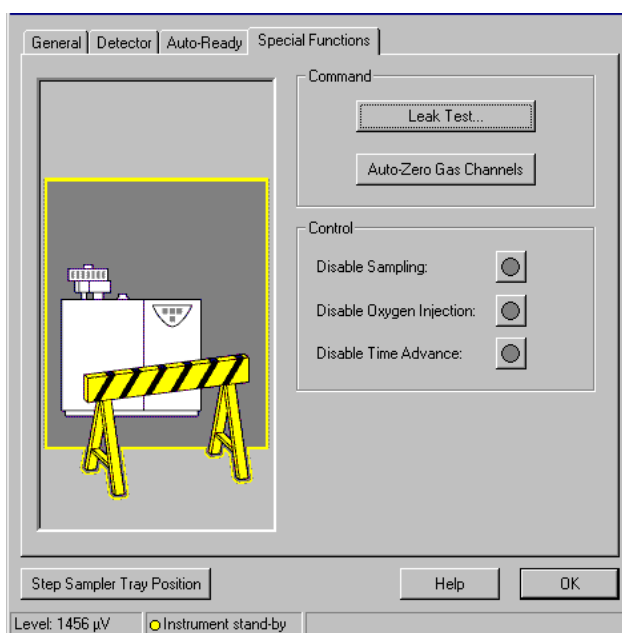
click the icon . The following window appears.

**Figure 90.** Status of the analyzer



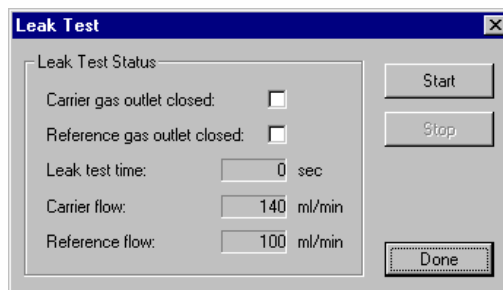
2. Select the option **Special Functions**. The following window will appear.

**Figure 91.** Special function window



3. In the section **Command** click the button **Leak Test**. The box of [Figure 92](#) is displayed with the leak test parameters.

Figure 92. Leal test parameters box



- a. Click **Start** to begin the operation. A window will appear where you will be requested to perform the Autozero. After 300-360 seconds (**Leak test time**), **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.

- b. 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. To terminate the leak test and restore the flow operating values, click **Stop** and **Done**.

## Detector Signal Level

To adjust the level of the TCD detector signal, follow the instructions reported in the following operating sequence.

### ❖ To Adjust the Detector Signal Level


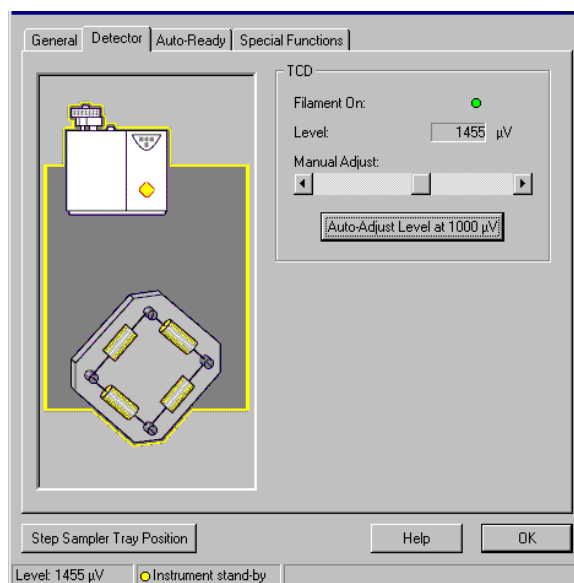
1. In Main Menu select **View > View Elemental Analyzer Status** or justclick the icon . The window of [Figure 90](#) on [page 147](#) will be displayed.
2. Select the option **Detector**. The following window will appear.

Figure 93. Detector parameters status



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



## Guide to Run Analyses

This chapter contains information and operating sequences to run sample analyses, and it also describes the comparison methods for a correct evaluation of results. Practical advice for daily operation is also provided.

### Contents

- [Introduction](#)
- [Directory for Analyses](#)
- [Current Maintenance Program](#)
- [Instrument Calibration](#)
- [Sample Table](#)
- [Determination of the Blank Value](#)
- [Sequence of Analyses](#)
- [Quality Control and Check of Analytical Results](#)
- [Post-Analysis Operations](#)
- [Analytical Troubleshooting](#)

## Introduction

To program and analyze any type of sample, do the following:

- Create a directory of analyses
- Program the current maintenance (recommended operation)
- Choose the calibration
- Set a sample table
- Determine the blank value
- Run the sequence of analyses

**IMPORTANT** Before starting operating sequences, make sure that the instrument start-up operations have been performed as described in [Chapter 9, “Instrument Start-up,”](#)

## Directory for Analyses

Before analyzing a sample, you should create a directory where you will store the operating method comprising:

- Sample table
- Integration parameters
- Calculation parameters
- and all that is necessary to run the analysis.

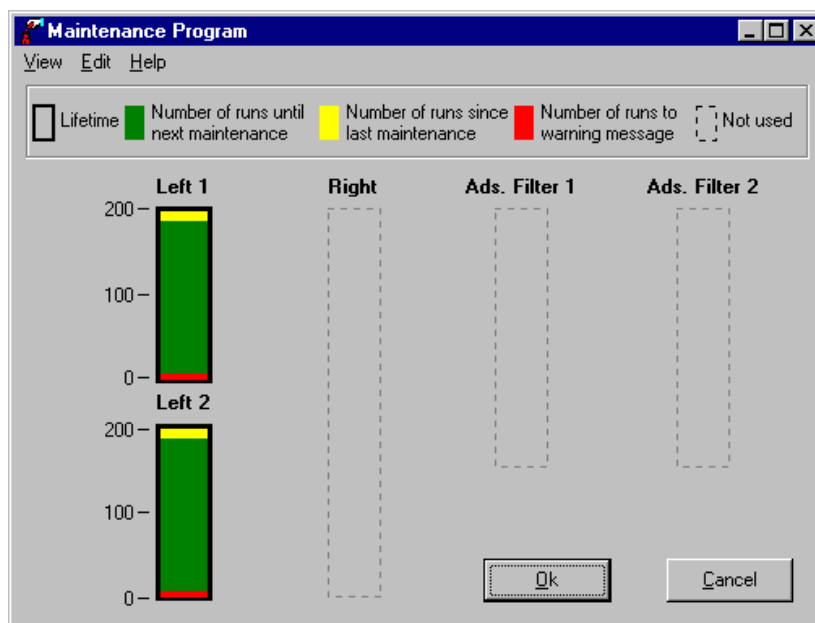
## Current Maintenance Program

Each reactor, each filter and relevant fillings need to be replaced according to the analytical configuration used. For each configuration, an average life of its components has been established. The **View Maintenance** option will indicate when the different components have to be replaced. To start the maintenance program, do what described in the following operating sequence.

### ❖ To Start-up the Current Maintenance Program

1. In the main menu select **View > View Maintenance**. According to your analytical configuration, a window like the following one is displayed.

**Figure 94.** Maintenance program schedule



The example of [Figure 94](#) shows the analytical circuit components for which the maintenance routine is required.



Component	Description
Left	Represents the oxidation reactor In Figure 94 Left 1 represents the crucible or ashes, whereas Left 2 represents the oxidation reactor.
Right	Represents the reduction o pyrolysis reactor.
Ads Filter 1	Represents the first adsorbent filter.
Ads Filter 2	Represents the second adsorbent filter.

In the diagram the active components are indicated with colored areas and show on the left the numerical scale of their lifetime. The meaning of each colored area is indicated in the upper section of the diagram. The components not present in the concerned instrument configuration are indicated by a dashed line.

- To view in detail the default conditions of the components of the concerned instrument configuration, in the menu **Edit** select **Set Maintenance > Default**. A window like the one below will appear. The values shown cannot be changed.

Figure 95. Maintenance: Example of default condition

	Left 1	Left 2	Right	Ads. Filter 1	Ads. Filter 2
<input type="checkbox"/> Lifetime	200	200	0	80	0
<input checked="" type="checkbox"/> Number of runs to warning message	10	10	10	10	10
<input checked="" type="checkbox"/> Number of runs until next maintenance	200	200	0	80	0
<input checked="" type="checkbox"/> Number of runs since last maintenance	0	0	0	0	0

Condition	Description
Life time	indicates the preset maximum number of analyses each individual component can perform.
Number of runs to warning message	indicates that when any of the components will still have to run only 10 analyses to reach the number set in Lifetime, each program page will display the message <b>Check Maintenance</b> . If the message is ignored and analyses are continued, when the preset number of runs is reached, the message <b>Alarm</b> will be displayed. This does not stop the analytical cycle.
Number of runs until next maintenance	indicates the number of analyses to be performed before next maintenance
Number of runs since last maintenance	indicates the number of analyses performed after last maintenance

- If you want to use a different maintenance program from the default one, in the menu **Edit**, select the option **Set Maintenance > Manual**. A window like the following will be displayed.

**Figure 96.** Maintenance: Manual program

	Left 1	Left 2	Right	Ads. Filter 1	Ads. Filter 2
<input type="checkbox"/> Lifetime	200	200	0	80	0
<input checked="" type="checkbox"/> Number of runs to warning message	0	0	0	0	0
<input checked="" type="checkbox"/> Number of runs until next maintenance	200	200	0	80	0
<input checked="" type="checkbox"/> Number of runs since last maintenance	0	0	0	0	0

In the window of [Figure 96](#), you can change any value by clicking on the different boxes and entering the desired new value.

## Instrument Calibration

*Eager Xperience* offers three calibration methods:

- K-Factor
- Linear
- Non Linear

All tests are performed with the **K-Factor** method that is generally used by most users. This method consists in obtaining a constant of calculation by means of the following formula:

$$K = \%Th * (I - b) / w$$

where:

*Th* = Theoretical percentage of the standard

*w* = Weight in milligrams of the standard

*I* = Area integral of the standard

*b* = Blank area integral

For the calculation of an **Unknown** sample, *Eager Xperience* will use the reverse formula:

$$\%Unknown = K * w / (I - b)$$

where:

$K$  = K-factor

$w$  = Weight in milligrams of the sample

$I$  = Area integral of the sample


$b$  = Blank area integral

The **Linear** method is generally used when samples very different from each other are analyzed in the same analytical sequence. In this way the errors due to the detector response linearity are minimized.

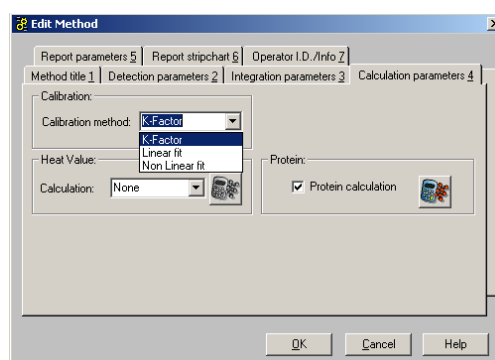
The **Non-Linear** method is used when the analyzer is connected to another detector having a response of exponential type. To select the calibration method or view the calibration curves of a memorized method, do as described in the following operating sequence

To select the calibration method or view the calibration curves of a memorized method, do what described in the following operating sequence.

#### ❖ To Calibrate Method and Curves

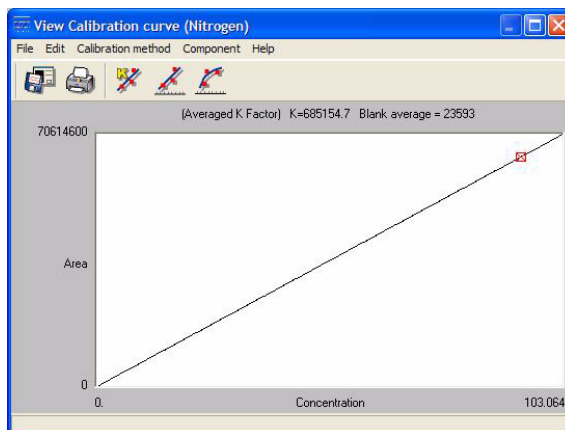
1. Select the calibration method.
  - a. In Main Menu choose **Edit > Method** or just click the  icon. The following window will be displayed:

**Figure 97.** Method editor



- b. Select the option **Calculation Parameter**.
      - i. In the section **Calibration Method**, select the calibration method required.
      - ii. If desired, in the section **Heat value**, select in the box **Calculation** the type of sample between the options **None**, **Liquids** and **Solids**. Click on the icon shown on the box right side to display the calculation scheme related to the selected option.
      - iii. In the case of **N/Protein** and **N-Brew** configurations, in the section **Protein**, tick **Protein Calculation**. Click on the icon shown on the box right side to display the relevant calculation scheme.
2. View the calibration curves.
  - a. At the end of analyses of the standard samples, it is possible to visualize the calibration curve operating as follows:
  - b. In Main Menu choose **View > View Calibration curve**. A window similar to one below will be displayed.

Figure 98. Example of Calibration curves

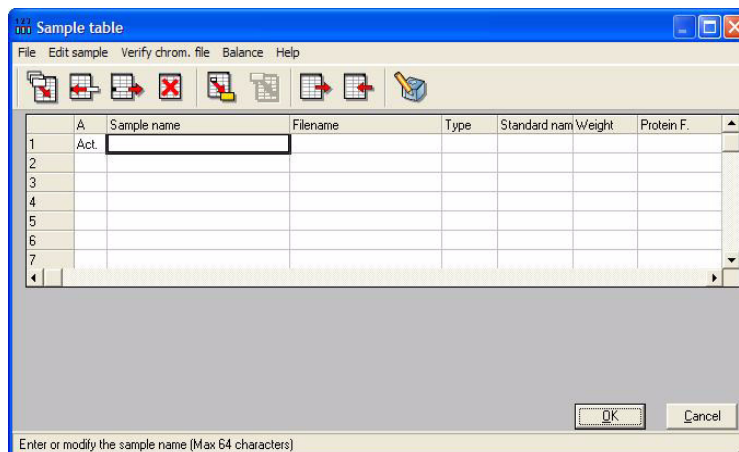


- c. Select **Calib. Method**. According to the calculation method, the calibration points with peak area and concentration will be displayed.

## Sample Table

The sample table of the analytical method contains all information concerning the series of samples to be acquired and processed. To enter the sample table, do as described in the following operating sequences.

Figure 99. Example of Sample table



### ❖ To Fill Sample Table


1. Select **Edit sample > Fill Sample table** or just click the icon . The following window will be displayed, which offers all the functions necessary to fill, change or cancel Sample Table.

Figure 100. Window to fill Sample Table

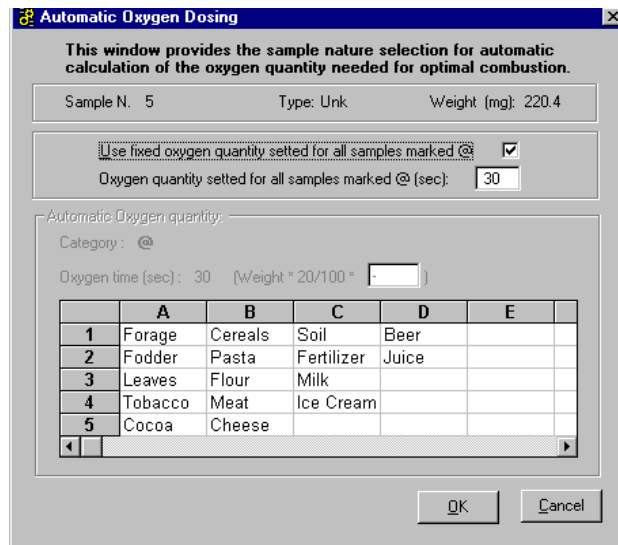
The screenshot shows a software dialog box titled "Fill samples table". It is organized into several sections:

- Samples:** Includes text boxes for "Sample name" (with "Name" entered), "Filename" (with "File" entered), and three numeric input boxes: "No. samples" (1), "Sample name idx" (1), and "Filename idx" (1). There are radio buttons for "Unknown" (selected) and "Standard".
- Instrument methods:** Includes a dropdown menu for "AS Method", a "Repeat" dropdown (1), a checked "Increase Vial #" checkbox, and a "Vial #" dropdown (1).
- Bottom section:** Includes input boxes for "Weight" (1), "Protein factor" (6.25), and a dropdown for "Category" (A).

At the bottom right of the dialog are three buttons: "Replace", "Add", and "Cancel".

- a. In the section **Samples** edit as follows:
  - i. Do not enter **Sample Name** now. Sample name, or sample monogram will be entered from time to time as required.
  - ii. In the text box **Filename**, enter the filename to be used to save the sample.
  - iii. In the box **Number samples**, enter the number of samples, up to 200, to be analyzed.
  - iv. Check that **Unknown** is ticked
2. Leave **Sample name idx** and **Filename idx** set as 1.
  - a. In the section **Sampler**, enter the parameters if the instrument is equipped with the autosampler for liquids.
  - b. In the left bottom section, enter the following.
    - i. Leave **Weight** set as **1**.  
The weight is entered time by time. In case of direct injections of constant volumes, the sample volume can be entered directly.
    - ii. Set **Protein factor** only if the instrument configuration is N/Protein or N-Brew. For all other configurations set 0 (zero).
    - iii. Select the **Category** to which the sample belongs only if the instrument configuration is N/Protein or N-Brew. For all other configurations this parameter is not edited. To enter the sample category, press the arrow. The following window is displayed.

Figure 101. Window to select the sample category



- At the end of editing, click **Replace**. The sample table appears again. In the sample table grid you will find all information entered.

**Note** In the case of liquid samples analyses with the sampler for liquid, the column Density will be displayed in the sample table grid in which insert the density of the liquid sample that will automatically be turned into weight.

The following Figure 102 and show an example of edited sample table referred to the N/Protein instrument configuration.

Figure 102. N/Protein: Window of sample table editing

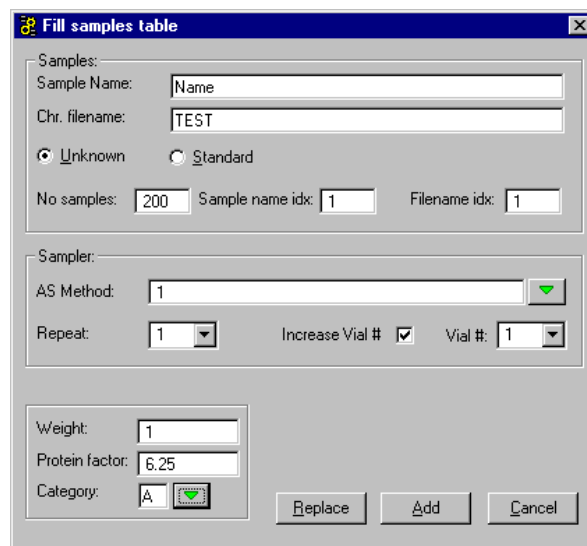


Figure 103. N/Protein: Example of sample table

ID	Sample name	Filename	Type	Std name	Weight	Protein Factor	Category	AS	Vial
1	Act. Name001	TEST001	Unk		1	6.25	@		
2	Name002	TEST002	Unk		1	6.25	@		
3	Name003	TEST003	Unk		1	6.25	@		
4	Name004	TEST004	Unk		1	6.25	@		
5	Name005	TEST005	Unk		1	6.25	@		
6	Name006	TEST006	Unk		1	6.25	@		
7	Name007	TEST007	Unk		1	6.25	@		

Enter or modify the sample name (Max 64 characters) WARNING!! MAINTENANCE REQUIRED (Left 1) AT SAMPLE N.-104

## Determination of the Blank Value

For the determination of the blank value, do what described in the following operating sequence.

### ❖ To Determine the Blank Value


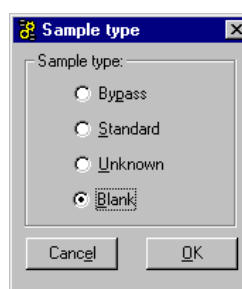
1. Put in the autosampler an airtightly closed container. When N/Protein is used, it is suggested to introduce about 50-80 mg of sugar into the container to avoid that a high quantity of oxygen freely flows on the reduction reactor.
2. In Main Menu select **Edit > Sample table** or just click the icon . The sample table appears. See Figure 99 on page 156.
  - a. In the sample table grid, click on the column **Type**. Click on the arrow displayed, and the following window will appear for the sample type selection.

Figure 104. Window for the sample type selection



- b. In the window of Figure 104 select **Blank**, then click **OK** to confirm and go back to the sample table.
    - c. Click **OK** to confirm and go back to Main Menu.
3. In Main Menu, select the menu **Run > Start Single Sample Data Acquisition**.
4. Once the analysis run time has elapsed, compare the chromatogram obtained with that of the final test provided with the instrument.

To compare the chromatogram obtained with that of the final test and to check the blank value, there are two procedures according to whether a printer is available or not.

- In the first case refer to the operating sequence “To Check the Blank Value with a Printer Available” on page 160.
- In the second case refer to the operating sequence “To Check the Blank Value with No Printer Available” on page 162.

#### ❖ To Check the Blank Value with a Printer Available

Operate as follows:


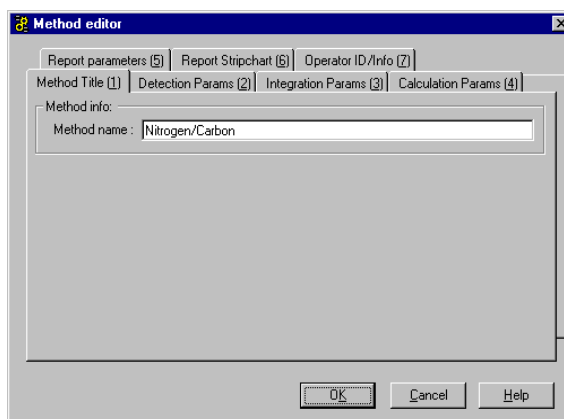
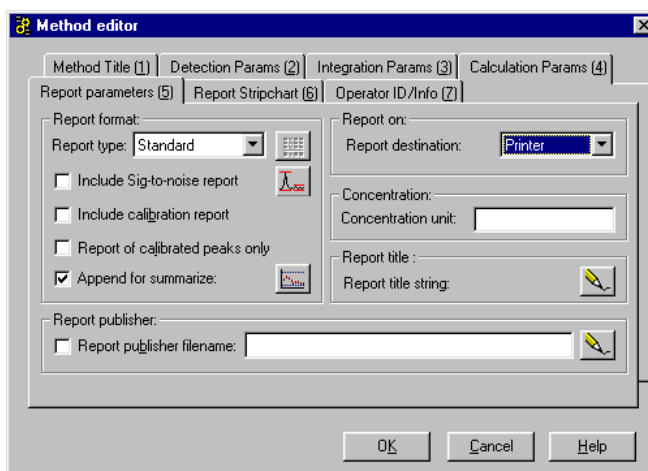
1. In Main Menu select **Edit > Method** or just click the icon . The Method editor window is displayed.

Figure 105. Example of Method editor windows



2. Select **Report Parameters**. The window of Figure 106 will appear.

Figure 106. Report parameter window

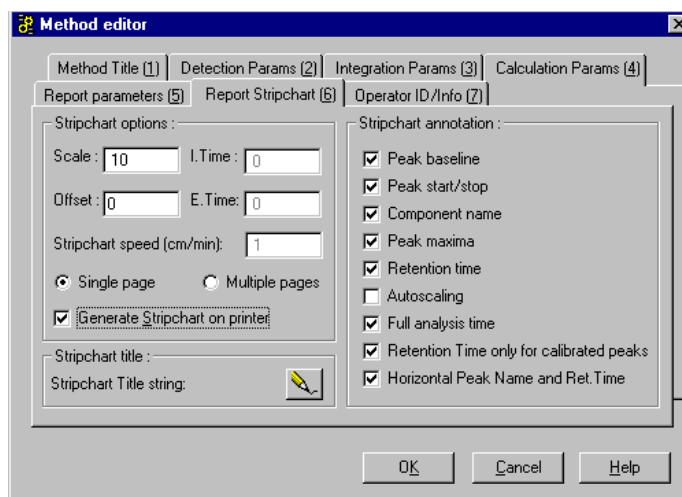


- a. In the section **Report format**, select in the box **Report type** the option **Standard**, then tick the option **Append to summarize**.



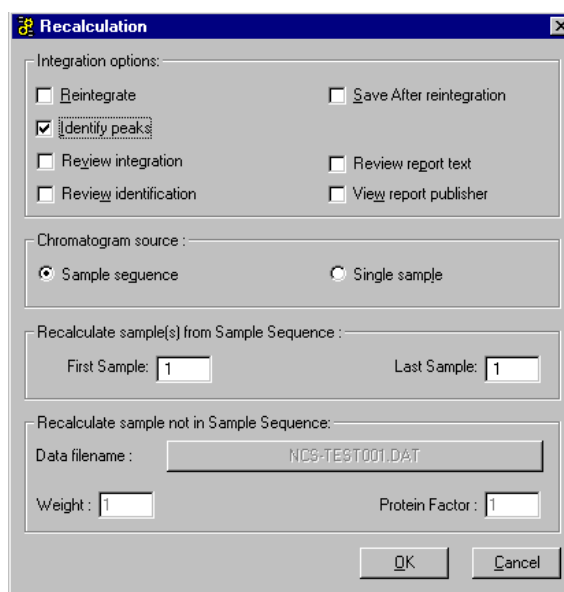
- b. In the section **Report on**, select in the box **Report destination** the option **Printer**.
3. In the window of [Figure 105](#) select the tag **Report Stripchart**. The following dialog window is displayed.

**Figure 107.** Report stripchart window



- a. In the section **Stripchart annotation** uncheck the function **Autoscaling**.
- b. If required, in the section **Stripchart options** modify the values as desired.
4. Click **OK** to confirm the settings.
5. In the main menu select **Recalculation > Recalculation**. The following dialog window is displayed.

**Figure 108.** Recalculation window



- a. In the section **Integration options** tick the function **Identify peaks**.
- b. In the section **Chromatogram source** tick the function **Sample sequence**.

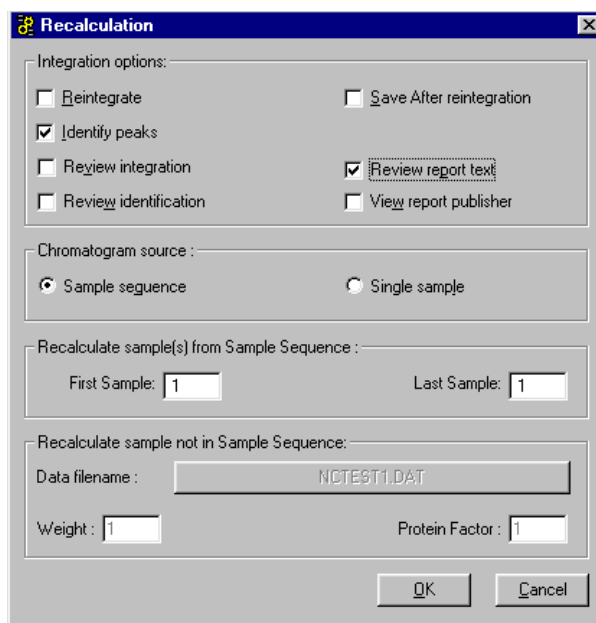
- c. In the section **Recalculate sample(s) from Sample Sequence** set **First Sample 1** and **Last Sample 1**.
6. Click **OK**. The report of values will be printed.

❖ **To Check the Blank Value with No Printer Available**

To check the blank value with no printer available, do the following:

1. In Main Menu select the menu **Recalculation > Recalculation**. The dialog window of [Figure 109](#) is displayed.

**Figure 109.** Recalculation window



- a. In the section **Integration options** tick the function **Review report text**.
  - b. In the section **Integration options** tick the function **Identify peaks**.
  - c. In the section **Chromatogram source** tick the function **Sample sequence**.
  - d. In the section **Recalculate sample(s) from Sample Sequence** set **First Sample 1** and **Last Sample 1**.
2. Click **OK**. The report of values is displayed.

## Evaluation of the Blank Value

The blank value is a function of the type of containers used and of Oxygen purity. Check that the values found are within acceptable limits versus those reported in the final test certificate. If the values found are higher, refer to the following table.

**Table 43.** Blank value diagnostic guide

Blank	Cause and remedy
Nitrogen	<p>If the nitrogen value is definitely higher than that indicated, repeat the blank according to the above described procedures.</p> <p>If the area value decreases, it means that the connection tube between oxygen cylinder and instrument contains air. To solve this problem disconnect the joint for some time and let oxygen flow to the atmosphere.</p> <p>Should the blank repetition not cause a significant decrease in the area value, it means that oxygen used has not a proper purity degree. Use oxygen of required purity.</p>
Carbon	High values are due to contamination. Always work on perfectly clean surfaces and always keep the containers in a closed container.
Hydrogen	The hydrogen blank comes from catalysts and more generally from the circuit. The value tends to decrease in time.
Sulfur	Generally absent or negligible
Oxygen	Generally negligible. High values are due to the container contamination.

**IMPORTANT** All blank values are memorized and subtracted to the sample values. As a consequence, definitely high values may affect analytical precision.

## Sequence of Analyses

**IMPORTANT** Before shipment, every instrument is submitted to an analytical final test procedure according to the concerned instrument configuration. The results of this test are included in the documentation set accompanying the equipment. When the instrument is used for the first time, before analyzing any unknown sample, it is advisable to repeat the test maintaining the selection of standards and their weighing range.

To correctly run the analysis, do what described in the following operating sequence.

### ❖ To Perform the Analytical Sequence

The sample weights may be manually entry or automatically transferred from the balance to the PC. These two modes are explained below.

- “Mode 1: Manual Entry of the Weights” on page 164
- “Mode 2: Automatic Entry of the Weights from the Balance to the PC” on page 165

### Mode 1: Manual Entry of the Weights


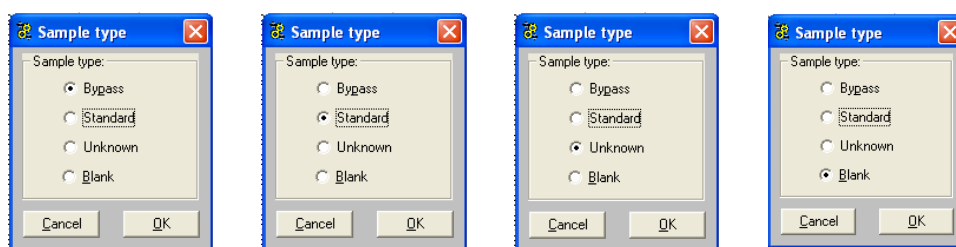
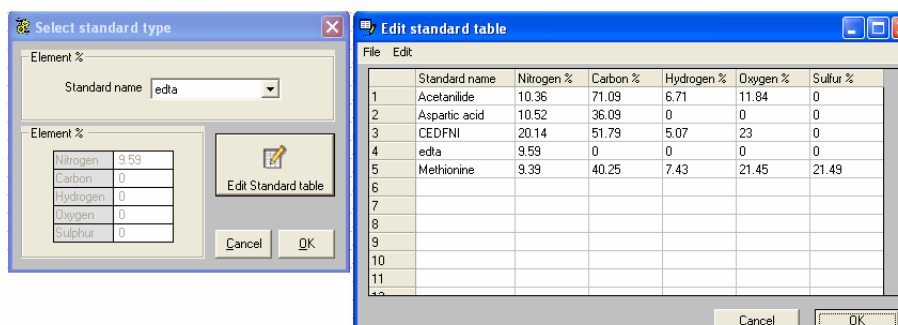
1. Weigh the samples and put them sequentially into the autosampler tray.
2. In Main Menu select **Edit > Sample Table** or just click the icon . The sample table of Figure 99 on page 156 is displayed.
  - a. Enter the **sample name, type** and **weight**. In case of N/Protein and N-Brew analyzers, also specify the sample **category**. See Figure 99 on page 156.
3. The analytical sequence, after the blank analysis, includes an analysis named **Bypass**: a standard substance is analyzed to condition the instrument and at the same time to show the progress of the analysis to the operator, by means of the chromatogram obtained, then three standards and three unknown samples of other composition than the standard substance are analyzed.
  - a. In the sample table grid, click on the column **Type**. Click the arrow shown to display the window for the sample type selection.
  - b. According to the sample type, select **Bypass, Standard, Unknown** or **Blank** as shown in Figure 110.

Figure 110. Selection of the sample type



**Note** Selecting **Standard**, the following window appears. Set here the desired standard.

Figure 111. Selection of the standard




4. Click **OK** to confirm and go back to the sample table.

**Note** New standards may be memorized by clicking **Edit Standard Table**. After the number 5, add the standards of interest and the percentages of the relevant elements. Refer to the following Figure 112.

Figure 112. Edit standard table

	Standard name	Nitrogen %	Carbon %	Hydrogen %	Oxygen %	Sulfur %
1	Acetanilide	10.36	71.09	6.71	11.84	0
2	Aspartic acid	10.52	36.09	0	0	0
3	CEDFNI	20.14	51.79	5.07	23	0
4	EDTA	9.59	0	0	0	0
5	Methionine	9.39	40.25	7.43	21.45	21.49
6						
7						
8						
9						
10						
11						

5. At the end of the sample table editing, select the number following the last edited sample.
6. Select the menu **Edit sample** > **Insert line** or just click the icon .
7. In the main menu, select the menu **Run** > **Start sequence of sample**.
8. Click **Start Now**.
9. At the end of the analytical sequence, the results obtained must be compared with those of the final test, doing what described in the operating sequence “[Comparison Between Analytical Results and Final Test Results](#)” on page 167.

### Mode 2: Automatic Entry of the Weights from the Balance to the PC

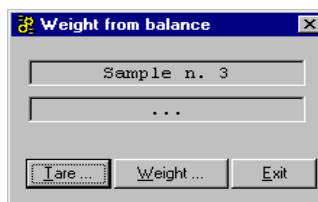
1. Connect the RS 232 connecting cable between balance and PC.
2. Select **Sample Table** > **Balance** > **Balance Setup**. The following window is displayed:

Figure 113. Balance parameters setting

3. Select the computer serial port to which the balance is connected. Pay attention that the serial port must be different respect the serial port selected for the analyzer.
4. Select **Balance** menu.
  - a. Choose the type of balance in use.
  - b. Press **OK**. The dialog window of [Figure 113](#) is displayed again.


- c. Press **OK** to return to Sample Table.
5. In Sample Table select **Balance > Receive weight**. After you have selected the sample number desired (e.g. sample number 3), click on Weight. Pressing the down arrow the following window is visualized:

**Figure 114.** Weight from balance



6. Put the container on the balance plate. Perform the tare pressing **Tare**. Introduce the sample then wait for stabilization.
7. Clicking **Weight**, the value of the weight is automatically transferred. The sample table is ready to acquire the value of the next weight.

**Note** If the balance Mettler Toledo AB54S is used, perform the tare by using the command located on the balance control panel.

- a. At the end of the sample table editing, to automatically stop the sample sequence, select the number following the last edited sample.  
Select the menu **Edit sample > Insert line** or just click the icon .
8. In Main Menu, select the menu **Run > Start sequence of sample**.

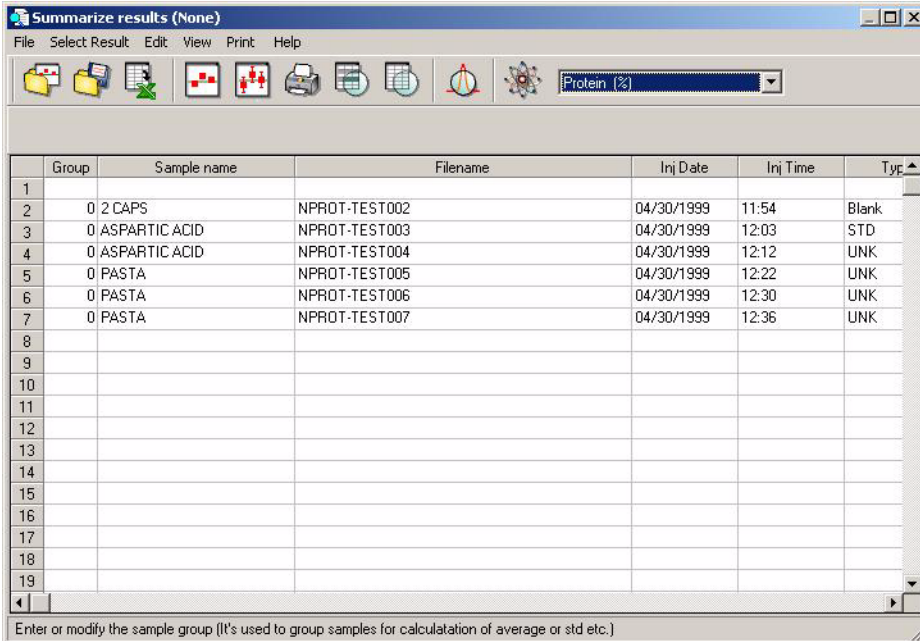
At the end of the analytical sequence, the results obtained must be compared with those of the final test, doing what described in the operating sequence “[Comparison Between Analytical Results and Final Test Results](#)” on [page 167](#).

❖ **Comparison Between Analytical Results and Final Test Results**

To highlight results, do the following.

1. In Main Menu, select **Recalculation > Summarize results** or just click the icon . The following table is displayed.

**Figure 115.** Example of Summarize Results



	Group	Sample name	Filename	Inj Date	Inj Time	Typ
1						
2	0	2 CAPS	NPROT-TEST002	04/30/1999	11:54	Blank
3	0	ASPARTIC ACID	NPROT-TEST003	04/30/1999	12:03	STD
4	0	ASPARTIC ACID	NPROT-TEST004	04/30/1999	12:12	UNK
5	0	PASTA	NPROT-TEST005	04/30/1999	12:22	UNK
6	0	PASTA	NPROT-TEST006	04/30/1999	12:30	UNK
7	0	PASTA	NPROT-TEST007	04/30/1999	12:36	UNK
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						

Enter or modify the sample group (It's used to group samples for calculation of average or std etc.)

2. In the text box **Group**, enter number 1 for samples 5, 6 and 7.
  - a. If you want the data printout, select **Print > Print single group**.
  - b. If you want to read the statistical result, select one by one samples 6, 7 and 8, then select **View > Statistical calculation**. The display will show a window with the statistical data referring to the selected item.

**Interpretation of Results**

If the results obtained are satisfactory, go on with your samples sequence. If on the contrary the results are not correct, try to identify the cause and find the remedy.

**Tip** It is suggested to see [Chapter 7, “Preparing the Sample,”](#)

The cause of the error is generally due to incorrect sample weighing. Always observe the indicated weighing ranges using, if possible, the direct connection between balance and computer, selecting in the Sample Table the option **Receive Weight from Balance** in **Balance** menu.

**Note** Should an electronic balance be connected to the instrument, remember to check the parameters of the connection, selecting in the Sample table the menu **Balance** and then the option **Balance setup**. Refer to [“Mode 2: Automatic Entry of the Weights from the Balance to the PC”](#) on page 165.


3. If the weight of an unknown sample is wrong, the error immediately becomes clear from its percentage result. On the contrary, if the error is due to the weight of one or more standards, the three results of the unknown sample will all be wrong.
4. Another cause of error is the incorrect integration of the peak. If it happens, the correction of the baseline is required proceeding as follows:
  - a. Open the chromatogram selecting the sample to adjust. In Main Menu choose **View > View Chromatogram > Peak**.
  - b. To modify the baseline, choose the option **Move peak start** or **Move peak end** accordingly.
  - c. **Save** the new chromatogram.
  - d. Recalculate the chromatogram following the instruction in sub-paragraph **Sample Recalculation**.

## Quality Control and Check of Analytical Results

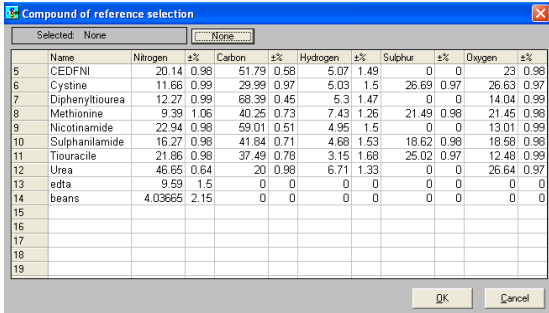
Quality control, particularly for food, often requires the daily analysis of the same materials, specially in nitrogen-protein analyses. Therefore it is important to define the maximum and minimum acceptable values. These values can be stored and used as comparative parameters for next analyses. A similar condition occurs if the user has many samples to run for which he knows the supposed theoretical values. In this case too, we recommend to memorize the maximum and minimum values to make the comparison between the theoretical and found values easier and quicker. To use this comparison method, do what described in the following operating sequence.

### ❖ Method to Compare Results

After having analyzed the standard samples and checked the instrument precision, do the following:

1. In Main Menu, select the menu **Recalculation > Summarize** results or just click the icon . The Summarize result table will be displayed. See [Figure 115](#) on [page 167](#).
2. Select the menu **Edit** and then the option **Select reference compounds**. A table like the following will be displayed.

**Figure 116.** Example of the reference compounds selection table



Name	Nitrogen	Carbon	Hydrogen	Sulphur	Oxygen
5 CEFUNI	20.14	51.79	5.07	0	23
6 Cystine	11.66	29.99	5.03	26.69	26.63
7 Diphenyltiourea	12.27	68.39	5.3	0	14.04
8 Methionine	9.39	40.25	7.43	21.49	21.45
9 Nicotinamide	22.94	59.01	4.95	0	13.01
10 Sulphanilamide	16.27	41.84	4.68	18.62	18.58
11 Thiouracile	21.86	37.49	3.15	25.02	12.48
12 Urea	46.65	20	6.71	0	26.64
13 edia	9.59	0	0	0	0
14 beans	4.03685	2.15	0	0	0
15					
16					
17					
18					
19					

- a. In the textbox **Name**, enter the sample name.



- b. In the text boxes **Nitrogen, Carbon, Hydrogen, Sulphur, Oxygen**, enter the supposed theoretical percentages of the different elements to be analyzed.
3. Click **OK**.  
For each value a minimum-maximum percent deviation (box  $\pm\%$ ) versus the entered value is automatically calculated. The calculation of this deviation is made by an algorithm, which takes into account the entered percentage and the instrumental error.

**Note** If the acceptable error range is wider than that automatically calculated, it can be manually changed

### Comparison between Found Values and Theoretical Values

At the end of the analytical cycle, to compare the values found with the theoretical ones, do the following:

4. Go to the table of [Figure 116](#) doing what described in steps 1 and 2.  
Select the sample to be compared, then click **OK**. The Summarize Results table will be displayed.
5. In Summarize Results, select the sample to be compared as shown in the example of [Figure 117](#).

**Figure 117.** Summarize Results Table: Comparison of samples (1)

Group	Sample name	Filename	Nitrogen
1	0 pasta	ottobre001	2.02
2	0 pasta	ottobre002	2.04
3	0 sugar	ottobre003	0.01
4	0 pasta	ottobre004	2.04
5	0 pasta	ottobre005	2.06
6	0 cap	ottobre006	0.00
7			

Enter or modify the sample group (It's used to group samples for calculation of average or std etc.)

- a. Select **View > Compare to reference compound**. On the left side over the Summarize Results table, as shown in [Figure 118](#), you will read the name of the analyzed sample followed by one or more LEDs according to the number of analyzed elements.

**Figure 118.** Summarize Results Table: Comparison of samples (2)

Group	Sample name	Filename	Nitrogen
1	0 pasta	ottobre001	2.02
2	0 pasta	ottobre002	2.04
3	0 sugar	ottobre003	0.01
4	0 pasta	ottobre004	2.04
5	0 pasta	ottobre005	2.06
6	0 cap	ottobre006	0.00
7			

This box contains the name of the sample

---


<b>Green light</b> =	Indicates that the values found are close to the theoretical values and within the preset error limits.
<b>Red light</b> =	Indicates that the values found are far from the theoretical values.

---

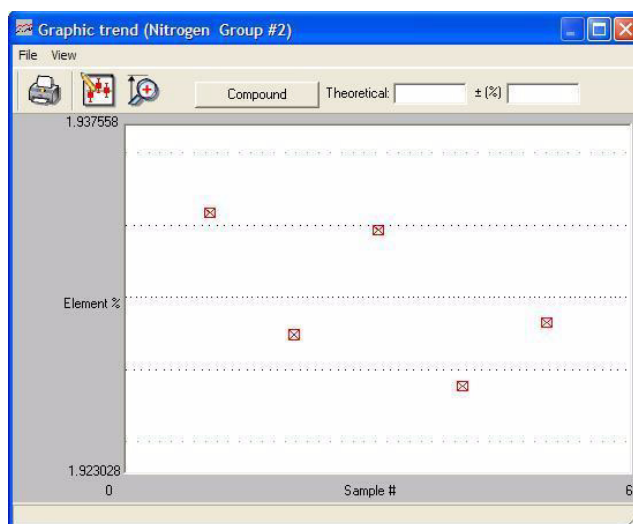
#### ❖ Graphic Display of the Result

If a single element is analyzed, the result can be graphically displayed. This is particularly useful when the sample is routinely analyzed and when it is more important to have the result within the preset error limits rather than the absolute value found.

To display the graph of the result, do the following:

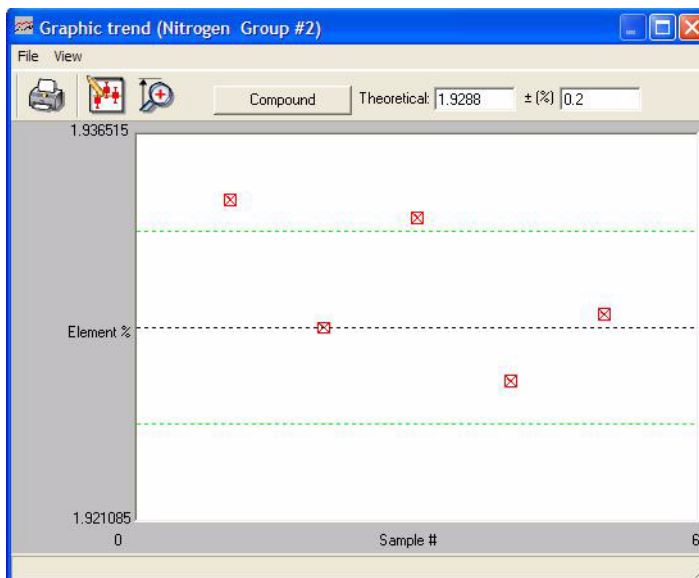
1. In the **Summarize results** table, select the group of samples to be displayed.
2. Click the **Show summary graph of selected group** icon . A window like the following is displayed.

**Figure 119.** Summarize graphic (1)



- a. Click **Compounds**. In the **Compound of reference selection** window that appears, select the sample corresponding to the analyzed one and click **OK**.
- b. A window like the following appears, where a graph is plotted consisting of two parallel green lines, a white middle line and red small squares.

**Figure 120.** Summarize graphic (2)



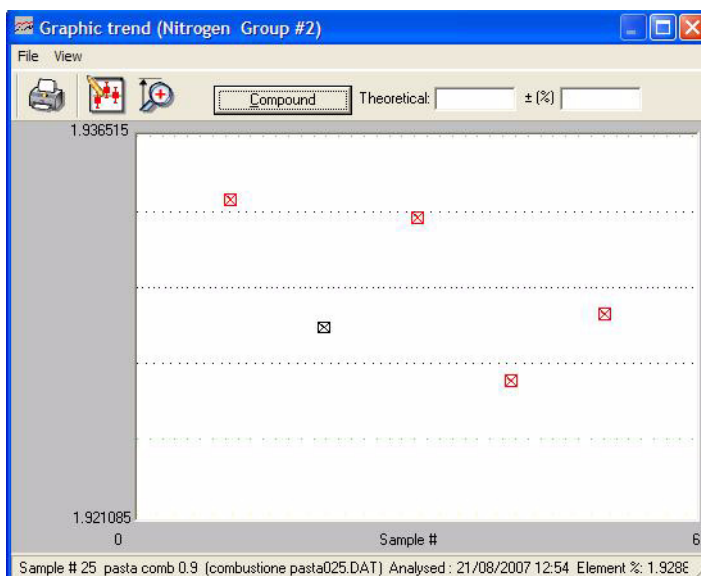
Middle line = Theoretical value

Green lines = Minimum and maximum acceptable values

Red square = Sample analyzed

- c. If the analyzed sample, represented in the graph by a red square, is within the two green lines, it means that, independently of its absolute value, it has a value close to the theoretical one and is within acceptable error limits. The red square, when clicked, becomes white and the percent value is displayed, as shown in Figure 121.

**Figure 121.** Summarize graphic (3)



## Post-Analysis Operations

To obtain precise accurate and constant in time results, and at the same time to reduce operating costs, we recommend you follow some practical suggestions.

### Putting the Instrument in Standby Mode

When the work session is over, the instrument should be put in **Standby Mode**. In this condition, the temperatures of the Left and Right Furnaces are reduced by 50% versus operating temperatures, and the Helium flows on both channels are brought to 20 mL/min.

When the instrument goes in **Standby Mode**, the deactivated adsorption filter is regenerated. After the regeneration the carrier flows through both adsorption filters and the filter purge is stopped.

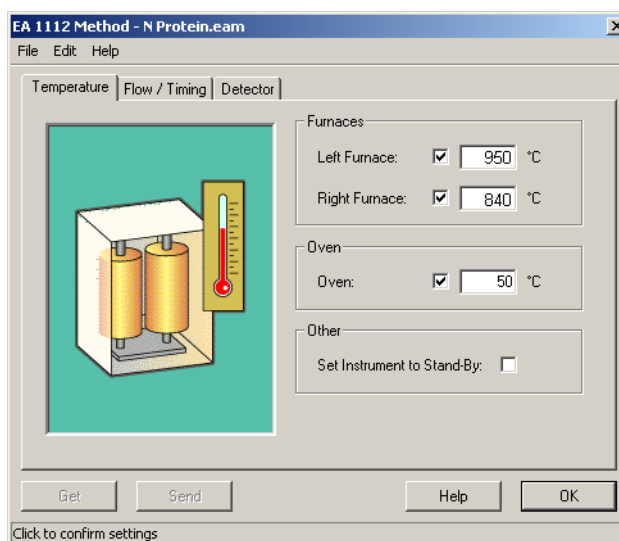
The Standby function can be activated manually or automatically at the end of the analytical sequence.

#### ❖ To Set the Standby Function Manually

Proceed as follows:


1. In Main Menu, select **View > View Elemental Analyzer Status** or just click the icon . The page of [Figure 122](#) is displayed.

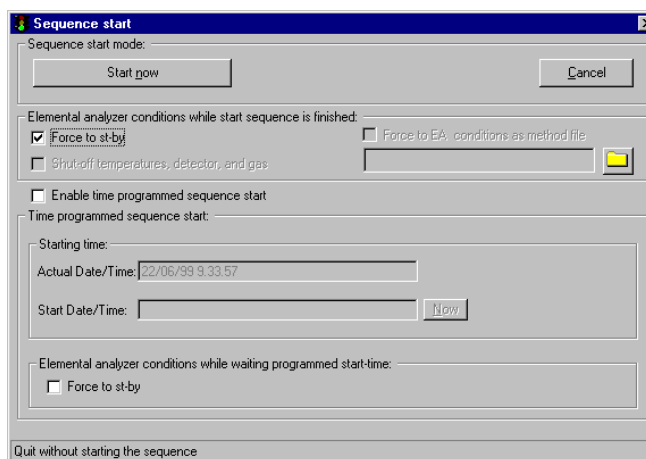
**Figure 122.** Analyzer parameters



2. In the section **Other**, select the function **Set instrument to St-By** by ticking the proper box.
3. Click **Send** to send the command to the instrument and click **OK**.

❖ **Automatic Setting of the St-By Function**

1. In Main Menu, click the icon . The window of [Figure 123](#) is displayed.

**Figure 123.** Sequence start window (1)

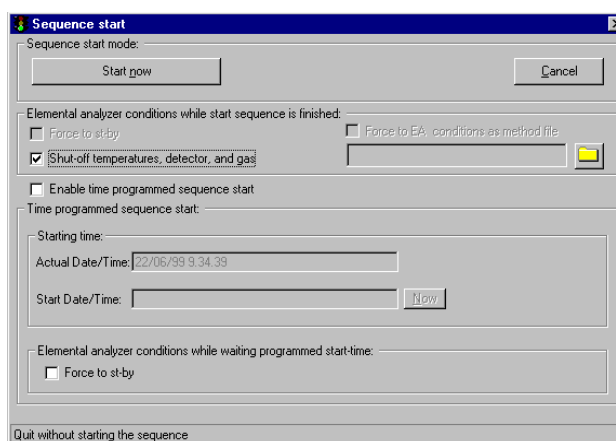
2. In the section **Elemental analyzer conditions while start sequence is finished**, enable the **Force to Stand-by function** by ticking the appropriate check box. The analyzer will automatically go to the *Standby* condition, when the last sample has been analyzed.


## Shutting Off Furnaces, Detector and Cutting Off Gas Flows

The shut off of furnaces and detector and the cut off of gas flows can be programmed as described in the following operating sequence.

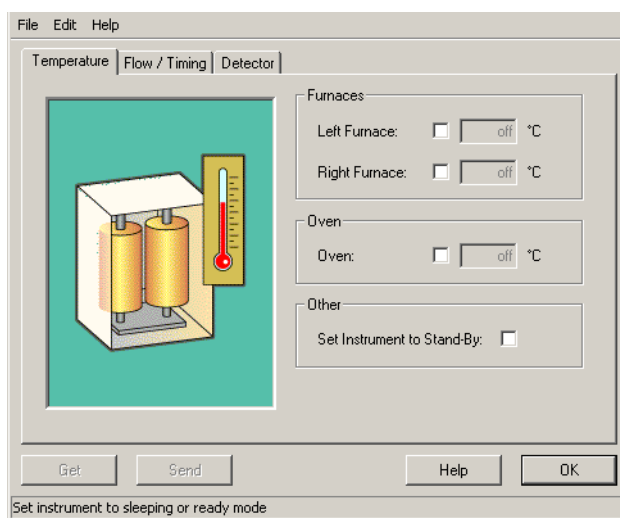
❖ **To Shut Off Temperature, Detector and Gas**

1. In Main Menu, just click the icon . The window of [Figure 124](#) is displayed.

**Figure 124.** Sequence start window (2)

2. In the section **Elemental analyzer conditions while start sequence is finished**, enable the function **Shut-Off temperature, detector and gas** by ticking the appropriate check box.
3. When the oxidation reactor requires cleaning, the furnace should be switched off and then the Helium flow reduced using the **Stand-by** function.
  - a. In Main Menu, select **Edit > Edit Elemental Analyzer parameters** or just click the icon . The window of [Figure 125](#) on is displayed.

**Figure 125.** Edit elemental analyzer parameters



- b. In the section **Furnaces**, tick the box **Left Furnace** to enable the **Off** condition.
- c. In the section **Other**, enable the function **Set instrument to Stand-by** by ticking the appropriate check box.
- d. Click **Send** to send the command to the instrument, then click **OK**.

## Wake-up and Auto-Start Functions

These are timed functions, which can be programmed to minimize dead times.



### ❖ To Set Wake-up Function

To pass from **Stand-by** to **Ready** status, operate as follows:


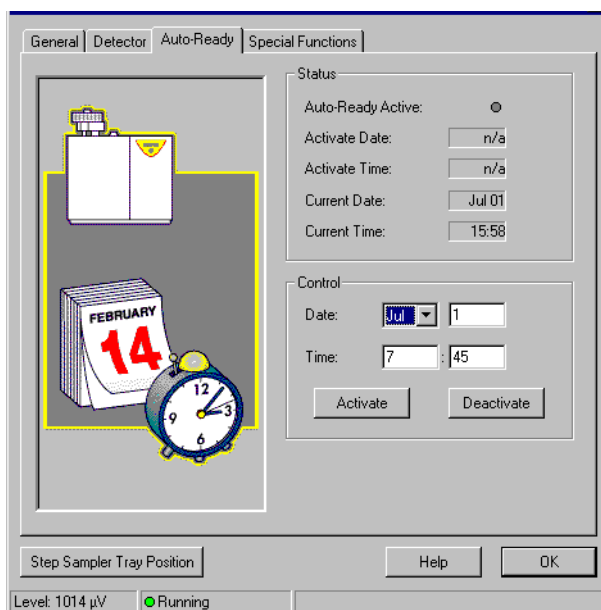
1. In Main Menu, select **View > View Elemental Analyzer status** or just click the icon .
2. Select the **Auto-ready** menu. The window of [Figure 126](#) is displayed.

Figure 126. Status of the analyzer



- In the section **Control**, set the desired date and time of activation of the **Wake-up** function.
- Click **Activate** and then **OK** to confirm.

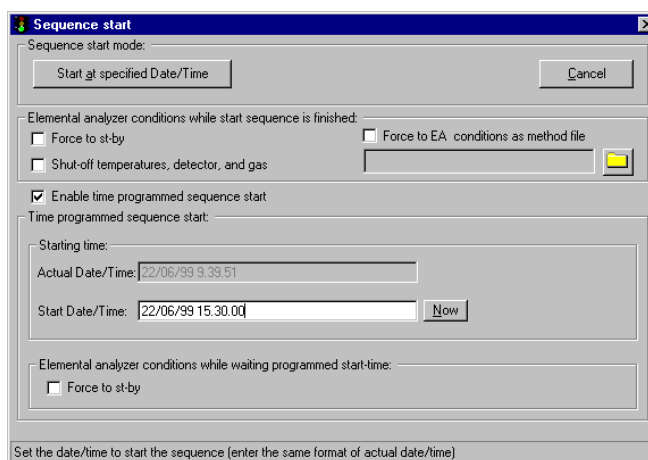
## Auto-Start Function

If you desire Auto-Ready to be followed by Auto-Start, programming an analytical sequence, do the following:

### ❖ To Set Auto-Start Functions

- In Main Menu, click the icon . The following window is displayed.

Figure 127. Start sequence



2. Enable the function **Enable time programmed sequence start** by ticking the appropriate check box.
  - a. In the section **Starting time** click **Now**.
  - b. In the **Start Date/Time** text box enter the date and time of the function activation.

**IMPORTANT** The function activation should be programmed with a delay of at least 60 minutes versus the time programmed for the Auto-Ready Function to allow the analyzer to reach a good thermal equilibrium.

## Analytical Troubleshooting

If the instrument has been correctly installed, the gas characteristics are as required and maintenance has been regularly carried out, Flash 2000 will provide correct results. The lack of the above conditions will be indicated by anomalies in the chromatograms and the relevant analytical reports. The following table reports the most common anomalies with the relevant diagnosis and remedy.

**Table 44.** Analytical troubleshooting guide

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 any 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 44.** Analytical troubleshooting guide, continued

<b>Problem</b>	<b>Diagnosis</b>	<b>Remedy</b>
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.
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 ashes 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



# Applications

This chapter contains guidelines referring to the applications of the Flash 2000 elemental analyzer.

## Contents

- [Introduction](#)
- [Sample Oxidation](#)
- [Automatic Oxygen Dosage](#)

## Introduction

Elemental analysis has many fields of application. Among the most important are:

- *Pharmaceuticals*, with synthesis products.
- *Petrochemical Industry*, with oil and its derivatives,
- *Industrial chemistry*, with polymers.
- *Environment*, with the analyses of soils, sediments, waters.
- *Food*, with protein analysis, etc.

The Flash 2000 analyzer, thanks to the Eager Xperience software, can analyze different types of samples. You only have to follow the indications concerning the sample weighing to obtain precise and reproducible results, not only with standard substances, but also with all other substances you will analyze later.

However, sometimes samples to be analyzed may not be homogeneous and others may be of difficult combustion. In any case, as a general rule, weigh a quantity of substance adequate to the sample nature and to the type of determination.

## Sample Oxidation

To obtain precise and reproducible results the sample must be completely oxidized.

This is simple enough when the sample weighing range is narrow, but when the weight is doubled or tripled, oxygen requirements must strictly follow the weight increase.

This is obtained by changing the rate and time of oxygen introduction. On this matter please refer to the instructions given in the operating sequence [To Change the Oxygen Quantity](#).

If the major objective of the analysis is to obtain the best precision of results, it is particularly important that catalysts, and specially **copper**, last as long as possible. Therefore you have to establish how much Oxygen is required to burn a sample of that particular nature as a function of its weight.

For example, graphite requires more Oxygen than a soil or an organic substance, though at equal weight. If we always use for all samples the maximum Oxygen quantity, we will obtain excellent results, but copper will only last for few analyses.

To establish the Oxygen quantity required to combustion, do what described in the operating sequence [“To Establish the Required Oxygen Quantity”](#) on page 181.

For N/Protein and N-Brew analyzers we recommend to always use the **OxyTune**<sup>®</sup> function (*Automatic Oxygen Dosing System*). Refer to paragraph [“Automatic Oxygen Dosage”](#) on page 182.

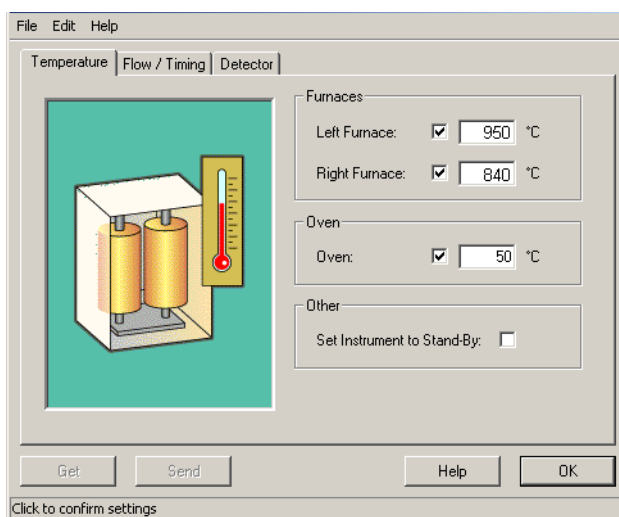
#### ❖ To Change the Oxygen Quantity

1. In Main Menu select **Edit > Edit Elemental Analyzer Parameters** or just press the icon



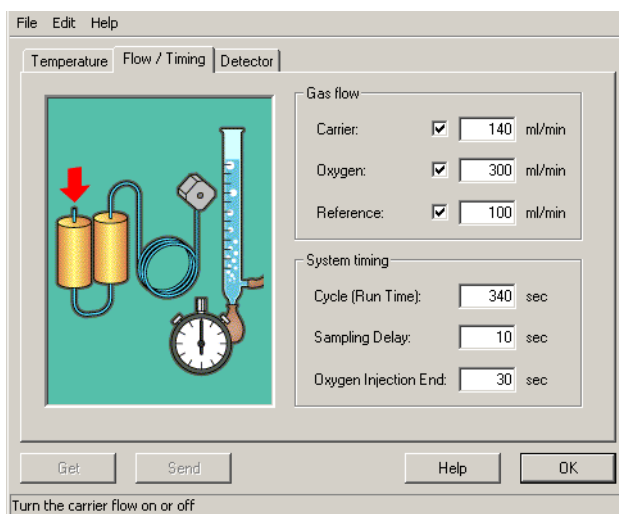
. The following window is displayed with the analyzer operating parameters.

**Figure 128.** Example of analyzer parameters



2. Select Flow/Timing tag. The following tag is displayed.

Figure 129. FlowTiming tag



- To change the time of oxygen introduction, enter the desired value in the appropriate box **Oxygen Injection End** in the section **System timing**.
- To change the oxygen flow rate, enter the flow value in the appropriate box **Oxygen** in the section **Gas flow**.

#### ❖ To Establish the Required Oxygen Quantity

Do the following:

- Analyze the sample setting the Oxygen flow to its maximum value (*300 mL/min*).
- At the end of the analysis, run a blank. If the area value found is equal or very close to the traditional blank value ( $\pm 50\%$ ), it means that the sample is completely burnt without leaving any *memory effects*.
- To know how much Oxygen was given in excess, repeat the sample run reducing the Oxygen flow until the blank value is definitely higher than the traditional one. So you will establish the Oxygen quantity required for the combustion of that kind of sample with that particular weight.

**Note** The same result can be obtained by presetting a value of oxygen flow and varying the time of oxygen injection Oxy Inj End.

**IMPORTANT** The calibration and analysis of samples must be performed under the same conditions of Oxy Flow and Oxy Inj End.

This procedure is useful when you have to analyze a number of samples of the same nature. In the analytical sequence, you will try to keep the same weighing range for all samples.

## Choosing the Weighing Range

This choice is a function of the sample kind, but also of the type of determination.

- **Simultaneous Analysis of 3 or 4 elements (CHN, CHNS, NCS)**  
The weighing range is generally between 1 and 3 mg for organic substances, and up to maximum 20 mg for inorganic samples (e.g. soils, sediments, rocks).
- **Nitrogen-Carbon Analysis**  
The weighing range is generally between 1 and 3 mg for organic substances, and up to maximum 100 mg for soils and sediments.
- **Single Runs of Sulfur and Oxygen**  
The weighing range is generally between 1 and 3 mg for organic substances, and up to maximum 20 mg for inorganic samples.
- **Nitrogen analyses in samples of any nature except food samples**  
We recommend a weighing range between 1 and 20 mg independently of the sample nature.
- **Nitrogen analyses in geological materials (soils, sediments, etc.)**  
We recommend a weighing range between 100 mg and 1g according to the sample nature. Refer to “[Method for Oxygen Dosage \(OxyTune®\)](#)” on [page 182](#) on.
- **Nitrogen analyses in food and agricultural samples**  
We recommend a weighing range between 100 and 500 mg according to the sample nature. Refer to “[Method for Oxygen Dosage \(OxyTune®\)](#)” on [page 182](#).

## Automatic Oxygen Dosage

In nitrogen-protein analyses you may often need to analyze samples of very different composition both as percentage and nature.

There are samples that for their nature require a minimum Oxygen quantity versus other samples that require greater Oxygen amounts. To avoid that the user be obliged every time to modify the weighing range or adjust the Oxygen quantity, the software provides a table comprising various categories where samples of different nature are memorized or can be memorized. Refer to paragraph “[Sample Table](#)” on [page 156](#).

Selecting, after the weight entry, the category to which the sample belongs, the system will deliver the right Oxygen quantity required for a complete combustion. This condition is obtained by a feature provided thanks to the method below:

### Method for Oxygen Dosage (OxyTune®)

1. A fixed Oxygen flow of 300 mL/min was set.
2. The blank value (container + 50-80 mg of sugar) was checked by repeatedly injecting Oxygen (using Oxygen of 99.995% purity grade) for 30 seconds in a resulting theoretical quantity of 150 mL.
3. Standardization was performed using Aspartic Acid with weight ranging from 50 to 100 mg and injecting fixed Oxygen amounts for 30 seconds in a resulting theoretical quantity of 150 mL.
4. Samples having different nature were analyzed as follows:

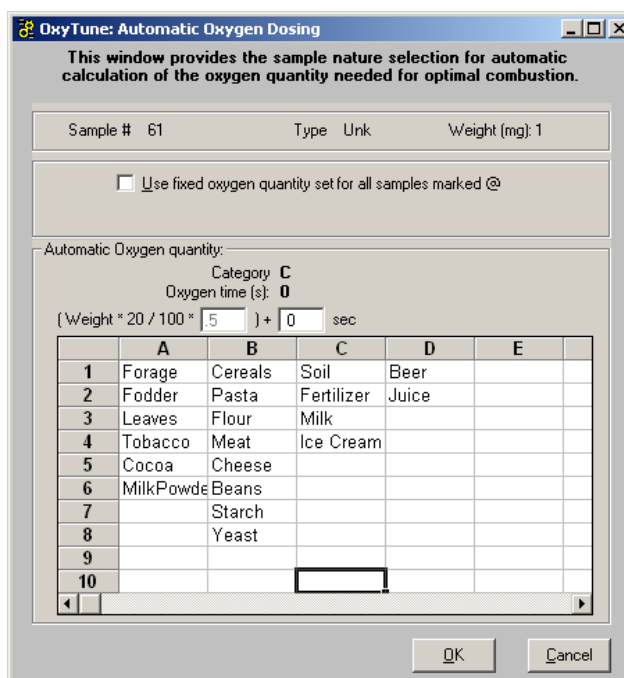
- 200 mg of sample weighed and Oxygen was injected for 60 seconds in a resulting theoretical quantity of 300 mL.
- At the end of the analysis, after taking note of the value, the blank was run again.
- The same procedure (sample analysis and subsequent blank analysis) was followed reducing the injection time at every sequence run. Checking the increased blank value, the combustion critical point was established.
- After having established the combustion critical point, the ideal Oxygen/sample weight factor (obtained dividing the Oxygen quantity injected by the sample weight) was found and stored in an appropriate category.
- The sample analysis was repeated starting by reducing the weight to 100 mg, and then progressively increasing the sample weight until reaching the maximum quantity accepted by the container. Being ideal factor multiplying value memorized, the Oxygen amount for each single analysis was automatically calculated simply selecting the category the sample belongs to.

Based on these results, the following table was created:

### Table of Sample Category

This table, shown in [Figure 130](#) is displayed following the instructions of the operating sequence “To Fill Sample Table” on [page 156](#).

**Figure 130.** Table of sample category



The table contains:

- Four categories of samples - A, B, C, D. where the ideal factor values (Oxygen/sample weight-sample nature) of the samples have been prefixed.

- The nature of samples.
- Four free categories - E, F, G, H - where sample names not considered (extraneous) in categories - A, B, C, D - can be memorized.  
When a free category is selected, the ideal factor value, calculated by using the Oxygen Dosage method OxyTune<sup>®</sup>, must be entered in the appropriate box.  
Before memorizing the sample name in the new category and the ideal factor value, the procedure previously described must be repeated.

**Note** The correct selection of the group to which the sample belongs helps you keep the analyzer perfectly efficient.

**CAUTION** Non compliance with allocation criteria may adversely affect analytical results.

The sample category - Blank, Bypass and Standard - is the same and is marked by the symbol @.

When @ is selected, a set time is entered and consequently an equal Oxygen quantity independently of the sample quality and nature.

This time has been preset at 30 seconds, but it can be changed according to particular analytical requirements.

**CAUTION** In analyses of Nitrogen traces, e.g. in starches analysis where Nitrogen content is below 0.1%, it is advisable to evaluate the blank value injecting the same Oxygen quantity as that used for the sample.

The @ category can also be used for Unknown samples, when sample quantities below 100 mg are weighed. Independently of the nature of the weighed samples, enter:

- 10 seconds of Oxygen for samples between 0 and 20 mg.
- 20 seconds of Oxygen for samples between 20 and 50 mg.
- 30 seconds of Oxygen for samples between 50 and 100 mg.



## Use of Eager Simplified User Interface

This chapter provides information about the use of the simplified user interface of *Eager Xperience*.

### Contents

- [Simplified User Interface](#)

The possibility of using a simplified version of *Eager Xperience* is mainly addressed to users who do not possess a specific expertise in the instrumental field. This option however also concerns those who, having to run every day analyses of samples of the same nature, do not need any special functions of *Eager Xperience*. For this purpose the software is provided with a “password” system, which makes the application of its functions more or less extended. Normally the password is dedicated to the laboratory head, which allows the use of the entire software, besides one or more passwords dedicated to the operator or operators of the instrument. In this way the laboratory head has the possibility to prevent the operator from using the appropriate software functions he deems and to act in any moment in case of need. To use the simplified version of *Eager Xperience*, do what described in the following operating sequence.

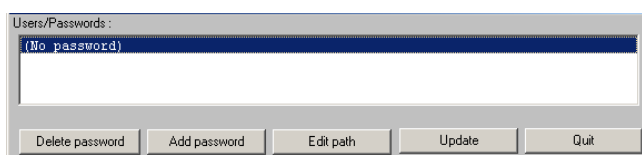
**ATTENTION** The use of the simplified user interface is NOT available with Eager Xperience USB and A/D versions.

## Simplified User Interface

### ❖ To use the Simplified User Interface

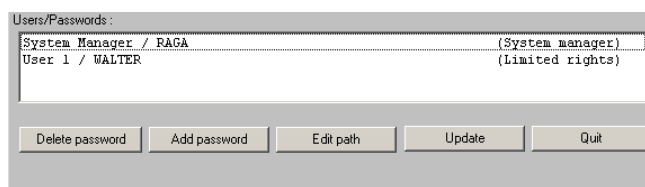
1. In Main Menu, select **File > System administration**. Reply by clicking **Ok** to the message displayed. The window of [Figure 131](#) is visualized, where passwords must be entered.

**Figure 131.** User password window (1)



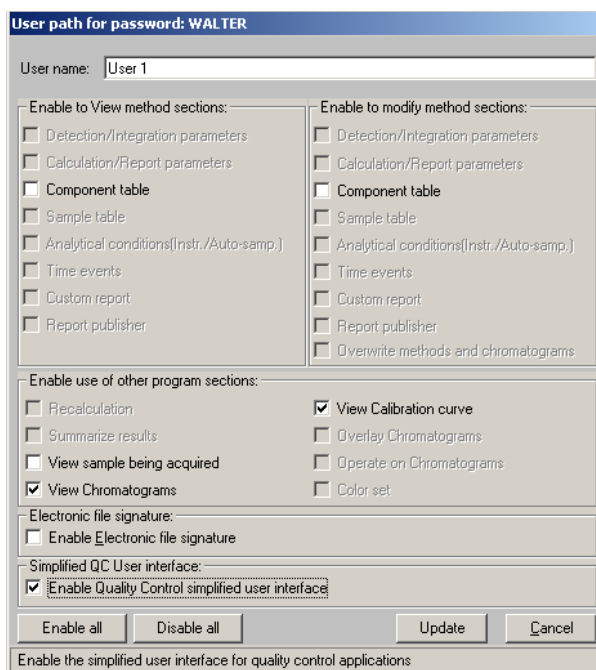
- The first password, dedicated to the laboratory head (*System Manager*), becomes active by clicking **Add Password** and entering the name in the text-box. Press **Ok** to confirm. See the top section of [Figure 132](#).
- The second password, dedicated to the operator (*Limited rights*), becomes active using the same procedure. See the bottom section of [Figure 132](#).

**Figure 132.** User password window (2)

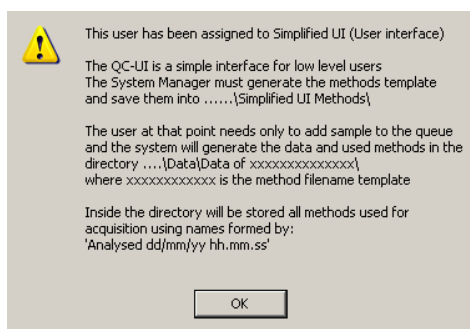


2. To exclude the functions that have not to be available to the operator, click **Edit Path**. The window of [Figure 133](#) is displayed.

**Figure 133.** User path window



3. Select the option **Enable Simplified user interface** by ticking the appropriate box.
4. Select the functions that have to be available by ticking the relevant boxes.
5. Click **Update**. In the page displayed, it is requested to generate a directory for this application.



6. Click **OK**. In the window of Figure 132 click **Update** again. A message concerning the password is displayed.

❖ **to Start The Simplified User Interface**

1. When the operator (*Limited Rights*) opens the software, a window like that shown in Figure 134 will be displayed.

**Note** The number of icons in the window corresponds to the previously selected functions.

**Figure 134.** Sample table of the simplified user interface (1)

	Status	Sample name	Chromatogram filename	Data system method	Type of sample
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					


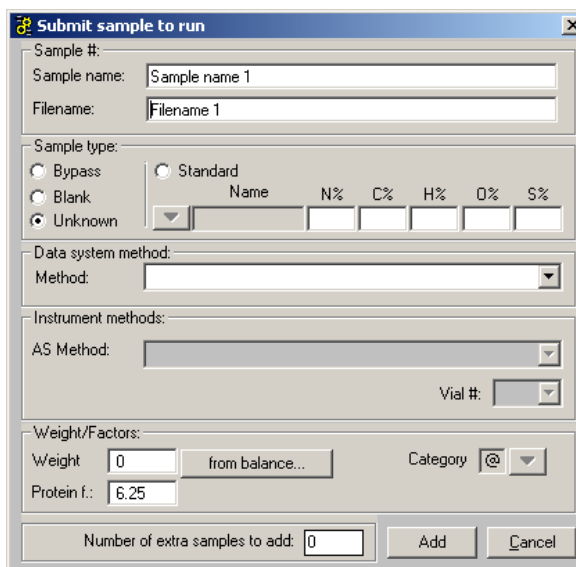
2. In the window of Figure 135 select the  *Submit new sample to analyze* icon to edit the sample table. A window like the one below is displayed.


Figure 135. Window of submit sample to run



3. Enter the **Sample name** and the **chr filename**, followed by a number. Then the file names and the sample names will continue to be progressively numbered.
4. Select the type of the sample to be analyzed: **Sample type**.
5. In the section **Instrument** select, in **Data Method**, one of the methods previously saved in the folder **Simplified UI Method**.
6. Enter the sample **Weight** and, in case of N/Protein and N-Brew configurations, the multiplying factor **Protein f**: and the sample **Category**. Click **Add**.
7. Edit the table for all samples to be analyzed. At the end of editing, the sample table will appear filled with all information previously entered, as shown in the example of [Figure 136](#). The boxes of the **Status** column show a **Q** (Queue).

Figure 136. Sample table of the simplified user interface (2)

	Status	Sample name	Chromatogram filename	Data system method	Type of sample	Standard r
1	Q	Sample name 3	Filename 3		Unknown	
2	Q	Sample name 4	Filename 4		Unknown	
3						
4						
5						

8. To send the start to the instrument select the  icon and reply **Yes** to the question **Are you sure to run queued samples?**. At the end of the analytical sequence the instrument automatically stops.
9. To view the results, choose **View > View only result of sheet**.

**Note** If at the end of each analysis you want to read directly the result, in the menu *View* enable the function *Last sample calculated results*. If for any reason you need to stop the analytical cycle, click the icon named *Put sample on queue (no run)*.

---

Part

1

# **Maintenance and Troubleshooting**

---

## Maintenance

This chapter provides information on the current and periodic maintenance of the instrument, and it also contains the operating sequences for installation and maintenance of the MAS 200R Autosampler and the CM2 Manual Sampler.

### Contents

- [Instrument Maintenance](#)
- [Installing and Servicing the MAS 200R Autosampler](#)
- [Installing and Servicing the CM2 Manual Sampler](#)



**WARNING** If, for technical reasons, it is necessary to work on parts of the machine that may involve hazardous operations (moving parts, components under voltage, etc.). Thermo Fisher Scientific authorized Technical Service has to be called. This situation can be identified because the access to these moving parts is possible only using a tool and because the concerned removable protective covers bear a warning symbol that draws the operator's attention to the specific warnings included in the documentation accompanying the instrument. In case the work has to be carried out by the operator, the latter must prove to be adequately trained to perform the specific maintenance operation.



**WARNING** When the instrument is switched off, consider that it does not cool down immediately, but heat tends to concentrate in the upper part of the furnaces area. The openings provided for the chamber aeration will cause a slow cooling of 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.

## Instrument Maintenance

**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 the crucible done with same material.

## Periodic Maintenance

- Replacement of the gas chromatographic column. The column lifetime in Flash 2000 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. Follow the instructions given in paragraph “[Shutting Off Furnaces, Detector and Cutting Off Gas Flows](#)” on [page 173](#).

## 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 paragraph “[Current Maintenance Program](#)” on [page 152](#).

Replace and install reactors and filters according to the operating sequences described in [Chapter 6, “Connecting Reactors and Adsorption Filters,”](#)

- “[To Remove the Quartz Reactors from the Furnaces](#)” on [page 93](#)
- “[To Remove Special Steel Reactors from the Furnaces](#)” on [page 94](#)
- “[To Remove the Adsorption Filter](#)” on [page 95](#)
- “[To Install the Quartz Reactors into the Furnaces](#)” on [page 84](#)
- “[To Install the Special Steel Reactors into the Furnaces](#)” on [page 87](#)

## Replacement of the Filling Materials

The replacement of reactors and adsorption filters requires the replacement of their filling materials. This operation comprises two steps:

- 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**

**Material required**

Tool for cleaning quartz reactors P/N 276 06010 (included in the Standard Outfit of all Flash Configurations)

Filling materials



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

Remove the quartz reactor from the furnace following the instructions given in the operating sequence “[To Remove the Quartz Reactors from the Furnaces](#)” on [page 93](#), then do the following:

1. Introduce the cleaning tool into the reactor as shown in [Figure 137](#).

**Figure 137.** 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 138](#).
4. Repeat steps 1 and 2 until complete elimination of the exhausted filling materials.

**Figure 138.** 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 do this, refer to paragraphs [Chapter 5, “Preparation of Reactors and Adsorption Filters,”](#) according to the analyzer configuration.



### ❖ To Replace the Filling Material in Special Steel Reactors

#### Material required

Tool for cleaning special steel reactors P/N 276 06025 (included in the Standard Outfit of N/Protein, N-Brew and NC-Soils, NC-Sediments and NC-Filters Configurations)

Filling materials



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

Remove the special steel reactor from the furnace following the instructions given in the operating sequence “[To Remove Special Steel Reactors from the Furnaces](#)” on [page 94](#), then do the following:

1. Introduce the cleaning tool into the reactor as shown in [Figure 139](#).

**Figure 139.** Removal of the Filling Material from a Special Steel Reactor



2. Rotate the tool exerting a slight pressure to scrape off the filling material and collect the removed filling material as shown in [Figure 140](#).
3. Repeat steps 1 and 2 until complete elimination of the exhausted filling materials.

**Figure 140.** Collection of the Filling Material Removed from a Special Steel Reactor



4. At the end of the operation restore the layers of filling materials introducing into the reactor the new ones. To do this, refer to paragraphs “[Introduction to the Preparation of Reactors and Filters](#)” on [page 57](#) and “[Preparing the Reactors](#)” on [page 74](#), according to the analyzer configuration.

### ❖ To Clean the Crucible

#### Materials Required

Tool for cleaning quartz reactors P/N 276 06010

Quartz wool

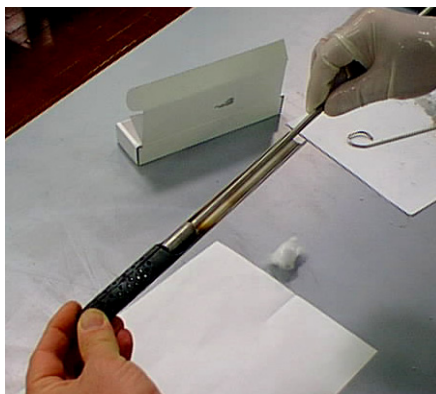
Remove the crucible following the instructions given in the operating sequence “To Prepare the Crucible” on page 78, then do the following:



**CAUTION** Perform the operation with the furnaces at room temperature.

1. Introduce the cleaning tool into the crucible as shown in Figure 141.

**Figure 141.** Removal of Quartz Wool and Ashes from the Crucible



2. Rotate the cleaning tool exerting a slight pressure in a way to scrape off the filling material and collect the material removed.
3. At the end of the operation, introduce new quartz wool into the crucible as described on page 78.

### ❖ To Replace the Filling Material in Adsorption Filters

#### Material Required

Filling materials

Remove the adsorption filter from the detector compartment according to the instructions given in the operating sequence “To Remove the Adsorption Filter” on page 95, then do the following:

1. Unscrew the filter nut and remove the filling material.

**Note** Some adsorption filters contain materials that can be regenerated, such as for instance molecular sieves. Therefore it is recommended to properly collect the material removed from the filter.

2. Restore the sequence of the layers of filling materials introducing the new ones into the filter. To do this, refer to paragraphs “[Introduction to the Preparation of Reactors and Filters](#)” on [page 57](#) and “[Preparing the Adsorption Filters](#)” on [page 79](#), according to the analyzer configuration.

## Replacement of the Gas Chromatographic Column

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

### ❖ To Replace the Gas Chromatographic Column

#### Material Required

Open end wrenches for the column fittings

The gas chromatographic column can be installed outside or housed in the detector compartment.

1. Open the instrument right door. Also refer to paragraph “[Detector Compartment](#)” on [page 30](#).

**Figure 142.** View of the Detector Compartment



2. 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.

3. Undo the 4 fixing screws securing the protection panel.

**Figure 143.** Access to the detector

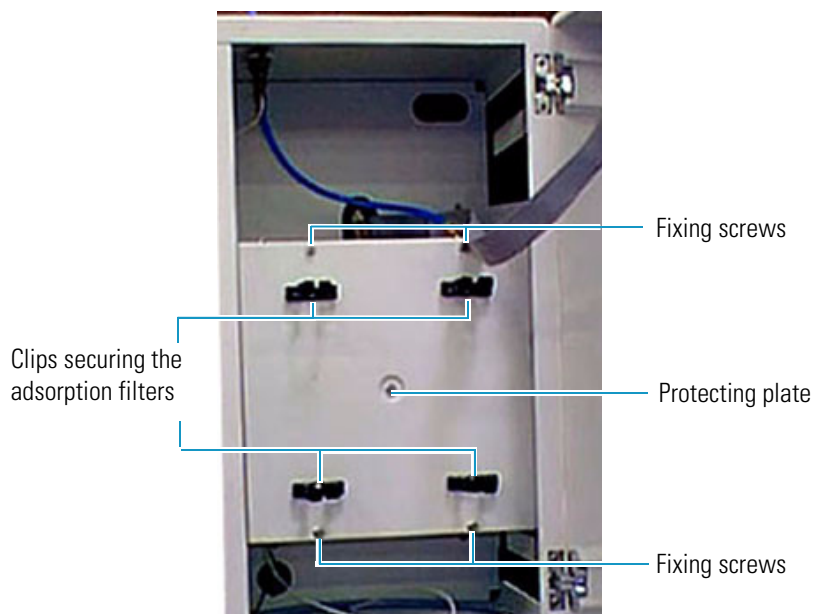
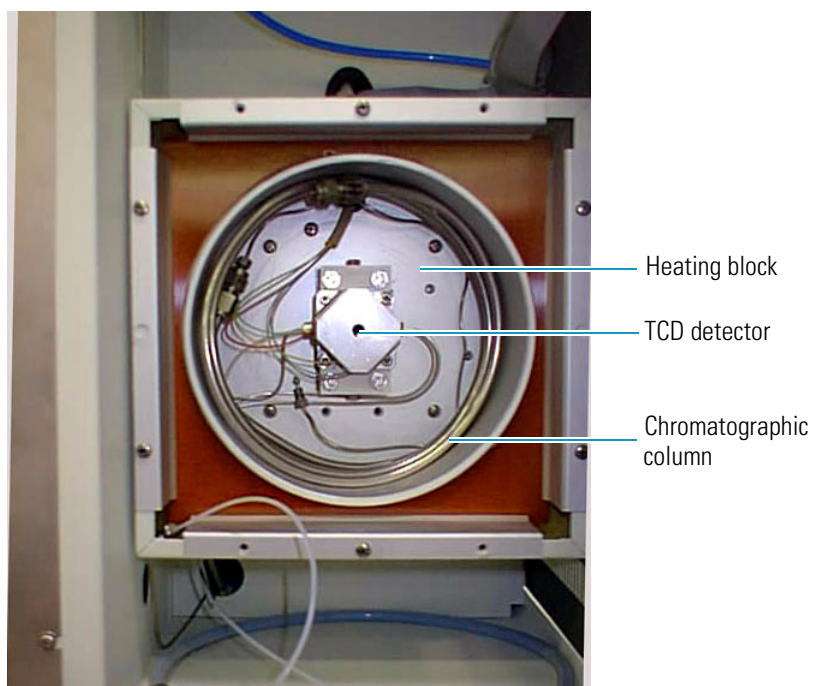


Figure 144 shows the detector compartment, the heating block where the detector is housed, and the gas chromatographic column.

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

**Figure 144.** Compartment housing the chromatographic column



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

## Replacement of the O-Rings of the Reactors Coupling Unions

To perform this operation, operate as described in the following operating sequence.

### ❖ To Replace the O-Rings of the Reactors Coupling Unions

#### Materials Required

Allen wrench

Screwdriver

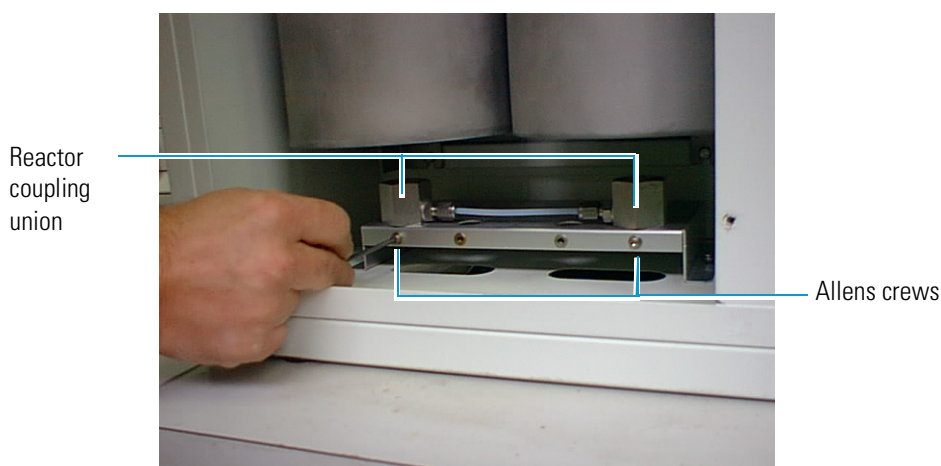
Spare seals



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

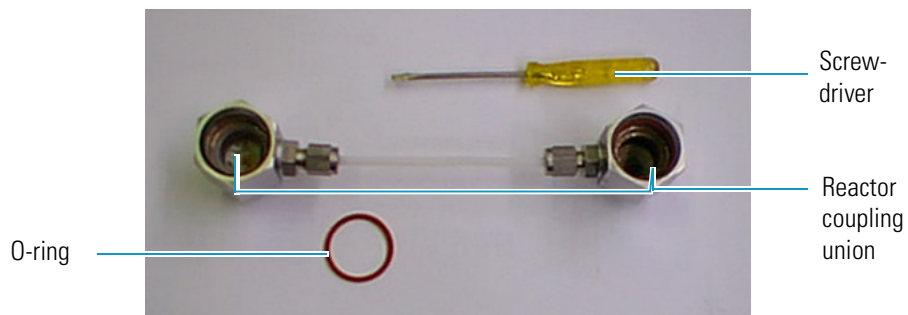
1. Open the furnaces compartment by lifting the cover and removing the protection wall. Also refer to paragraph “Furnaces Compartment” on page 28.
2. Remove the reactors from the furnaces according to the instructions given in the operating sequence “To Remove the Quartz Reactors from the Furnaces” on page 93 or “To Remove Special Steel Reactors from the Furnaces” on page 94.
3. Loosen the allen screws securing the reactors coupling unions to the base as shown in Figure 145.

**Figure 145.** Removal of the Reactors Coupling Unions



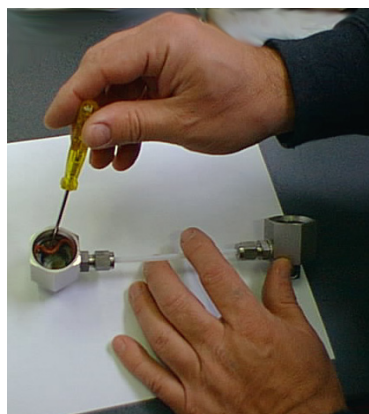
4. Remove the reactors coupling unions from the base and rest them on a clean surface, as shown in Figure 146.

**Figure 146.** View of the reactors coupling unions



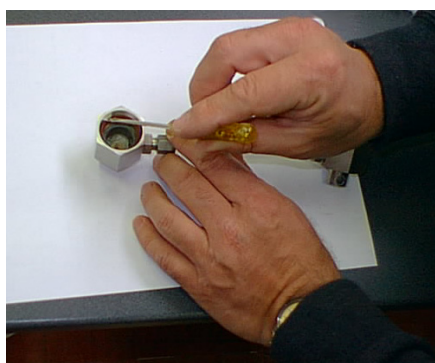
5. Using the small screwdriver, remove the O-ring from each union, as shown in [Figure 147](#).

**Figure 147.** Removal of the O-ring from the coupling union



6. Put a new O-ring into each union making sure, by using an appropriate tool, it correctly fits its seat, as shown in [Figure 148](#).

**Figure 148.** Introduction of the O-ring from the coupling union



# Installing and Servicing the MAS 200R Autosampler

## Introduction

The MAS 200R autosampler for solids consists of an anodized aluminium block having on its left side the inlets for carrier and purge gas lines. Its modular structure allows to run up to 125 unattended analyses.

The base unit is provided with one 32-position sample tray, but it can accommodate three other 32-position trays 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.

## Installation

The autosampler is directly installed on the connecting nut of the concerned reactor.

### ❖ To Install the MAS 200R Autosampler on the Flash 2000

The following operating sequence provides instructions to install the autosampler on the Flash 2000.

**Note** The installation sequence is common to all instrument configurations.

#### Material Required

8 mm wrench

Proceed as follows:

1. Connect the tubes coming from the gases connections **1** and **2** located on the analyzer, to the relevant connections **1** and **2** of the autosampler.
2. Manually screw the autosampler nut on the concerned channel.

**Note** The MAS 200 R Standard Outfit includes two reactor connectors: 18 mm OD for Quartz reactor and 25 mm OD for HPAR. Screw the suitable connectors to the autosampler inserting the right relative o-ring. Also the o-ring are included in the Standard Outfit.

**Figure 149.** Installation of the MAS 200R on the Flash 2000

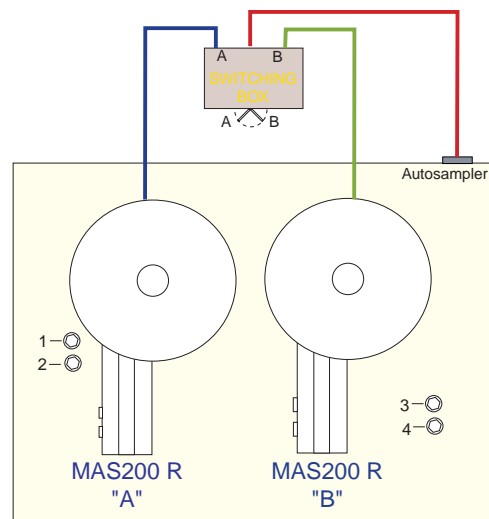


3. Connect the signal cable of the MAS 200R autosampler to the 2-pin connector, marked **Autosampler**, on the rear panel of the analyzer.

## Switching Box

When the analyzer is equipped with two MAS 200R autosamplers, it is possible to switch from a channel to the other by using a dedicated Switching Box as schematically shown in the following figure:

**Figure 150.** Scheme of the switching box



## The Sample Tray

The sample tray, shown in [Figure 151](#), can contain up to 32 samples numbered 0 to 31. It is provided with a reference pin to be introduced into the seat marked 0 (zero). The correct alignment of the pin is critical for the installation of the sample tray on the MAS 200 R autosampler.

**Figure 151.** Sample tray





**❖ To Install the Sample Tray**

The following operating sequence provides instructions for the correct installation of the sample tray.

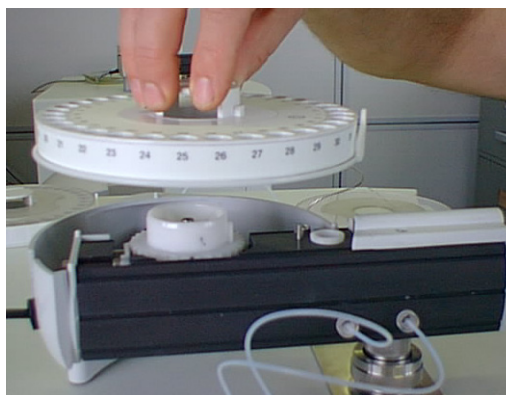
1. Manually rotate the toothed wheel clockwise until the guide located on its rim is perfectly aligned with the metal pin of the autosampler body, as shown in [Figure 152](#).

**Figure 152.** Alignment of the Toothed Wheel



2. Check that the tray reference pin is in correspondence with the seat marked “zero”, as shown in [Figure 151](#).
3. Place the sample tray, with the reference pin in correspondence with the “zero” seat, onto the toothed wheel, paying attention to have the base match with the guides, as shown in [Figure 153](#).
4. Rotate the sample tray clockwise to select the sample position 1.

**Figure 153.** Installation of the Sample Tray



5. Place the protection cover over the sample tray, as shown in [Figure 154](#), with the surface marked “Side up” turned towards you.

**Figure 154.** Protection cover



**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 to not invert the cover; the surface marked “*Side-up*” must be turned towards you.

## Current Maintenance of the MAS 200 R Autosampler

The MAS 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.

To do this, operate as described in the following operating sequence.

### ❖ To Clean the Shaft of the MAS 200R Autosampler


This operating sequence provides instructions for the MAS autosampler maintenance.

#### Materials Required

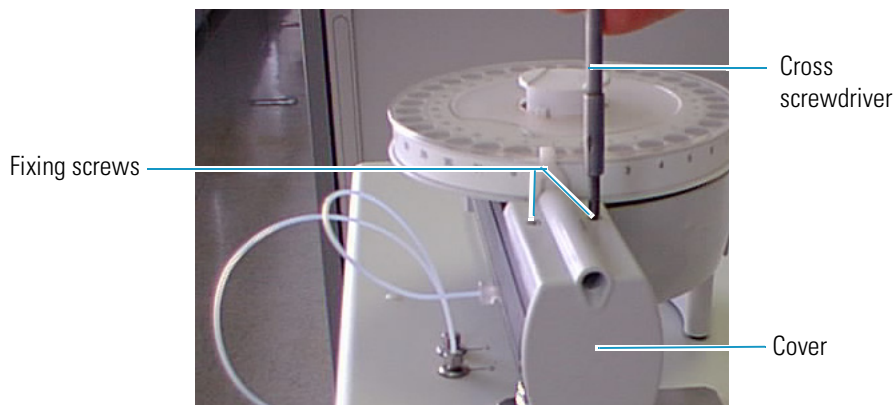
Cross screwdriver


Cloth

Silicon grease (For use at pressures down to  $10^{-6}$  mm Hg)

1. In Main Menu, select the menu **Edit > Edit Elemental Analyzer parameters** or just click the icon . The **Analyzer Parameter** window will be displayed.
2. Select the **Flow/Timing** option. Set **Carrier** Off in the **Gas Flow** box.
3. Click **Send** and wait 2-3 minutes to discharge the gas out the circuit.
4. Remove the autosampler cover undoing the two fixing screws, as shown in [Figure 155](#).

**Figure 155.** Removal of the MAS 200R autosampler cover



5. From Main Menu select the menu **View > View Elemental Analyzer status** or directly 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 156](#).

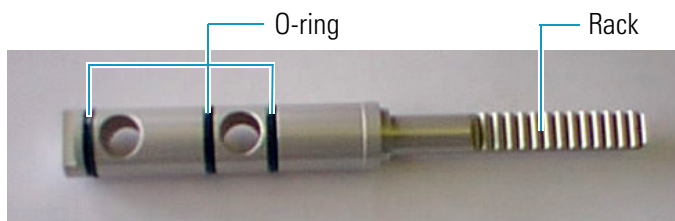
**Figure 156.** Removal of the shaft



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.

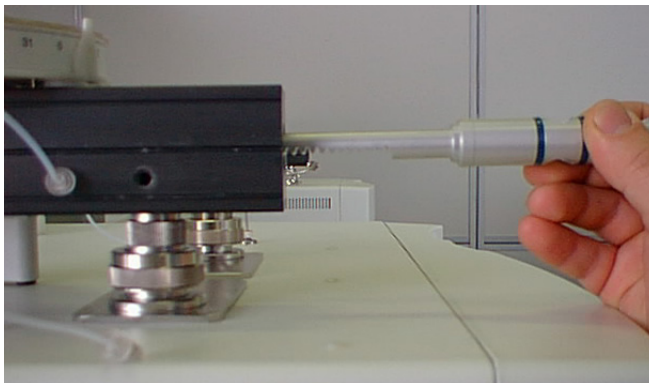
[Figure 157](#) shows the shaft of the MAS autosampler.

**Figure 157.** Shaft of the autosampler



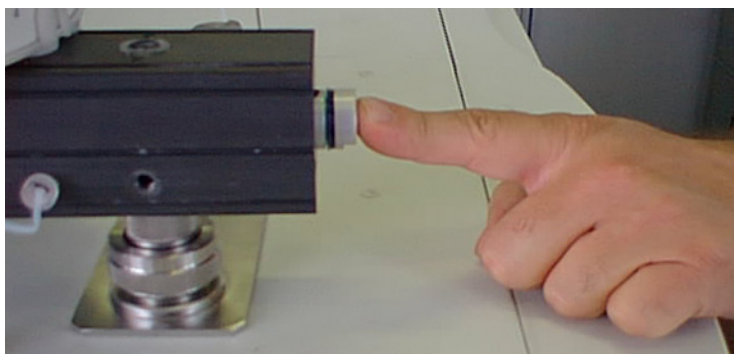
10. Re-introduce the shaft into the autosampler until in place keeping its rack turned downward, as shown in [Figure 158](#).

**Figure 158.** Reinstalling the shaft (1)



11. Slightly pushing the shaft with one finger of your hand, as shown in [Figure 159](#), 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 159.** Reinstalling the shaft (2)



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

# Installing and Servicing the CM2 Manual Sampler

## Introduction

The CM2 manual sampler, shown in [Figure 160](#), consists of a base block provided with a sampling chamber and a lever for the injection of samples into the reactor.

The sample is placed inside the sampling chamber, which must then be closed by turning the appropriate nut. On the Start command, after the time delay set in the method, push the lever towards the sampler and the sample will fall into the reactor. The lever is brought to its starting position.

**Figure 160.** CM2 manual sampler



## Installation

The sampler is directly installed on the connecting nut of the concerned reactor.

### ❖ To Install the CM2 Manual Sampler

The following operating sequence provides instructions to install the autosampler on the Flash 2000.

**Note** The installation sequence is common to all instrument configurations.

#### Material Required

8 mm wrench

1. Connect the tubes coming from the gases connections, located on the analyzer, to the relevant connections on the sampler.
  - a. Connect the tube for Helium as carrier gas (connection 2) to the connection located on the left side of the sampler.
  - b. Connect the tube for Helium as reference gas (connection 1) to the connection located on the lower part of the sampler.
2. Manually screw the autosampler nut on the concerned channel.

## Current Maintenance of the CM2 Sampler

The CM2 sampler does not usually require maintenance. However, when the instrument is extensively used, it is a good practice to replace the three seals inside it. To do this, operate as described in the following operating sequence.

### ❖ To Replace the CM2 Sampler Seals

The following operating sequence provides instructions to maintaining the CM2 manual sampler.

**CAUTION** The dismounting and re-assembling of the sampler MUST be performed with great care and under very clean operating conditions, because the presence of even the smaller solid particle in the sampling mechanism may jeopardize the performance of the sampler producing high blank values.

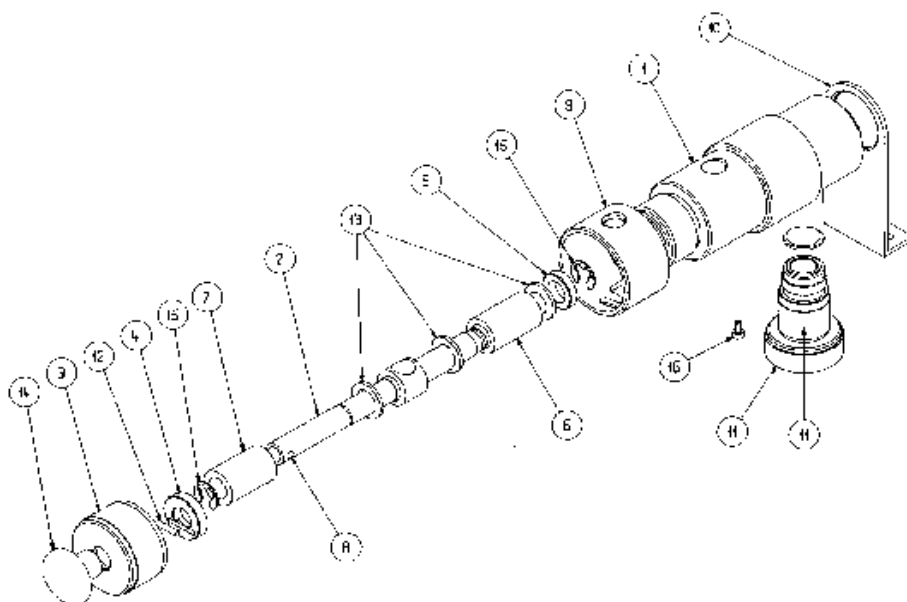
#### Material Required

Spanner

Seals (O-ring)

Screwdriver

**Figure 161.** Parts of the CM2 manual sampler



Proceed as follows referring to [Figure 161](#).

1. Remove the CM2 manual sampler from the analyzer.
2. Insert a spanner into the hole A and unscrew the handle 14.

## 13 Maintenance

### Installing and Servicing the CM2 Manual Sampler

3. Unscrew the locking nut **3**.
4. Remove the shaft **2** completely together with parts **12**, **4**, **15**, **7**, **13**, **6** and **8**.
5. Take off the pin **12** from the shaft **2** and remove the ring **4**.
6. Remove clip **15** from both ends
7. Take off **6**, **7** and **8**.
8. Replace the three o-rings **13**.
9. Reassemble all parts in the reversed order.
10. When introducing the shaft **2**, check that its plane is down and make sure that the hole of the body **1** and the hole of **2** are aligned.
11. The manual sampler should slide smoothly and easily into the body. Otherwise check again the alignment between the shaft **2** and the pin **12**.
12. Screw the locking nut **3**.
13. Insert a spanner into the hole **A** and screw the handle **14**.

# Troubleshooting

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

## Contents

- [Safety Cut Off](#)
- [EFC-t Module](#)

## Safety Cut Off



Instrument malfunctioning, due to a component failure or to abnormal operating conditions, is identified by the red lighting of the Safety Cut Off LED indicator. When lit, this LED indicates that the furnaces and detector oven power has been cut off for safety reasons.

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

### ❖ To Display the Error Message

Proceed as follows:

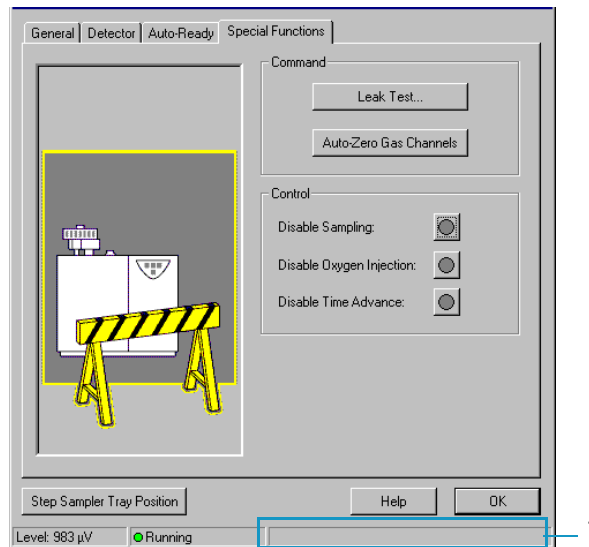
1. In the Main menu select **View > View Elemental Analyzer Status** or just press the icon



2. In the displayed dialog window, select the option **Special Functions**. The dialog window of [Figure 162](#) is displayed.
3. Read the error message in the reading box located on the lower right side of the window, below the buttons **Help** and **OK**.
4. Refer to [Table 45](#) to find out the error status and have mode information.



Figure 162. Special function window



1. Error Message Reading Box

The following Table 45 reports the error messages and the explanation of the relevant correlated problem.

Table 45. Error messages

Message	Description
safety cut off Voltage out limit	Voltage supplied to electric circuit is too low. Not significant error. When voltage returns within limits, the instrument automatically goes back to operating conditions.
safety cut off SSR thermal protection	Furnaces control SSR relay is overheated
safety cut off out of limit right	Right furnace has exceeded the set temperature limit. Temperature has been set with furnace missing
safety cut off out of limit left	Left furnace has exceeded the set temperature limit.
safety cut off Oven limit	The detector oven temperature exceeds the 220 °C. Error may be due to a probe malfunction, etc.
safety cut off Oxygen pressure too low	The Oxygen pressure supply is too low



**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 46](#) to find the component responsible of the EFCt module malfunctioning and to solve the relevant problem.

**Table 46.** 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 don't decrease up to zero performing the Leak Test.	EV3 and/or EV4	Check voltage supply Replace the solenoid valve



## Customer Communication

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

### How to Contact Us

This appendix contains contact information for Thermo Fisher Scientific office. To contact your local Thermo Fisher Scientific office or affiliate, please refer to:

#### **Thermo Fisher Scientific**

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United Kingdom

Tel: +44(0)1223 347400 Fax: +44(0)1223 347403

This appendix also contains a one-page *Reader Survey*. Use this survey to give us feedback on this manual and help us improve the quality of our documentation.

# Reader Survey

**Product:** Flash 2000 Elemental Analyzer  
**Manual:** Operating Manual  
**Part No.:** 317 082 41

Please help us improve the quality of our documentation by completing and returning this survey. Circle one number for each of the statements below:

	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 install the system using this manual.	1	2	3	4	5

If you would like to make additional comments, please do. (Attach additional sheets if necessary).

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Fax or mail this form to:

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Tel: +44 (0) 1223 347400  
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## Abbreviations

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**

<b>A</b> ampere	<b>ft</b> foot
<b>ac</b> alternating current	<b>g</b> gram
<b>ADC</b> analog-to-digital converter	<b>GND</b> electrical ground
<b>b</b> bit	<b>h</b> height
<b>B</b> byte (8 b)	<b>h</b> hour
<b>baud rate</b> data transmission speed in events per second	<b>H</b> hydrogen
<b>C</b> Carbon	<b>harmonic distortion</b> A high-frequency disturbance that appears as distortion of the fundamental sine wave
<b>°C</b> Celsius	<b>He</b> Helium
<b>cm</b> centimeter	<b>HPAR</b> High Performance Alloy Reactor
<b>CPU</b> central processing unit (of a computer)	<b>HV</b> high voltage
<b>CSE</b> Customer Service Engineer	<b>Hz</b> hertz (cycles per second)
<b>&lt;Ctrl&gt;</b> control key of the keyboard	<b>IEC</b> International Electrotechnical Commission
<b>d</b> depth	<b>in.</b> inch
<b>DAC</b> digital-to-analog converter	<b>I/O</b> input/output
<b>dc</b> direct current	<b>k</b> kilo ( $10^3$ or 1024)
<b>DS</b> data system	<b>K</b> Kelvin
<b>EMC</b> electromagnetic compatibility	<b>kg</b> kilogram
<b>ESD</b> electrostatic discharge	<b>kPa</b> kilopascal
<b>°F</b> Fahrenheit	<b>l</b> length
<b>FSE</b> Field Service Engineer	

## Glossary:

**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** sulphur

**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<sup>-1</sup> instead of °C/min

g L<sup>-1</sup> instead of g/L

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