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# Flash*Smart* Elemental Analyzer

Organic Elemental Analyzers

## Operating Manual

31707001 Revision E July 2020

ThermoFisher SCIENTIFIC

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# Legal Notices

## Original Operating Instructions



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Revision E released in July 2020.

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# Reader's Survey

**FlashSmart Operating Manual, P/N 31707001, Revision E**

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

fold

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

_____ _____
_____ _____
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# Customer Registration Card

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# Contents

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# Using this Manual

This manual provides detailed information for the use of the Thermo Scientific™ Flash*Smart*™ Elemental Analyzer.

### Contents

# About your System

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer is a fully automated Elemental Analyzer.

The Flash*Smart* EA operates with dynamic combustion of the sample (modified Dumas method) for nitrogen, carbon, hydrogen and sulfur determination, and oxygen determination by pyrolysis.

The Analyzer can be equipped with one or two totally independent furnaces. The double channels can be connected with a single pneumatic circuit. The system also allows the installation of two analytical circuits, which are used alternatively and completely automated through the Thermo Scientific™ MultiValve Control (MVC) Module. Each analytical circuit can receive its own autosampler.

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

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

# Typographical Conventions

This section describes typographical conventions that have been established for Thermo Fisher Scientific manuals.

## Signal Words

Make sure that you follow the precautionary statements presented in this manual. The special notices appear different from the main flow of text:

**NOTICE** Points out possible material damage, data loss, impaired data quality and other important information in connection with the instrument.

**Tip** Highlights helpful information that can make a task easier.

## Viewpoint Orientation

The expressions *left* and *right* used in this manual always refer to the viewpoint of a person that is facing the front side of the instrument.

## Data Input

Throughout this manual, the following conventions indicate data input and output via the computer:

- Messages displayed on the screen are represented by capitalizing the initial letter of each word and by italicizing each word.
- Input that you enter by keyboard is identified by quotation marks: single quotes for single characters, double quotes for strings.
- For brevity, expressions such as "choose **File > Directories**" are used rather than "pull down the File menu and choose Directories."
- Any command enclosed in angle brackets < > represents a single keystroke. For example, "press **<F1>**" means press the key labeled *F1*.
- Any command that requires pressing two or more keys simultaneously is shown with a plus sign connecting the keys. For example, "press **<Shift>** + **<F1>**" means press and hold the <Shift> key and then press the <F1> key.
- Any button that you click on the screen is represented in bold face letters. For example, "click **Close**."

## Topic Headings

The following headings are used to show the organization of topics in a chapter:

# Chapter Name

## Second Level Topics

### Third Level Topics

#### Fourth Level Topics

# Contacting Us

There are several ways to contact Thermo Fisher Scientific. You can use your smartphone to scan a QR Code, which opens your email application or browser.

| Contact | Link / Remarks | QR Code |
|---|---|---|
| **Brochures and Ordering Information** | www.thermofisher.com/OEA | |
| **Service Contact** | www.unitylabservices.com | |
| **EU REACH Statement**<br>**Health and Safety Form** | www.thermoscientific.com/Technicaldocumentation | |
| **Technical Documentation SharePoint** | ❖ **To get user manuals for your product**<br>1. With the serial number (S/N) of your instrument, request access on our customer SharePoint as a customer at www.thermoscientific.com/Technicaldocumentation<br>2. For the first login, you have to create an account. Follow the instructions given on screen. Accept the invitation within six days and log in with your created Microsoft™ password.<br>3. Download current revisions of user manuals and other customer-oriented documents for your product. Translations into other languages may be available there as well. | |
| **Customer Feedback** | ❖ **To suggest changes to this manual**<br>You are encouraged to report errors or omissions in the text or index. Send an email message to the Technical Editor at documentation.bremen@thermofisher.com.<br>The PDF versions of our manuals allow adding comments with Adobe Acrobat Reader or other freely available PDF reader programs. | |

To contact your local Thermo Fisher Scientific office or affiliate, please refer to:

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

**Dr. Liliana Krotz; OEA Application Manager**
Email: liliana.krotz@thermofisher.com

# Training

Thermo Fisher Scientific offers worldwide training on instruments and software. Experience has shown that maximum results can be obtained from a scientific instrument if the instrument operator receives an adequate training. We recommend that the key operator undertake training at Thermo Fisher Scientific Rodano - Milan (Italy), Thermo Fisher Scientific Bremen (Germany), at your site, or at one of the local Thermo Fisher Scientific offices. For information on training courses and enrollment, please contact your local Thermo Fisher Scientific office.

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

**Thermo Fisher Scientific S.p.A.**
Strada Rivoltana km 4
20090 Rodano, Milan, Italy
Tel. +39 02 950591

Email: support.OEA.bremen@thermofisher.com
Email: walter.galotta@thermofisher.com

2

# Safety

This chapter contains information that is important for your own safety or the safety of others, and that prevents damage to the instrument, Read this chapter carefully before you install or operate the instrument and its accessories, or come into contact with it.

**Contents**

# Safety Symbols and Signal Words in this Manual

Notices that concern the safety of the personnel who operate the Flash*Smart* Elemental Analyzer appear different from the main flow of text:

| | |
|---|---|
| ⚠ | Always be aware of what to do with and the effect of safety information. |
| ⚠CAUTION | Points out a hazardous situation that can lead to minor or medium injury if it is not avoided. |
| ⚠WARNING | Points out a hazardous situation that can lead to severe injury or death if it is not avoided. |
| ⚠DANGER | Points out a hazardous situation that will lead to severe injury or death if it is not avoided. |

## Observing this Manual

Keep this manual always near the instrument to have it available for quick reference.

| | |
|---|---|
| ⚠ | Be sure to read and comply with all precautions described in this manual. |

System configurations and specifications in this manual supersede all previous information received by the purchaser.

# Safety Symbols on the Instrument

Table 2-1 lists all safety labels on the instrument. See the indicated safety notices to prevent risk of harm to the operator and to protect the instrument against damage. If they are present, read and obey the instructions on the labels.

**Table 2-1.** Instrument marking and symbols

| Symbol | Description |
|---|---|
|  | Alternating current |
|  | On (supply) |
|  | Off (supply) |
|  | Instruction manual symbol affixed to product. Indicates that the user must refer to the manual for specific information to avoid personal injury or damage to the product. |
|  | Caution, risk of electric shock |
|  | Caution, hot surface |
|  | Caution, biohazard |
|  | Symbol in compliance to the Directive 2012/19/EU on Waste Electrical and Electronic Equipment (WEEE) placed on the European market after August, 13, 2005. |

## Labels Location on the Instrument

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

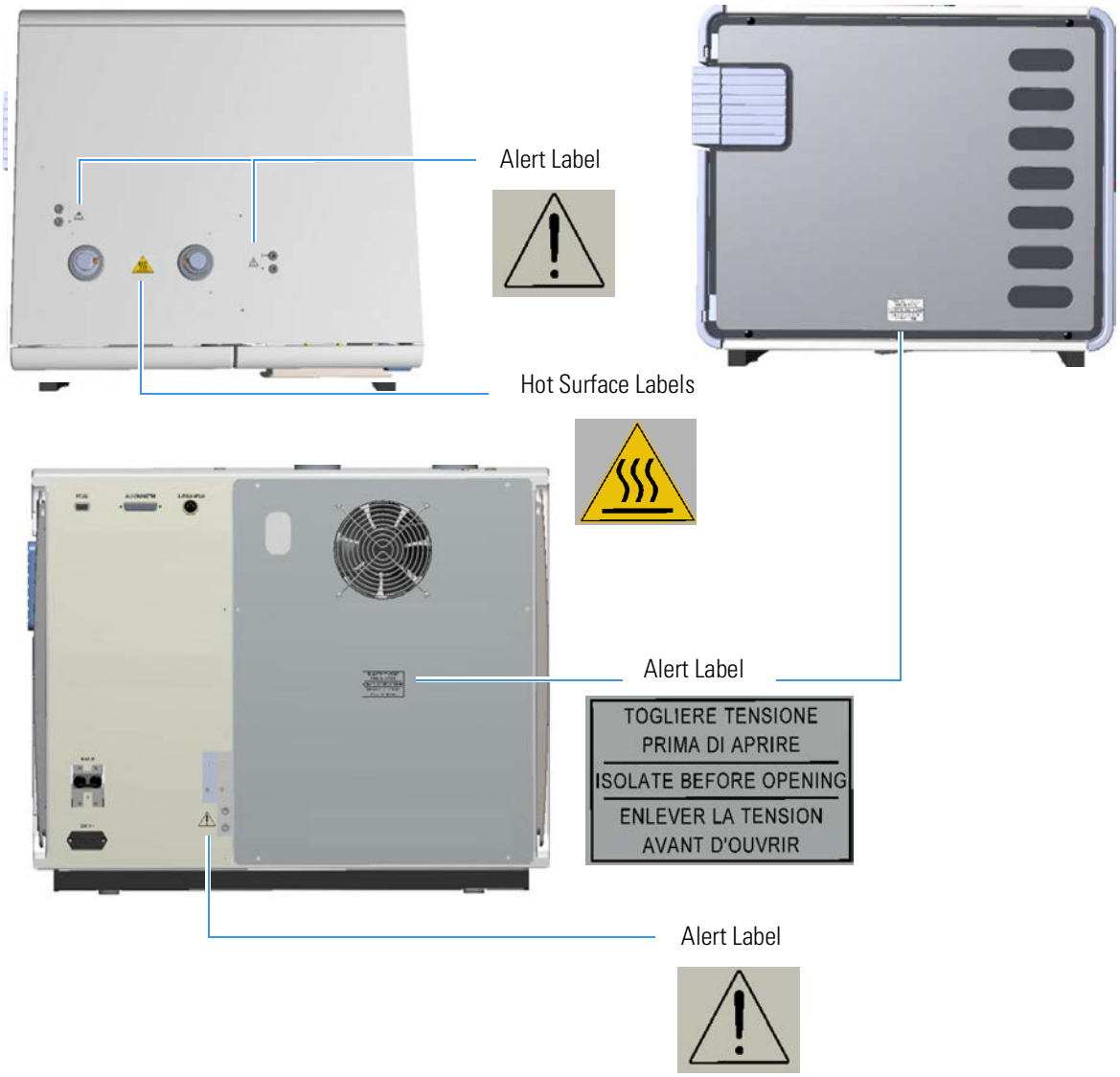



**Figure 2-1.** Flash*Smart*: alert labels

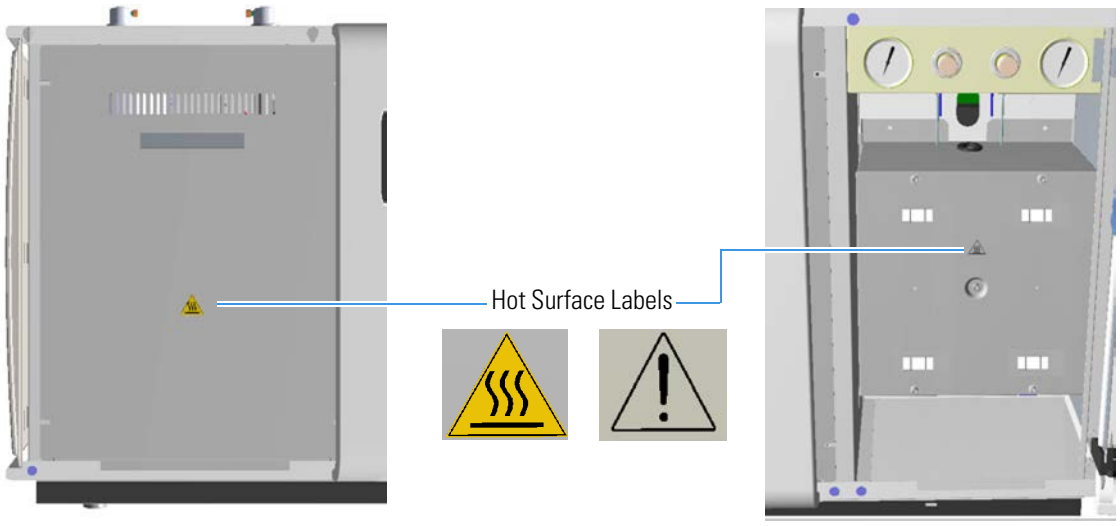



**Figure 2-2.** Flash*Smart*: Hot Surface labels

## Rating Plate

To identify correctly the instrument when you contact Thermo Fisher Scientific, always have the information from the rating plate available. The rating plate is attached to the power supply area at the rear side of the instrument. See Figure 2-3. It contains the serial number, which is important in any type of communication with Thermo Fisher Scientific. Especially, the serial number is needed to access the SharePoint of the Bremen Technical Documentation group. See "Contacting Us" on page 1-5.

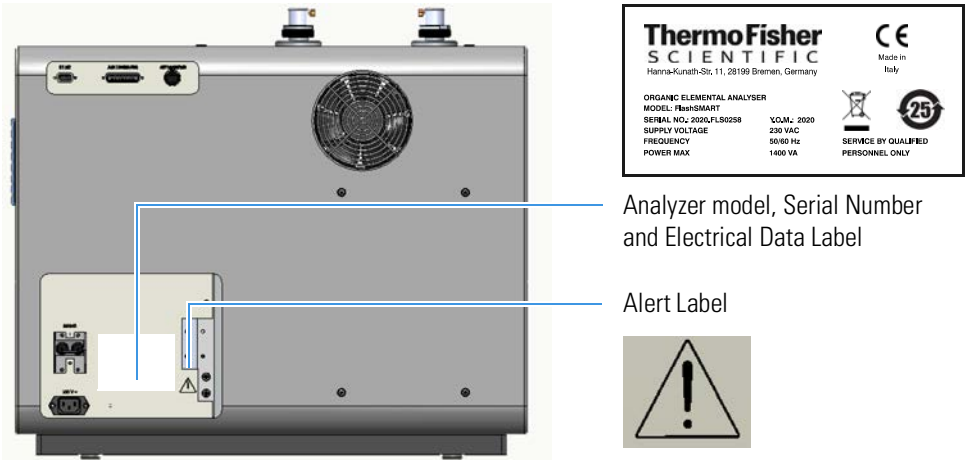



**Figure 2-3.** Flash*Smart*: rating plate (example)

# Intended Use

⚠ Obey the following usage guidelines when you operate your Flash*Smart* Elemental Analyzer:

- The instrument is designed to be placed on a bench in the laboratory. It is not designed for the use outdoors.
- The instrument is designed for use in research labs, quality control labs, contracts labs, etc. It is not designed for use in diagnostic or medical therapeutic procedures.

The instrument must be used according to the specifications of this guide. Improper use can adversely affect the instrument protection. If the equipment is connected to optional instruments, such as computer, balance, and so on, the degree of insulation of peripheral devices should be equivalent or higher (double or reinforced) than that of the Flash*Smart* EA.

The analyzer operation requires the use of chemical substances having different hazard specifications. Before you use chemicals, read the hazard indications and information reported in the Material Safety Data Sheet supplied by the manufacturer referring to the relevant CAS (Chemical Abstract Service) number.

### Notice on the Susceptibility to Electromagnetic Transmissions

Your instrument is designed to work in a controlled electromagnetic environment. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

### Notice on Lifting and Handling of Thermo Scientific Instruments

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

### Notice on the Proper Use of Thermo Scientific Instruments

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

## Qualification of the Personnel

⚠ Personnel that install or operate the Flash*Smart* Elemental Analyzer must have the following qualifications:

- Electrical Connections
  The electrical installation must be carried out by qualified and skilled personnel (electrician) according to the applicable regulations (for example, cable cross-sections, fuses, earth grounding connection). Refer to the *FlashSmart Preinstallation Requirements Guide* for the specifications.
- Installation
  Only employees of Thermo Fisher Scientific or personnel who act on behalf of Thermo Fisher Scientific are allowed to install the Flash*Smart* EA.
- General Operation
  The Flash*Smart* EA is designed to be operated by qualified laboratory personnel. Before they start, all users must be instructed about the hazards presented by the instrument and the chemicals applied. The users must be advised to read the relevant Material Safety Data Sheets (MSDSs).
- Decommissioning
  Only employees of Thermo Fisher Scientific or personnel who act on behalf of Thermo Fisher Scientific are allowed to decommission the Flash*Smart* EA.

# Electric Safety Precautions

**⚠ WARNING** **High Voltage.** High voltages that can cause an electric shock are used in the instrument.

Observe the following safety precautions when you operate or perform service on your instrument:

- The instrument is properly grounded in accordance with regulations when shipped. You must not any changes to the electrical connections or to the chassis of the instrument to ensure safe operation.
- Do not run the system without the housing on. Permanent damage can occur. When you leave the system, make sure that all protective covers and doors are properly connected and closed, and that heated areas are separated and labeled to protect unqualified personnel. Do not rig or override any safety switches or safety functions. Risk of electric shock, burn hazard or damage to your system can occur.
- Do not turn on the instrument if you suspect that any kind of electrical damage has incurred. Instead, disconnect the power cords and contact a Thermo Fisher Scientific field service engineer for a product evaluation. Do not try to use the instrument until it has been evaluated. Electrical damage may have occurred if the system shows visible signs of damage, or has been transported under severe stress.
- Do not place any objects on the instrument—especially not containers with liquids—unless it is requested by the user documentation. Leaking liquids might get into contact with electronic components and cause a short circuit.

## Safety Cutoff Device

When an alarm condition is detected, this device cuts off the power to the heating resistors of the oxidation, reduction furnaces. For more details, see "Safety Cutoff Device" on page 15-2.

# Residual Hazards

Users of the Flash*Smart* Elemental Analyzer must pay attention to these residual hazards.

**⚠WARNING** **High Voltage.** Do not open the electrical compartment because there are no user serviceable parts inside. Any operation inside the compartment must be carried out by authorized and trained Thermo Fisher Scientific personnel.

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

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

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

**NOTICE** Always use original Thermo Fisher Scientific materials and products. The use of materials that do not meet the technical specifications of our products does not ensure a good operation of the instrument and may even cause damage to it.

# Personal Protective Equipment

Appropriate safety clothing must be worn at all times while you operate the instrument, particularly when you handle hazardous material.

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

## Eye Protection

The type of eye protection required depends on the hazard. For most situations, safety glasses with side shields are adequate. Where there is a risk of splashing chemicals, goggles are required.

## Protective Clothing

When the possibility of chemical contamination exists, protective clothing that resists physical and chemical hazards should be worn over street clothes. Lab coats are appropriate for small chemical splashes and solids contamination, while plastic or rubber aprons are best for protection from corrosive or irritating liquids.

## Gloves

For handling chemical compounds and organic solvents, Thermo Fisher Scientific recommends white nitrile clean room gloves from Fisher Scientific or Unity Lab Services.

For handling hot objects, gloves made of heat-resistant materials (for example, leather) should be available.

## Mask

For handling chemical compounds and filling materials for preparing and cleaning reactors and filters, and for removing the ashes from the crucible when used.

# Gases Precautions

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

### Precaution for Helium

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

### Precaution for Oxygen

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

### Precautions for Argon

Argon is a nonflammable, nontoxic, colorless and odorless gas or refrigerated liquid. Although argon is not flammable or toxic, it can be dangerous. Argon gas is harmful if inhaled, and refrigerated liquid argon can cause tissue damage if it comes into contact with skin. When working with this gas good ventilation is essential. Otherwise, the gas can be hazardous to your health!

### Precaution for Nitrogen

Nitrogen is a nontoxic, odorless, colorless, nonflammable compressed gas stored in cylinders at high pressure. It can cause rapid suffocation when concentrations are sufficient to reduce oxygen levels below 19.5%.

### Precaution for Hydrogen

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

## Venting Toxic Gases

When you analyze toxic compounds, be aware that during the normal operation of the Flash*Smart* Elemental Analyzer some of the sample might be vented outside the instrument through the inlet and detector exits. Therefore, make sure to vent the exhaust gases to a fume hood. Consult local Environmental and Safety Regulations for instructions in exhausting fumes from your system.

3

# Getting Familiar with Your Flash*Smart* Elemental Analyzer

This chapter provides information to familiarize with the Thermo Scientific™ Flash*Smart*™ Elemental Analyzer. Here, a detailed description of the instrument's components are provided.

**Contents**

# Technical Features

Table 3-1 summarizes the major technical features of the Flash*Smart* Elemental Analyzer.

**Table 3-1.** Technical features of the Flash*Smart* Elemental Analyzer

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

# Instrument Configurations

The Flash*Smart* Elemental Analyzer is available in various versions. Analytical techniques, pneumatic circuits and standard outfits are different for each version. See Table 3-2.

Each configuration of the Flash*Smart* Elemental Analyzer has its own dedicated pneumatic circuit. For details, see Chapter 4, "Analytical Principles."

An optional MultiValve Control (MVC) Module can be installed on the back panel of the instrument in CHN/O and CHNS/O configurations. It is already installed as default in the double channel configurations MVC CHN/O, MVC CHNS/O, MVC CHN/CHN and MVC CHNS/CHNS.

For details, refer to the *MultiValve Control (MVC) Module for FlashSmart Elemental Analyzer Instruction Manual.*

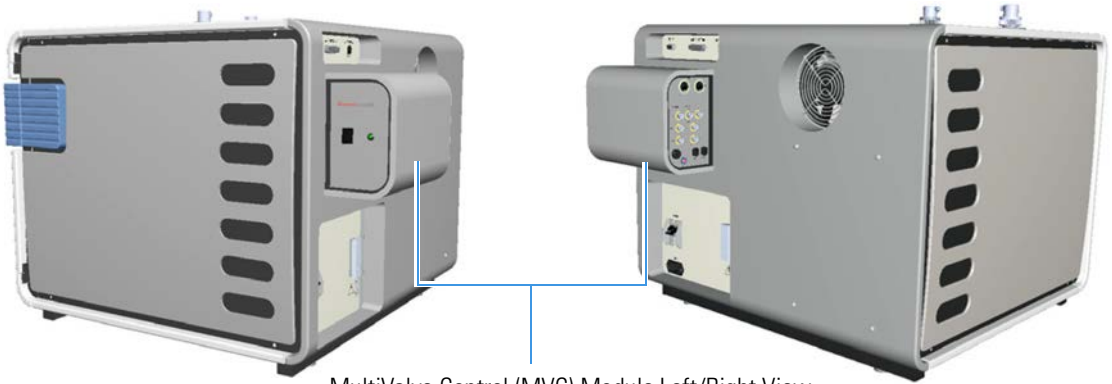



**Figure 3-1.** MVC Module

**Table 3-2.** Flash*Smart* Series: Instrument Configurations

| Analyzer Configuration | Description |
|---|---|
| **CHN** | 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. |
| **CHNS** | 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. |
| **CHN/O** | 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.<br><br>The determination of carbon, hydrogen and nitrogen is performed in a single sample analysis, whereas oxygen determination is performed separately. |

**Table 3-2.** Flash*Smart* Series: Instrument Configurations, continued

| Analyzer Configuration | Description |
|---|---|
| **CHNS/O** | 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. The determination of carbon, hydrogen, nitrogen and sulfur is performed in a single sample analysis, whereas oxygen determination is performed separately. |
| **N Org.** | 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. |
| **N Lubricant** | For the determination of the total amount of nitrogen present in lubricants, lubricant additives, fuel additives, petrochemical products. |
| **N/Protein** | 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** | For the determination of nitrogen content in samples of different nature, belonging to the brewing industry, such as malt, barley, wort, and beers. |
| **NC Org.** | For the determination of the amount (%) of nitrogen and carbon contained in materials of different kinds:<br>• *Synthetic materials:* polymers, rubbers, tires, and so on.<br>• *Explosives:* nitrocellulose, TNT, gun powder, and so on.<br>• *Special materials:* carbon fibers, glass fibers, conductive polymers, graphite, and so on.<br>• *Metallurgy:* metal powders, steels, and so on.<br>• *Environmental analyses:* muds, discards, organic wastes, and so on. |
| **NC Soils** | For the determination of the nitrogen and carbon content in soil samples. |
| **NCS** | 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. |
| **MVC CHN/O** | Includes the CHN channel, the O channel, two MAS Plus autosamplers and the MultiValve Control (MVC) Module. |
| **MVC CHNS/O** | Includes the CHNS channel, the O channel, two MAS Plus autosamplers and the MultiValve Control (MVC) Module. |
| **MVC CHN/CHN** | Includes two CHN channels, two MAS Plus autosamplers and the MultiValve Control (MVC) Module. |
| **MVC CHNS/CHNS** | Includes two CHNS channels, two MAS Plus autosamplers and the MultiValve Control (MVC) Module. |

# Instrument Basics

The Flash*Smart* Elemental Analyzer comprises:

- **Furnaces** — The instrument 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.

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

  The **furnace temperature regulation** is monitored by a thermocouple appropriately located in the furnace.

  The **furnace cooling** time varies according to the operating temperature setting.
- **Thermal conductivity detector (TCD)** — Located in a thermostatic chamber at controlled programmable temperature. This chamber can also accommodate the analytical column according to the instrument configuration.
- **Chromatographic columns** — 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 CHN/O, CHNS/O, MVC CHN/O, MVC CHNS/O, MVC CHN/CHN and MVC CHNS/CHNS instrument versions use two analytical columns placed inside the oven.
- **Adsorption filters** — They can be made of Pyrex™ or Plexiglas™ according to the analytical configuration.
- **Reactors** — Tubes made of quartz, alloy steel and ceramic with various materials according to the analytical configuration. See Chapter 8, “Preparing the Reactors and the Adsorption Filters.”
- **Autosampler** — Performs the automatic injection of samples into the reactor.
- **Pneumatic compartment** — Consists of two pressure reducers, two pressure gauges, and of several lines fitted with an thermo-regulator electronic flow controller (EFC-t), which ensures the switching between carrier gas and oxygen, and controls the flow values.
- **Electrical compartment** — Comprises the electronic boards for the instrument power supply and control.

- **User interface** — The instrument is not provided with independent keyboard and display.
  A status panel on the instrument front allows you to monitor the instrument status.

## Software Requirements

The instrument is fully controlled by the computer through the dedicated software named **Eager*Smart*** also used for data acquisition, data handling, and interpretation of the acquired results. The Eager*Smart* Data Handling Software is compatible with commercially available computers and Microsoft™ Windows™ 7, 8, or 10 operating system. See Table 3-3.

**Table 3-3.** Minimum Requirements for Personal Computer

| Components | Description |
|---|---|
| Computer | Any PC can be used, including laptop computer |
| | Operating System: Windows 7 / 8 / 10 |
| | Pentium Processor minimum 256 MHz |
| | Hard drive with at least 1 GB free |
| | One free COM port for instrument control |
| | One free COM port for balance, if required |
| | One free COM port for AI/AS autosampler for liquids, if required |
| | One free COM port for MultiValve Control (MVC) module (default or optional) |
| | One free USB port |
| | Alternatively, RS232/USB adapters can be used instead of the COM ports. |
| Monitor | Color 1024 × 768 or better |
| Printer | Any printer accepted by the operating system |

# Gases

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

The Flash*Smart* Elemental Analyzer uses the following gases:

- **Helium** (He) as carrier gas and purge gas [*CAS number 7440-59-7*]
- **Argon** (Ar) as carrier gas and purge gas [*CAS number 7440-37-1*]
- **Oxygen** ($O_2$) as gas for sample oxidation [*CAS number 7782-44-7*]
- **Nitrogen** ($N_2$) as service gas for saving the carrier gas when the instrument is not being used overnight, or on weekends, or for a prolonged period of time. [*CAS number 7727-37-9*]

**Tip** **Hydrogen** and **air** are required for supply the flame of the Flame Photometric Detector (FPD) when the Flash*Smart* Elemental Analyzer is coupled with the OEA/FPD System. For more information, refer to the *OEA/FPD System Instruction Manual*.

The Flash*Smart* EA requires the use of high purity gas chromatography grade (99,99%) gases. The maximum pressure of the Elemental Analyzer gas supplies is 700 kPa (7 bar).

The nominal pressure of the Elemental Analyzer gas supplies are:

- 250 kPa (2.5 bar) for helium (He), argon (Ar) (in N/Protein configuration), and nitrogen ($N_2$)
- 400–450 kPa (4.0–4.5 bar) for argon (in NC Soils configuration)
- 250–300 kPa (2.5–3 bar) for oxygen ($O_2$) according to the instrument configuration

**Tip** The pressure at the cylinder must be adjusted through the reducing valves at **50 kPa** higher than the nominal pressure.

## Using Hydrogen

Hydrogen and air are required for supply the flame of the Flame Photometric Detector (FPD) when the Flash*Smart* Elemental Analyzer is coupled with the OEA/FPD System.

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

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

Use these safety precautions when you use hydrogen:

- Make sure that the hydrogen cylinder complies with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure that the gas supply is turned off completely when you connect the hydrogen lines.
- Perform a *bubble test* to ensure that the hydrogen lines are leak-tight before you use the instrument as described in "Leak Test." Do not spray any electrical components during the bubble test.

## Leak Test

Before you start the system, perform a leak check as described in the following operating sequence.

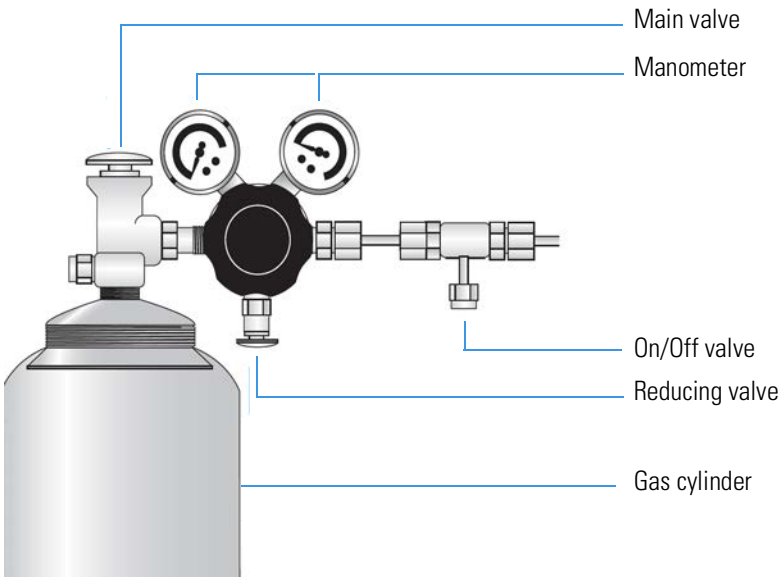



**Figure 3-2.** Gas Cylinder

### ❖ To perform a leak check

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

# Front Panel

The front panel of the Flash*Smart* Elemental Analyzer is shown in Figure 3-3. The front panel comprises:

- A furnace compartment. See page 3-13.
- A LED status panel. See page 3-27.
- An oven compartment including the pressure regulators, pressure gauges, the adsorption filter, and the thermostatic chamber with the TCD detector and the gas chromatographic column. See page 3-15.

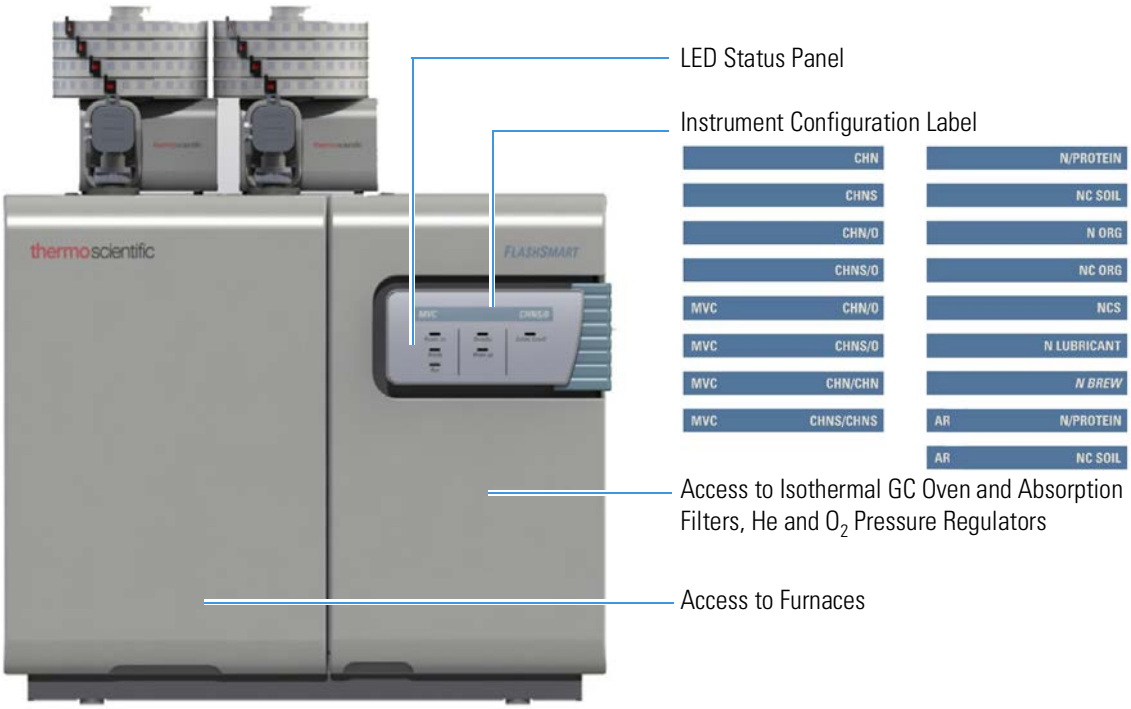



**Figure 3-3.** Instrument Front Panel

On the internal wall of the front door, you can find holders that are designed for a paper copy of the *FlashSmart Consumables and Spare Parts Catalog*, and for tools for maintenance. See Figure 3-4.

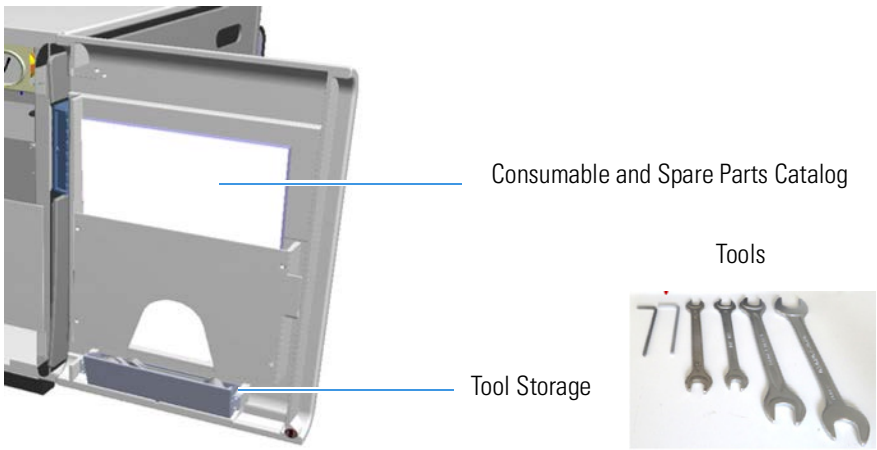



**Figure 3-4.** Right Door Internal View

# Rear Side Panel

The rear side panel of the Flash*Smart* Elemental Analyzer is shown in Figure 3-5. The rear side panel comprises:

- The cooling fan.
- The connections panel including the interface, gas inlets, and electrical connections. See page 3-22.
- The transformer compartment. Access to the transformer compartment is obtained by removing the back panel cover. See page 3-25.

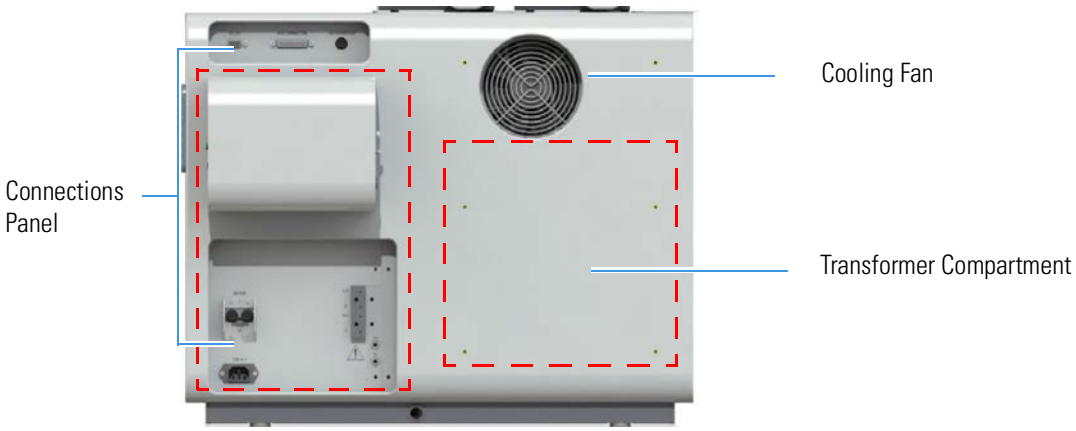



**Figure 3-5.** Instrument Back Panel

# Top Panel

The top panel of the Flash*Smart* Elemental Analyzer is shown in Figure 3-6. The top panel comprises:

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

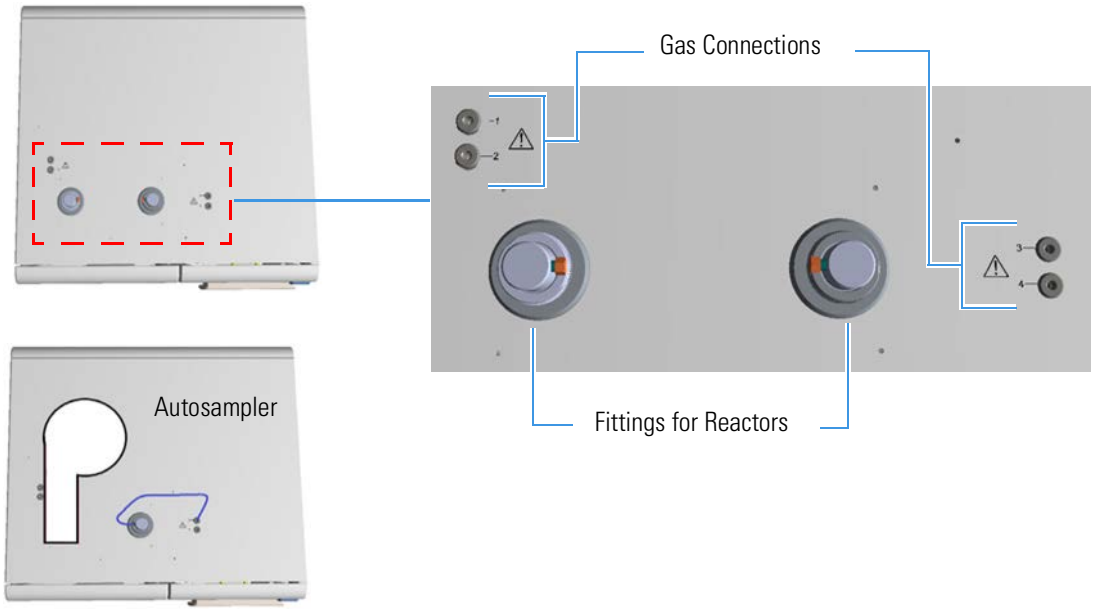



**Figure 3-6.** Instrument Top Panel

# Furnace Compartment

The furnace compartment can be accessed from the instrument front by removing (lifting) the cover. See Figure 3-7.

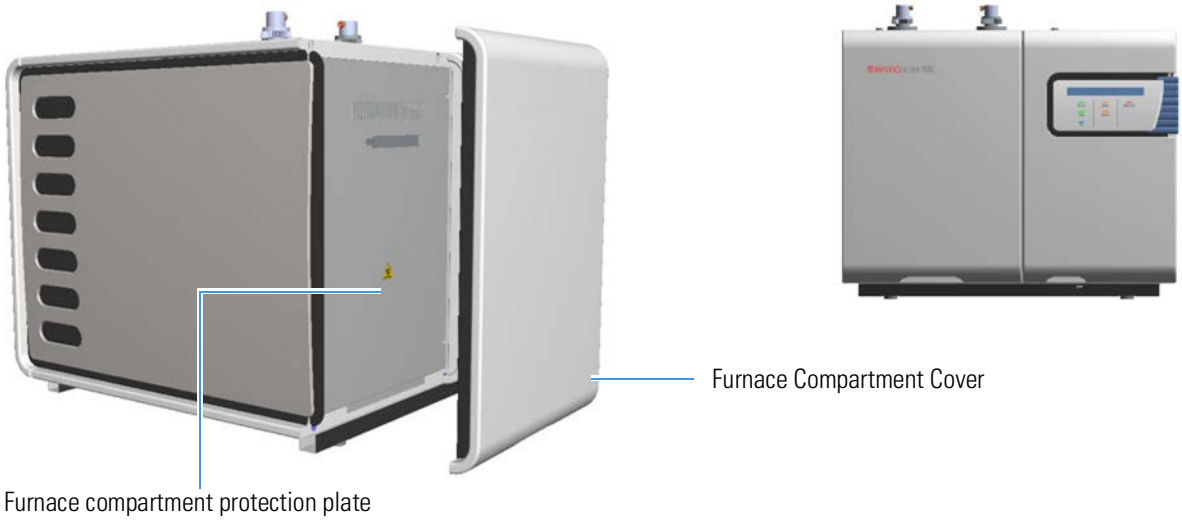



**Figure 3-7.** Furnace Compartment with Protection Plate

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

The furnaces are accessible by removing the protecting plate. See Figure 3-8.

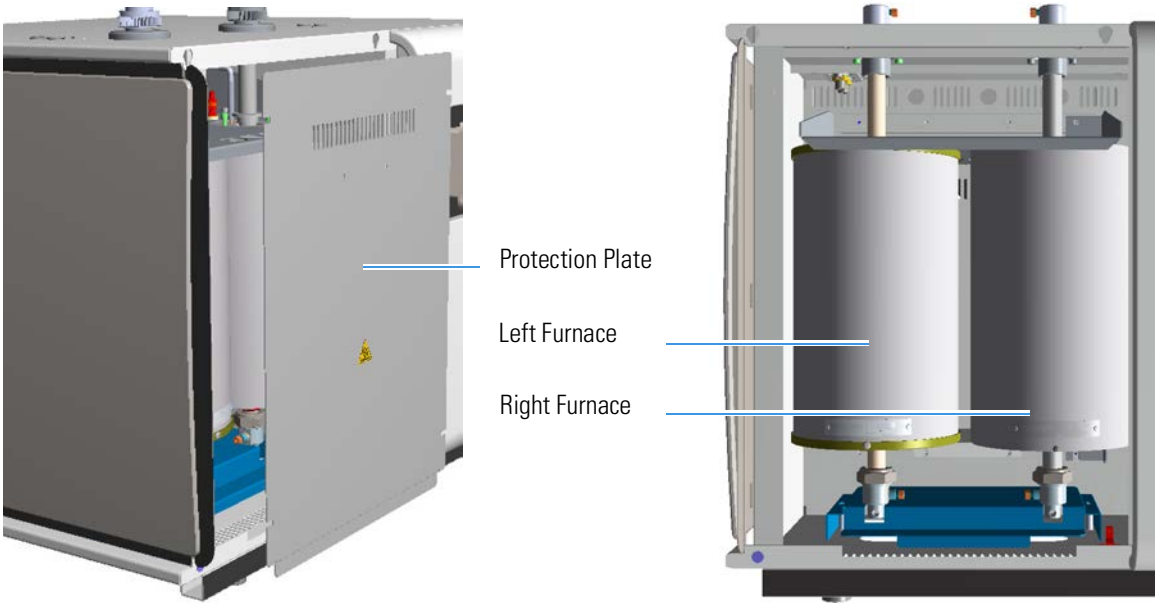



**Figure 3-8.** Furnaces Compartment

In the software, the furnaces are limited to the following maximum temperatures:

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

The furnace temperature is monitored by a thermocouple located inside the furnace with a real-time feedback to the software. The furnaces are cooled when required by the operator or if there is a safety cutoff. The cooling time depends on the operating temperature.

❖ **To operate the furnace**

1. The furnaces should not be operated above the maximum temperature stated in the operating instructions, which is 1100 °C and is limited by the Eager*Smart*™ Data Handling Software.
2. When the Elemental Analyzer is not used for sample analysis, either for periods of several hours (for example, overnight) or periods lasting several days or longer, the furnaces should be placed into Standy-By Mode as defined by the Standy-By Mode of the Eager*Smart* Data Handling Software, which can be activated automatically at the end of a sample sequence.

    a. If the instrument is not used for a long period of time (fore example, several weeks) it is recommended to switch off the furnaces. The furnaces should be cooled down with the furnace cool down procedure in the Eager*Smart* Data Handling Software. The cool down procedure is defined in the Operating Instructions of the Flash*Smart* Elemental Analyzer.

    b. After a Stand-By period or period where the furnaces are switched off, the furnaces should be brought back to the operating temperature following the furnace heat-up procedure defined in the Operating Instructions of the Flash*Smart* Elemental Analyzer.

3. The operator must not cause a short-circuit with the furnace heating element or its connections, or the furnace thermocouple. Only a qualified service engineer should check or test this. Operator interference on these parts is not covered by the warranty.

# Oven Compartment

The oven compartment is located behind the right front door of the instrument and can be accessed by opening the front door. Figure 3-9 shows the inside of the oven compartment.

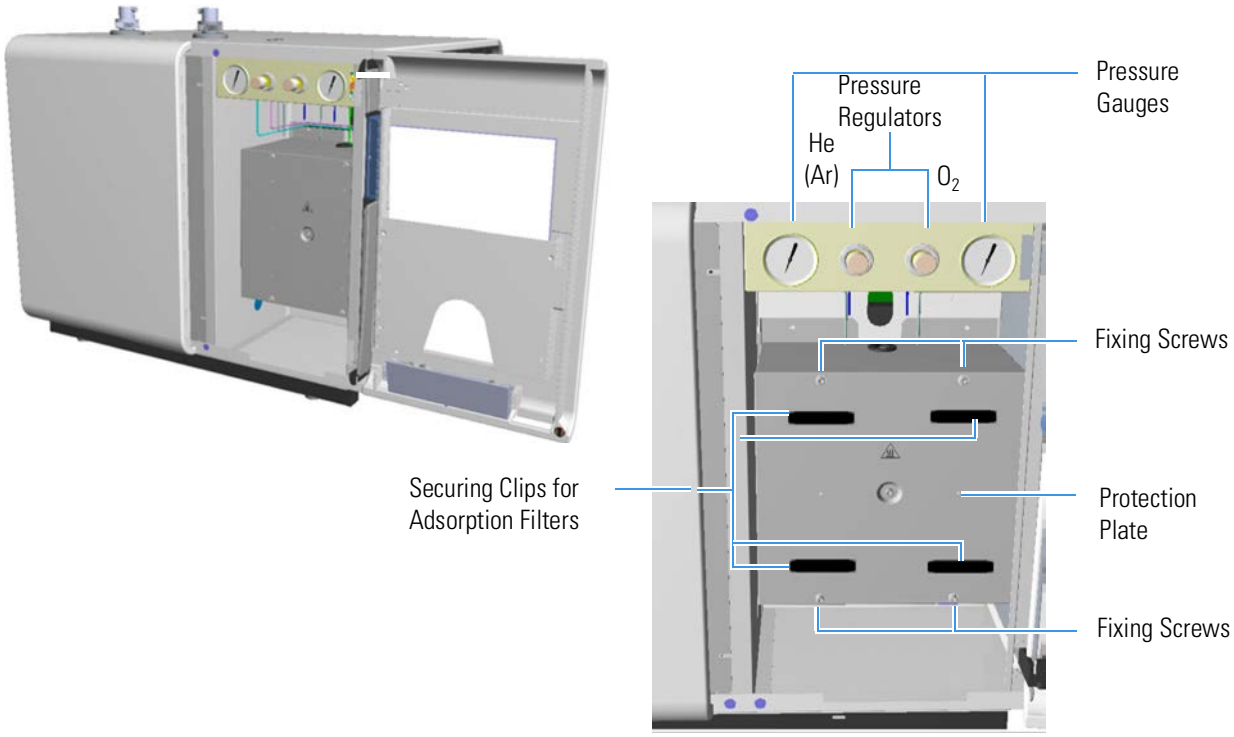



**Figure 3-9.** Oven Compartment Internal View

The oven compartment houses the helium (He) or argon (Ar) and oxygen ($O_2$) pressure regulators and pressure gauges, and the thermostatic chamber containing the thermal conductivity detector (TCD) and the gas chromatographic column located behind the protecting plate. See "To access the thermostatic chamber" on page 3-16.

The adsorption filters are housed in this compartment and are attached to the protecting plate with securing clips. One or two adsorption filters may be required according to the instrument configuration. Figure 3-10 shows two adsorption filters installed into the oven compartment.

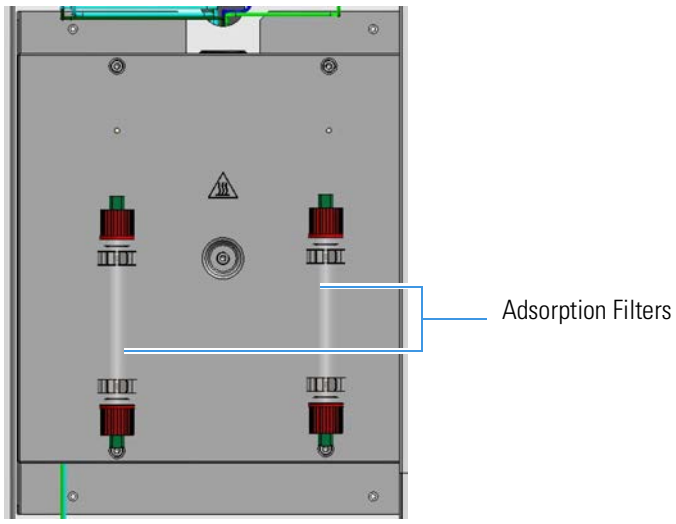



**Figure 3-10.** Adsorption Filters Installed

❖ **To access the thermostatic chamber**

1. Open the right side door, which can be moved 180°.
2. To access the TCD and the GC Columns, first remove the adsorption filters from the fastening clips.
3. Then, remove the four fixing screws on the protecting plate. See Figure 3-11.

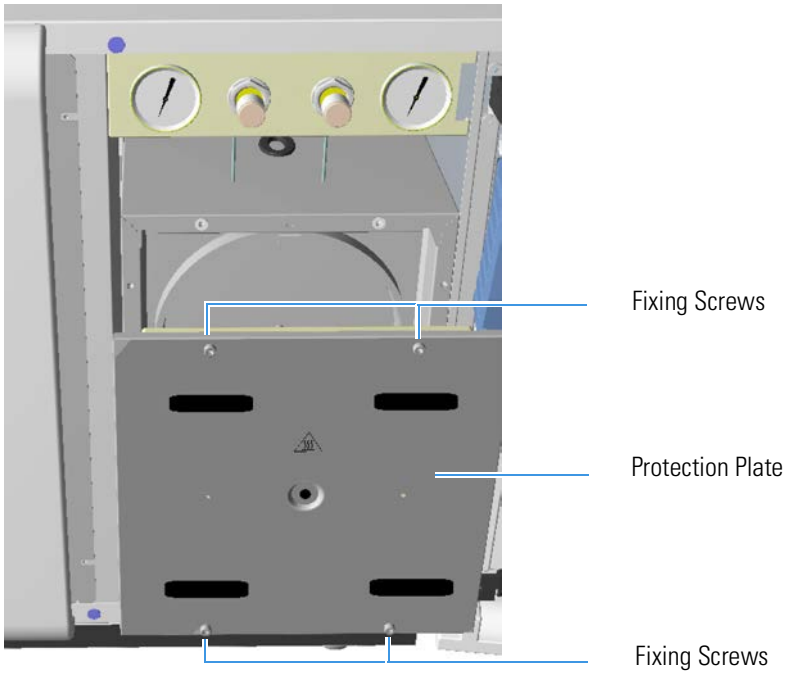



**Figure 3-11.** Access to Thermostatic Chamber

Figure 3-12 shows the detector compartment, the heating block surrounding the thermal conductivity detector (TCD), and the gas chromatographic column.

**Tip** One or two chromatographic columns may be required, according to the instrument configuration.

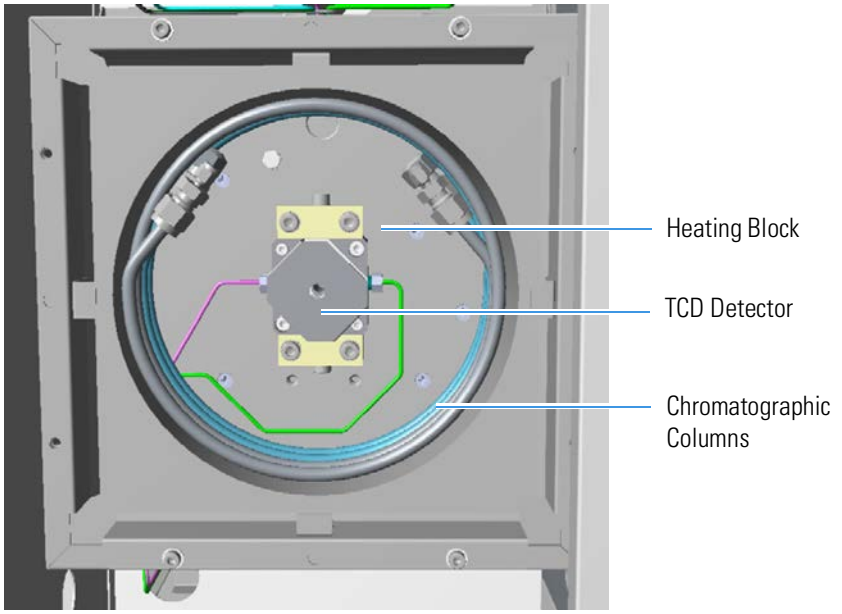



**Figure 3-12.** Thermostatic Chamber Internal View

# Detection System

The detection system consists of a thermal conductivity detector (TCD), which is sensitive to any substance with thermal conductivity other than that of the carrier gas used.

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

The two pairs of filaments are electrically connected according to a Wheatstone bridge circuit powered at constant voltage. The first pair of filaments is fed with pure carrier gas (reference channel). 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, while 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. Because electrical resistance is a function of temperature, the bridge unbalances and the detector generates a signal that is 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.

**Tip** The filaments are powered constantly at 5 V and they are electrically protected if their temperature exceeds 220 °C (Safety Cutoff).

# Electrical Compartment

The electrical compartment is located on the right part of the instrument, and it is accessible by removing the right side cover. Behind the electrical compartment, there is the Connections Panel. For more details, see "Connections Panel" on page 3-22.

**NOTICE**

Do not open the electrical compartment because there are no user serviceable parts inside. Any operation inside the compartment must be carried out by authorized and trained Thermo Fisher Scientific personnel.

The electrical compartment, shown in Figure 3-13, comprises:

- Low voltage compartment
- Main voltage compartment
- EFC-t thermo-regulated electronic flow controller for gas regulation

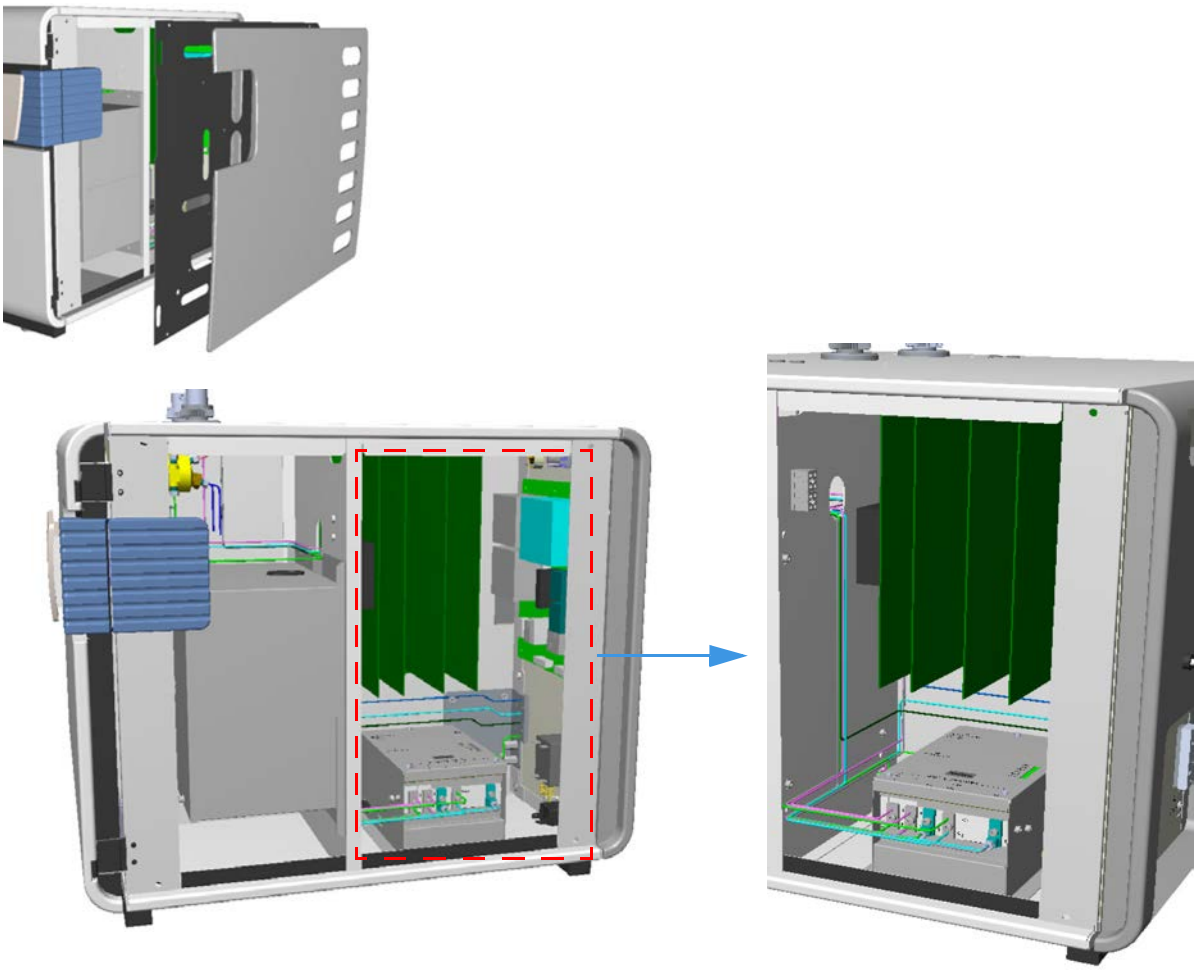


**Figure 3-13.** Electrical Compartment Internal View

## Low Voltage Compartment

The low voltage compartment contains the electronic boards to operate and control the instrument. These boards are interlocked through a mother board. Table 3-4 lists the function of each electronic board present in the low voltage section:

**Table 3-4.** Description of the Function of the Electronic Boards

| Board | Function |
|---|---|
| MB 1112 | Mother board. It provides interlocking between low voltage boards and with the rest of the instrument. This board can be connected to a NiCd 3.6 V; 280 mA/h rechargeable battery located nearby. The rechargeable battery replacement must be performed by specialized technical personnel. |
| CPU 1112 | This board has full control of the instrument operation. It controls the communication between operator and machine through Eager*Smart* Data Handling Software. It actuates the Safety Cutoff device, which puts the instrument in safe conditions, when an alarm condition occurs. |
| HWD 1112 | Provides power supply to the TCD detector filaments. Allows the detector oven thermo-regulation and also amplifies and converts the detector signal to send it to the PC. |
| TCR 1112 | Operates and controls the furnaces thermo regulation and the MAS Plus autosampler movement. |
| PWR 1112 | Receives voltage supplies from the TRF 1112 transformers board. It generates voltage supply for the electronic control circuits. |
| FP 1112 | Synoptic panel |

## Main Voltage Compartment

The main voltage compartment contains the mains power circuits and the Safety Cutoff device. Table 3-5 details the function of each component present in the main voltage section.

**Table 3-5.** Description of the components of the main voltage compartment

| Component | Description |
|---|---|
| TRF 1112 Transformers Board | Receives the mains power and supplies it to the following devices:<br>• Cooling fan<br>• Furnaces transformers<br>• Heater of the detector thermostatic chamber<br><br>Six fuses are provided on the board. See Table 3-6. |
| AC 1112 Furnaces Power Supply | Supplies 48 V ac power to the furnaces. It contains the SSR relays for the furnaces control. Also see "Devices for the Furnaces Control" on page 3-26. Two fuses are provided on the board. See Table 3-6. |
| Breaker | Instrument On/Off main switch. |

**Table 3-6.** Fuses of the main voltage compartment

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

# Connections Panel

The connections panel is shown in Figure 3-14. The connections panel is subdivided into three connecting areas: **interface**, **power supply**, and **gases supply**.

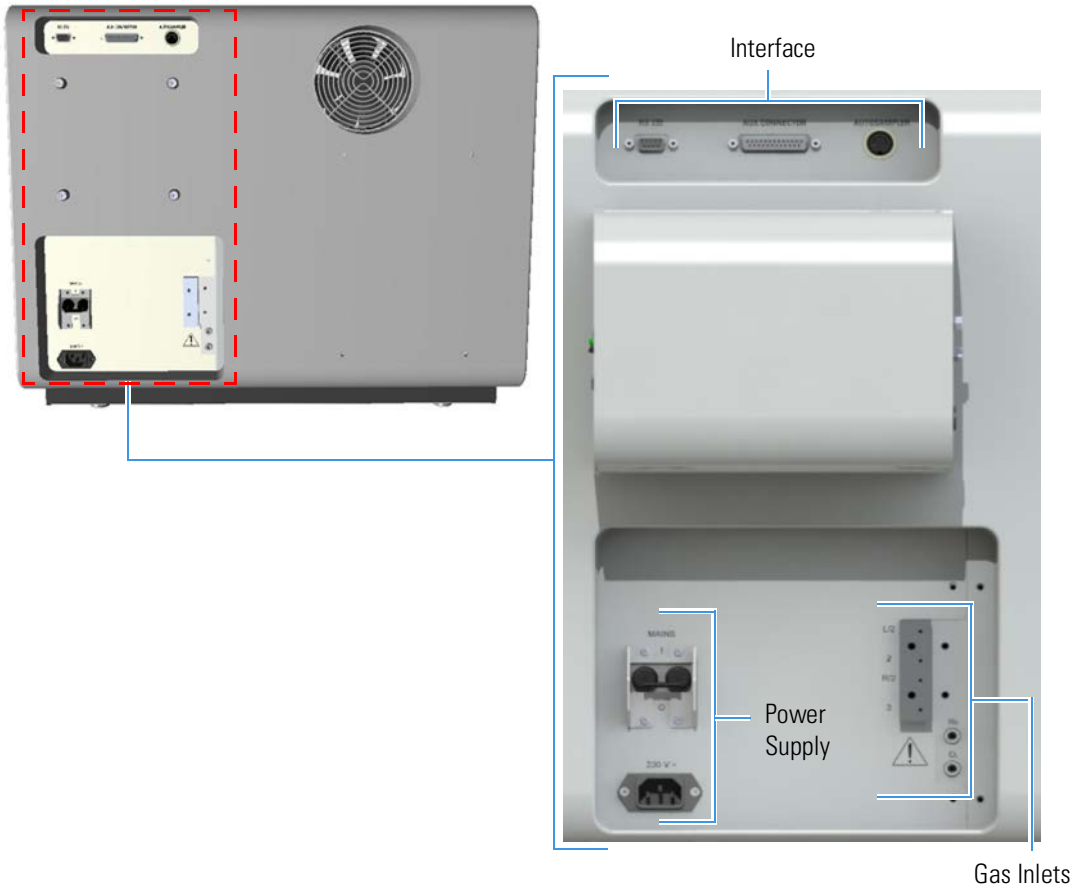



**Figure 3-14.** View of the Connections Panel

## Interface Area

**Figure 3-15.** Interface area

The **interface** area comprises:

- A 9-pin connector labeled **RS 232** to communicate with the computer via serial line.

- A 25-pin connector labeled **Aux Connector** for the remote control of the instrument.
- A 2-pin connector labeled **Autosampler** for the connection of the MAS Plus autosampler for solids.

## Power Supply Area


**Figure 3-16.** Power supply area

The **power supply** area comprises:

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

## Gas Supply Area


**Figure 3-17.** Gases Supply Area

The **gas supply** area comprises the gas inlet ports. See Figure 3-17.

- The **helium (argon)** and **oxygen** gas inlet ports, labeled **He** (**Ar**) and **$O_2$**, are directly connected to the pressure regulators.
- The **gas Connection bypass plate** is present on all the configurations of the Flash*Smart* Elemental Analyzer and must be removed when installing the MultiValve Control (MVC) module.

**Tip** When a Flash*Smart* Elemental Analyzer will be equipped with the optional **MultiValve Control (MVC) Module,** the bypass plate will be removed for allowing the proper connections of the gas lines between the device and the instrument. For details, refer to the *MultiValve Control (MVC) Module for FlashSmart Elemental Analyzer Instruction Manual.*

Table 3-7 details the pressure value to be set for each gas inlet port.

**Table 3-7.** Gas Inlet Ports and Pressure Setting

| Port | Description | Pressure value to be set |
|---|---|---|
| He | Inlet port for helium | 250 kPa (2.5 bar, 36 psig) |
| Ar | Inlet port for argon | 250 kPa (2.5 bar, 36 psig) (in N/Protein configuration)<br>400-450 kPa (4.0-4.5 bar, 26-31 psig) (in NC Soils configuration) |
| $O_2$ | Inlet port for oxygen | 250-300 kPa (2.5-3 bar, 36-44 psig) according to the instrument configuration |

The gas pressures must be set and controlled through the pressure regulators and the pressure gauges of the instrument. Table 3-8 provides indications on the most currently used units of pressure.

**Table 3-8.** Pressure units conversion

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

# Transformer Compartment

The transformer compartment is located in the right bottom part of the instrument. It is accessible firstly by removing the back panel of the instrument, then by removing the relevant protection plate. See Figure 3-18.

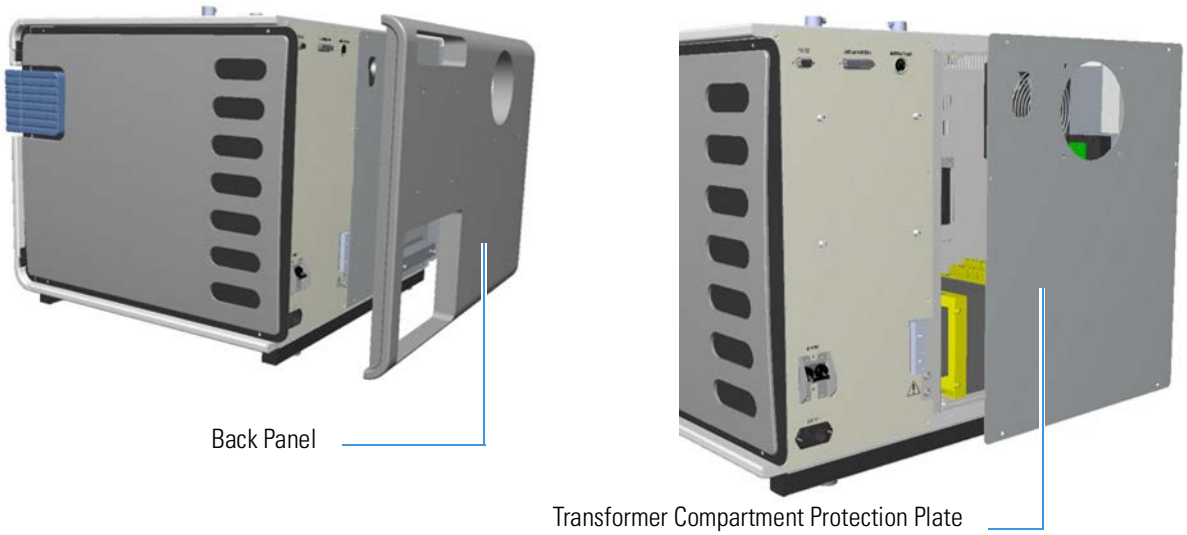



**Figure 3-18.** Accessing the Transformer Compartment

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

**NOTICE**

Do not open the transformer compartment because there are no user serviceable parts inside. Any operation inside the compartment must be carried out only by authorized and trained Thermo Fisher Scientific personnel.

Figure 3-19 shows the devices contained in this compartment.

**Figure 3-19.** Transformer Compartment Internal View

## Devices for the Furnaces Control

Table 3-9 describes the function of each device.

**Table 3-9.** Devices Controlling the Furnaces

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

## Devices that supply the Furnaces

Table 3-10 details the function of each device.

**Table 3-10.** Devices Supplying the Furnaces

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

# LED Status Panel

The LED status panel shows the instrument operating conditions, and it is located on the right side of the instrument front panel. See Figure 3-20.


**Figure 3-20.** LED Status Panel

Each LED lights up when the relevant function is active. Table 3-11 illustrates the meaning of each function:

**Table 3-11.** Status LED Description

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

# Autosamplers

The Flash*Smart* Elemental Analyzer can be configured with these autosamplers:

- **MAS Plus** for solid samples, viscous and liquid samples (weighted in hand tin containers, and
- **AI 1310/AS 1310** for liquid samples.

## MAS Plus Autosampler for Solid Samples

It is the standard autosampler for solid samples mounted directly on the connecting fitting of the concerned channel provided with the proper reactor tube. See Figure 3-21.


**Figure 3-21.** MAS Plus Autosampler for Solid Samples

Its modular structure allows to run up to 125 unattended analyses. The base unit is provided with one 32-position sample tray. It can accommodate three additional 32-position trays to reach a capacity of 125 samples. Each sample tray is installed in a specific position defined by the numbering. Therefore, they are not interchangeable. The sample numbering is detailed in Table 3-12.

**Table 3-12.** Sample Tray Numbering

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

For information about installing the MAS Plus autosampler on your Flash*Smart* EA, see Chapter 6, "Installing the MAS Plus Autosampler."

## AI 1310/AS 1310 Autosampler for Liquid Samples

These are optional autosamplers for the analysis of liquid samples. The are mounted on the Flash*Smart* Elemental Analyzer by means of the appropriate support. See Figure 3-22.

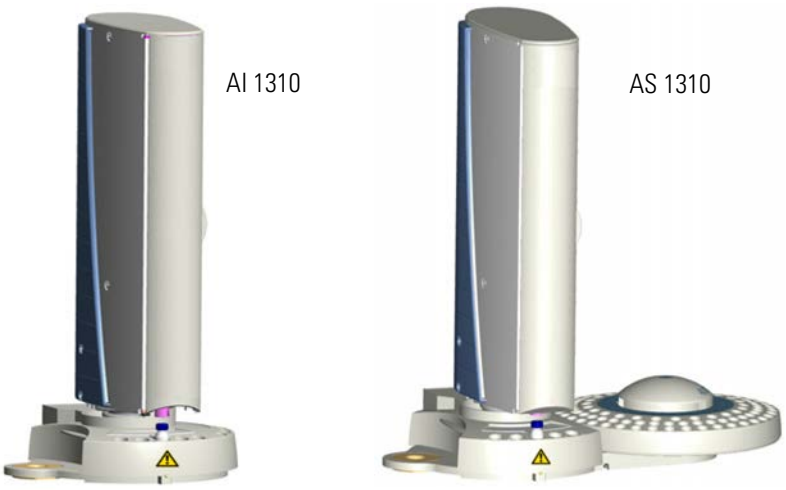



**Figure 3-22.** AI 1310 and AS 1310 Autosamplers for Liquid Samples

The autosampler consists of:

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

For information about installing the AI 1310/AS 1310 autosamplers for liquids on your Flash*Smart* Elemental Analyzer, see Chapter 7, "Installing the AI 1310/AS 1310 Autosampler." For information about the operation with the AI 1310/AS 1310 autosampler, refer to the *AI 1310/AS 1310 for Flash Elemental Analyzers User Guide*.

# MultiValve Control (MVC) Module for the Flash*Smart* EA

The MultiValve Control (MVC) Module is a device used for performing the following functions:

- Switches automatically from the left channel to the right channel.
- Saves helium or argon by switching from helium or argon, used as carrier gas for the analysis, to argon or nitrogen when the instrument is not used overnight, or on weekends, or for a prolonged period of time.

Figure 3-23 shows the MultiValve Control (MVC) Module installed on the back panel of the instrument.


**Figure 3-23.** MultiValve Control (MVC) Module

**Tip** Two MAS Plus autosamplers for solid samples are required for operation with the MultiValve Control (MVC) Module.

For information about installing, connecting, and operating with the MultiValve Control (MVC) Module, refer to the *MultiValve Control (MVC) Module for FlashSmart Elemental Analyzer Instruction Manual*.

4

# Analytical Principles

This chapter describes the analytical techniques with the correlated pneumatic circuits used for all the configurations of the Flash*Smart* Elemental Analyzer.

**Contents**



For the MVC CHN/O, MVC CHNS/O, MVC CHN/CHN and MVC CHNS/CHNS configuration with the MultiValve Control (MCV) Module, refer to *the MultiValve Control (MVC) Module for FlashSmart Elemental Analyzer Instruction Manual*.

# System Overview

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

All pneumatic circuits have the following common components:

- Two inlet gases pressure regulators and relevant pressure gauges. See "Pressure Regulators" on page 4-2.
- An Electronic Flow Controller (EFC-t) for gases. See "Electronic Flow Controller (EFC-t) Module" on page 4-3.
- A Thermal Conductivity Detector (TCD).

Depending on 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.

## Pressure Regulators

The pressure regulators allow the manual adjustment of the carrier gas (helium or argon) and the oxygen inlet pressures. The regulators are located in the detector compartment and they are schematically shown in Figure 4-1.

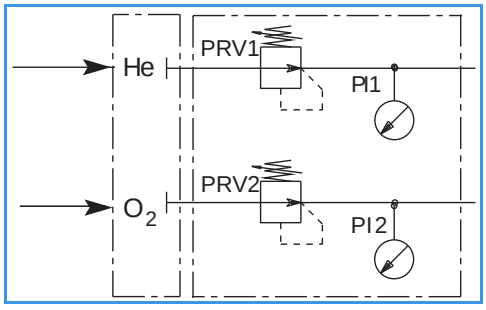



**Figure 4-1.** Pressure Regulators

The pressure regulators are common in all the configurations of the Flash*Smart* Elemental Analyzer. The regulators consist of the following components. See Table 4-1.

**Table 4-1.** Pressure regulators

| Component | Description and function |
|---|---|
| He (Ar) | Helium (argon) inlet port |
| O2 | Oxygen inlet port |
| PRV1 | Helium pressure regulator |
| PI1 | Helium pressure gauge |
| PRV2 | Oxygen pressure regulator |
| PI2 | Oxygen pressure gauge |

## Electronic Flow Controller (EFC-t) Module

The EFC-t module is present in all configurations of the Flash*Smart* Elemental Analyzer. There are two different EFC-t modules: one for helium carrier gas or one for argon carrier gas. Both have the same pneumatic but different electronics, they cannot be exchanged. The module is schematically shown in Figure 4-2.

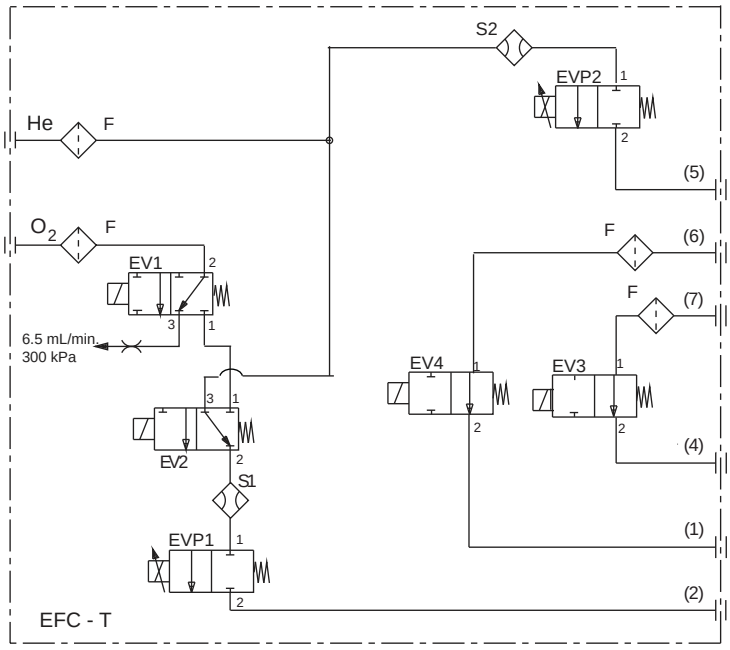



**Figure 4-2.** Thermo-regulated EFC-t Module for helium carrier gas

**Table 4-2.** Parts of the EFC-t Module

| Component | Description and function |
|---|---|
| He (Ar) | Helium (argon) inlet port |
| O2 | Oxygen inlet port |
| F | Stainless steel filter |

**Table 4-2.** Parts of the EFC-t Module, continued

| Component | Description and function |
|---|---|
| EV1 | Two-way solenoid valve to control oxygen inlet. |
| EV2 | Three-way solenoid valve to control helium (argon) inlet and to allow switching between helium (argon) and oxygen. |
| EV3 | Two-way solenoid valve, normally open, to control the inlet of helium (argon) 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 (argon) flowing back from the TCD detector reference channel. The gas is used to eliminate air from the MAS Plus autosampler. The valve is closed during the leak test. |
| S1 | Electronic flow sensor for helium (argon) as carrier gas and oxygen during the sampling stage. It cooperates with the EVP1 electronic controller (proportional valve). |
| S2 | Electronic flow sensor for helium (argon) as reference gas. It cooperates with the EVP2 electronic controller (proportional valve). |
| EVP1 | Electronic flow controller for helium (argon) as carrier gas and oxygen to control the flow rates of gases according to the flow values set. |
| EVP2 | Electronic flow controller for helium (argon) as reference gas to control the flow rate according to the required flow value. |

The analytical method used in each instrument configuration comprises various steps. They lead to the determination of the weight percent composition of the components of interest through the transformation of the solid or liquid sample into gas.

**Tip** To develop and perform the analytical cycle, see the Chapter 13, "Running Analyses."
For correct sample analyses, all pneumatic lines must be leak-free. Therefore, a preliminary leak check is recommended before you start the analytical cycles.

**Tip** All the pneumatic diagrams in this chapter are shown in the **Pre-analysis** stage.

# Analytical Principle for CHN Configuration

An autosampler **AS** is connected to a quartz reactor **R1** housed in an furnace at a temperature of **950 °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**.

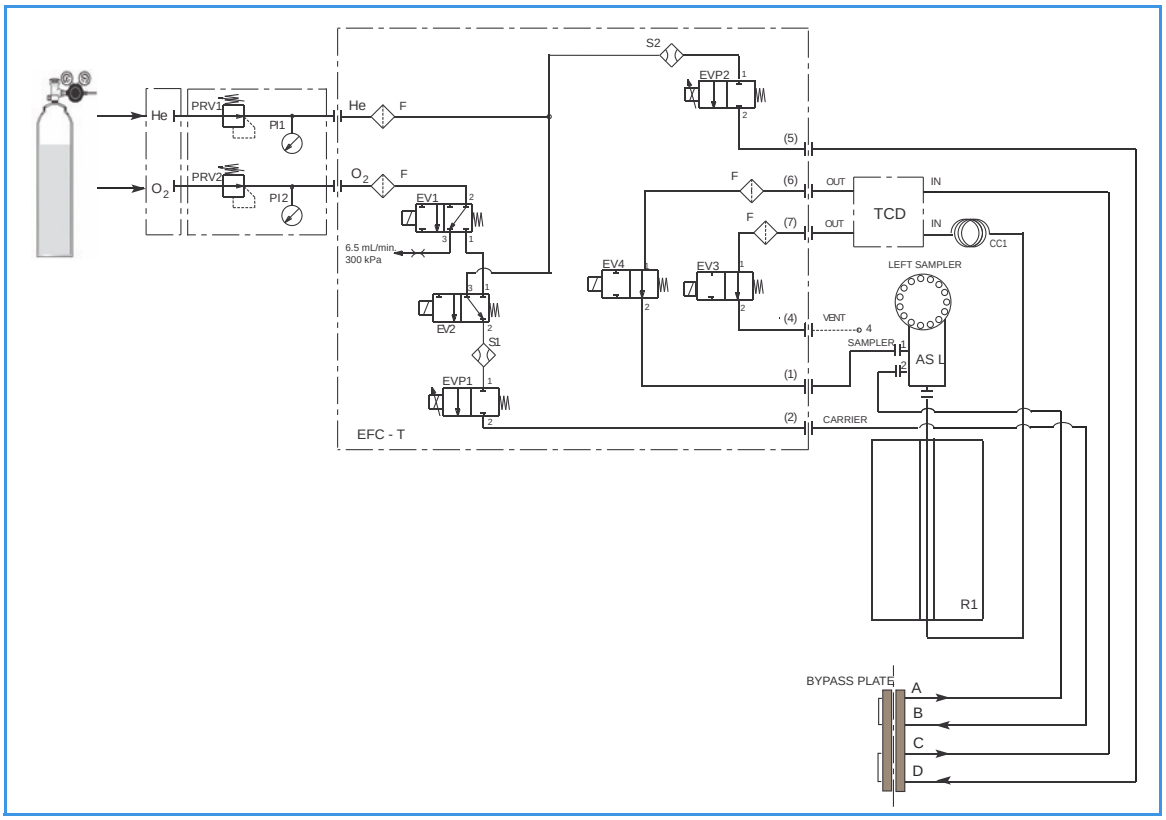



**Figure 4-3.** Instrument Parts Diagram and Pneumatic Diagram for CHN Configuration

**Table 4-3.** Components of the Pneumatic Circuit for CHN Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Reactor |
| F1 | Adsorption filter |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

### 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 $\mathbf{O_2}$ is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

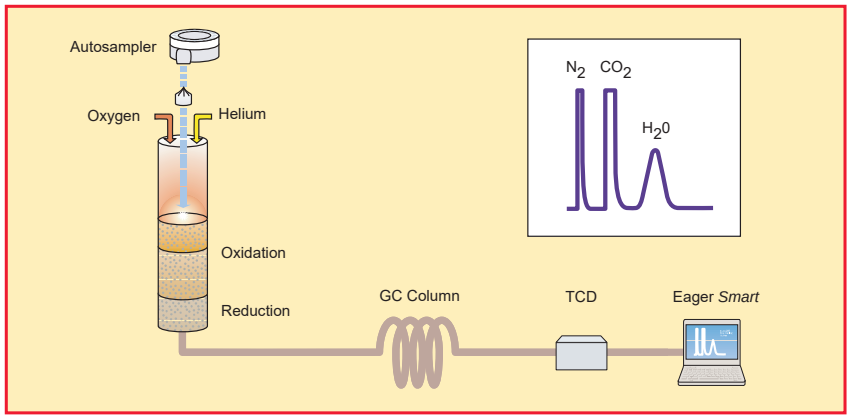



**Figure 4-4.** Schematic of CHN Configuration

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** switches from point 2-3 to point 2-1, **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.

**Tip** Sulfur and halogenated compounds (chlorine, bromine, and so on), possibly present in the sample, do not affect the analysis, because the silvered cobaltous/cobaltic oxide catalyst holds back both $SO_2$ and halogens.

Then the gas mixture ($N_2$, $CO_2$ and $H_2O$) flows into the chromatographic column **CC1**, where separation takes place. The eluted gases are conveyed to the thermal conductivity detector **TCD**. The electrical signals generated by the detector are properly processed by Eager*Smart* software providing nitrogen, carbon, and hydrogen percentages.

# Analytical Principle for CHN/O Configuration

**CHN Determination** — An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at a temperature of **950 °C**. This reactor is connected to the analytical column **CC1**, on its turn connected to a channel of the thermal conductivity detector **TCD**.

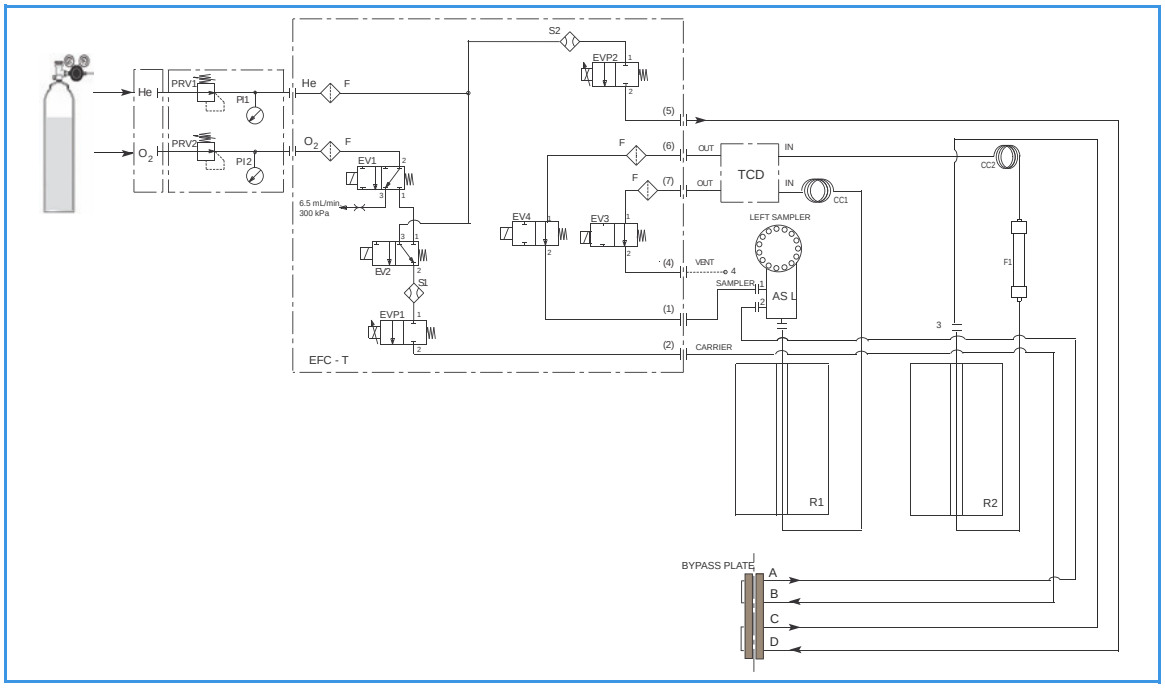



**Figure 4-5.** Instrument Parts Diagram and Pneumatic Diagram for CHN/O Configuration with One Sampler

**Oxygen Determination** — A second autosampler **AS** could be connected to a reactor **R2** placed in an 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**.

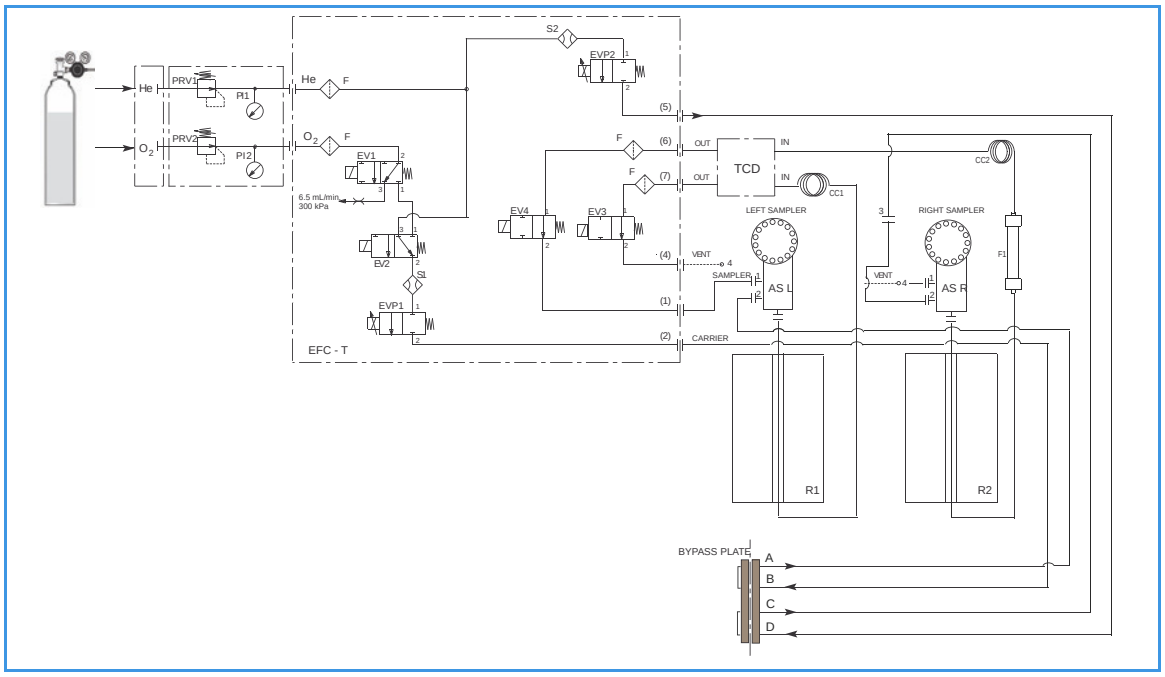



**Figure 4-6.** Instrument Parts Diagram and Pneumatic Diagram for CHN/O Configuration with Two Samplers

**Table 4-4.** Components of the Pneumatic Circuit for CHN-O Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| 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 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.

**Tip** 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 vice-versa. The oxygen line $\mathbf{O_2}$ is connected to the solenoid valve **EV1**.This valve controls the oxygen inlet.

### Sequence of the Method Stages

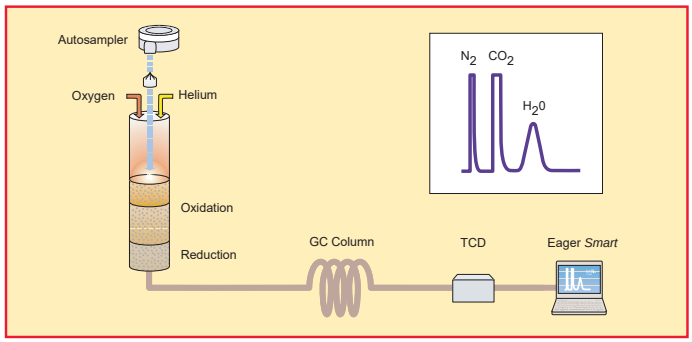



**Figure 4-7.** Schematic of CHN/O Configuration (CHN Determination)

**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** switches from point 2-3 to point 2-1, 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.

**Tip** Sulfur and halogenated compounds (chlorine, bromine, and so on), possibly present in the sample, do not affect the analysis, because the silvered cobaltous/cobaltic oxide catalyst holds back both $SO_2$ and halogens.

Next, the gas mixture ($N_2$, $CO_2$ and $H_2O$) flows into the chromatographic column **CC1**, where separation takes place. The eluted gases are conveyed to the thermal conductivity detector **TCD**. The electrical signals generated by the detector are properly processed by Eager*Smart* software providing nitrogen, carbon, and hydrogen percentages.

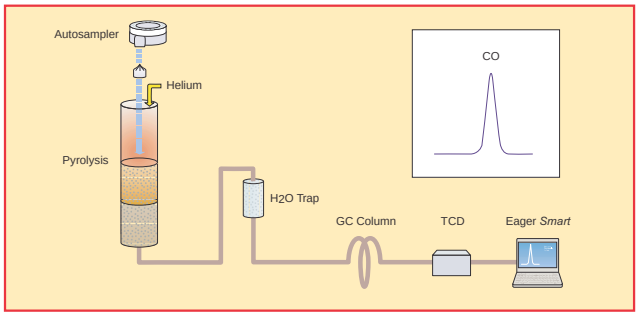



**Figure 4-8.** Schematic of CHN/O Configuration (O Determination)

**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, $N_2$, CO, and $H_2$ form. The pyrolysis products cross the adsorption filter **F** where halogenated compounds (chlorine, bromine, and so on) are retained.

The gas mixture flows into the chromatographic column **CC2**, where carbon monoxide is separated from other gases. Next, the eluted gases are conveyed to the thermal conductivity detector **TCD**.

The electrical signals generated by the detector are properly processed by Eager*Smart* software providing oxygen percentage.

# Analytical Principle for CHNS Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at a temperature of **950 °C**. This reactor is connected to the analytical column **CC**, on its turn connected to a channel of the thermal conductivity detector **TCD**.

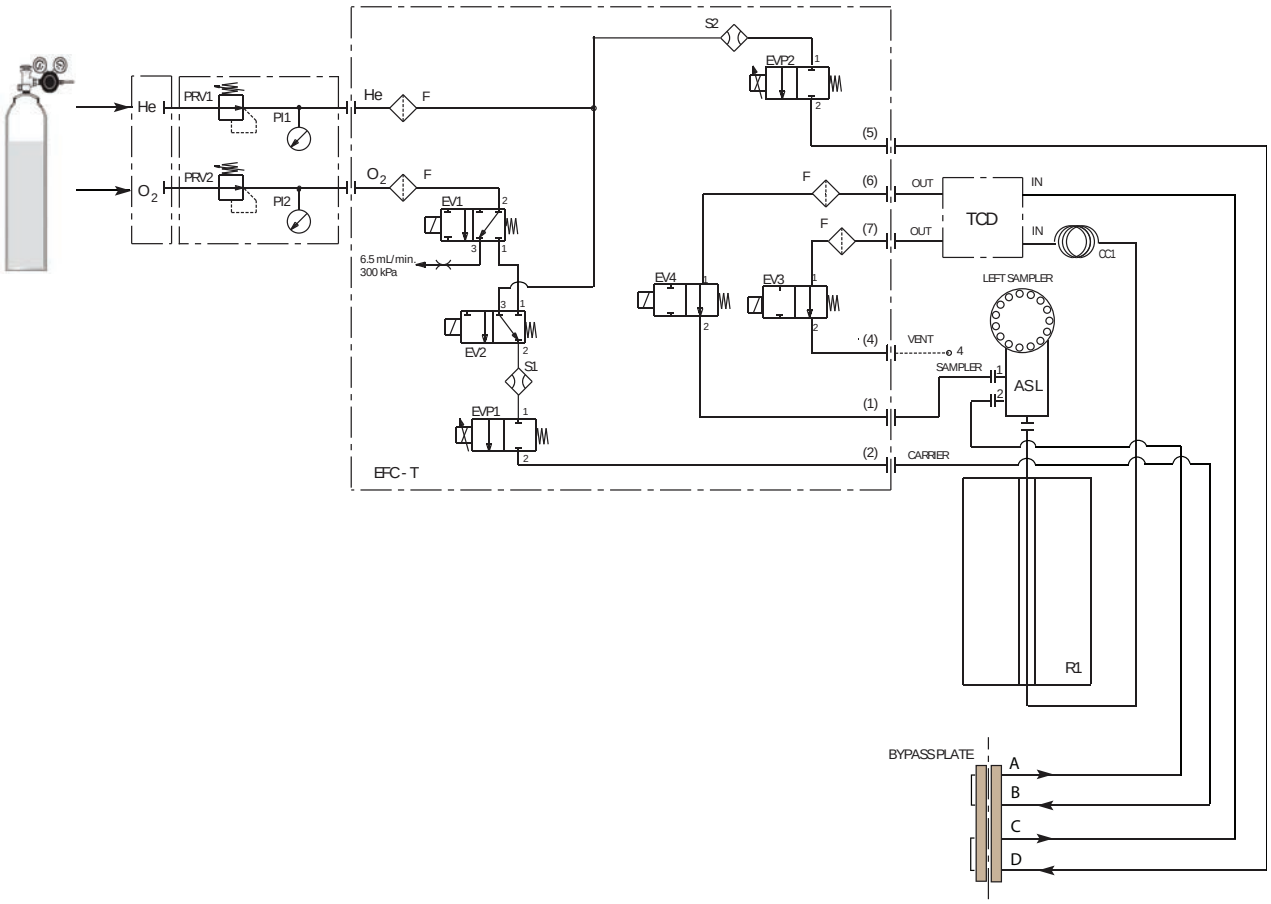



**Figure 4-9.** Instrument Parts Diagram and Pneumatic Diagram for CHNS Configuration

**Table 4-5.** Components of the Pneumatic Circuit for CHNS Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Reactor |
| F1 | Adsorption filter |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

### 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 **$O_2$** is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

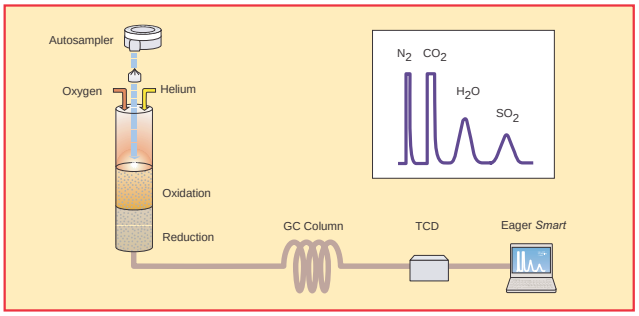



**Figure 4-10.** Schematic of CHNS Configuration

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** switches from point 2-3 to point 2-1, 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.

Next, the gas mixture ($N_2$, $CO_2$, $H_2O$, and $SO_2$) flows into the chromatographic column **CC1** where separation takes place. The eluted gases are sent to the thermal conductivity detector **TCD**.
The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the percentages of nitrogen, carbon, hydrogen, and sulfur contained in the sample.

# Analytical Principle for CHNS/O Configuration

**CHNS Determination —** An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of **950 °C**. This reactor is connected to the analytical column **CC1**, on its turn connected to a channel of the thermal conductivity detector **TCD**.

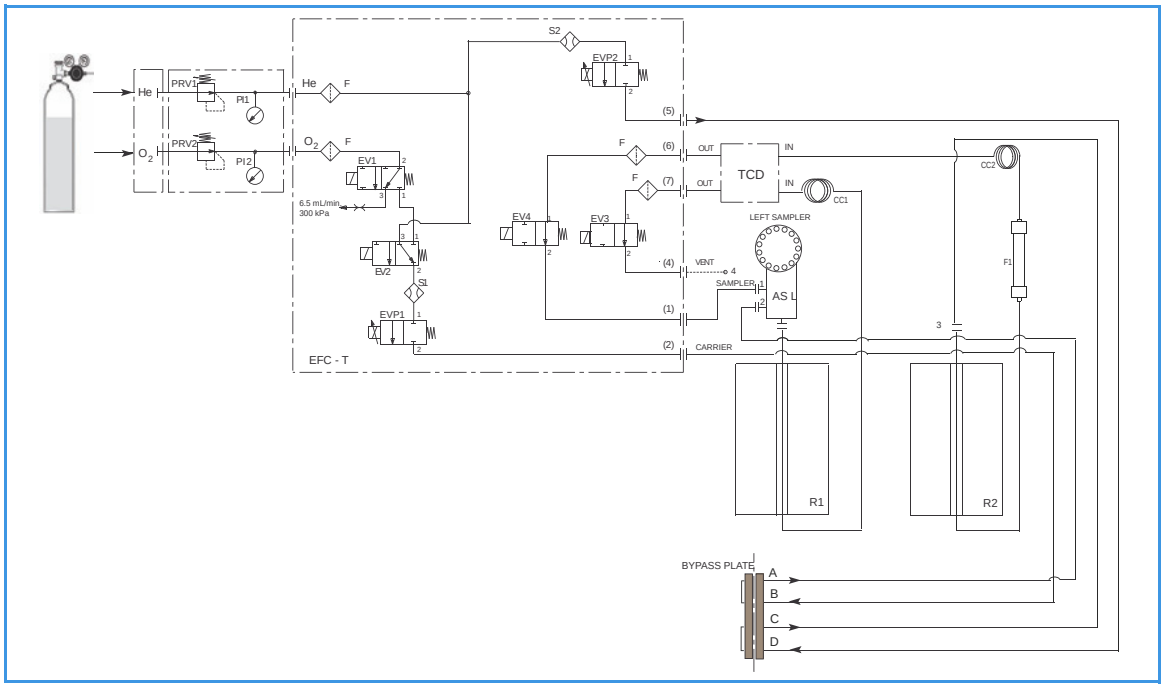



**Figure 4-11.** Instrument Parts Diagram and Pneumatic Diagram for CHNS/O Configuration with One Sampler

**Oxygen Determination —** A second autosampler **AS** could be 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**.

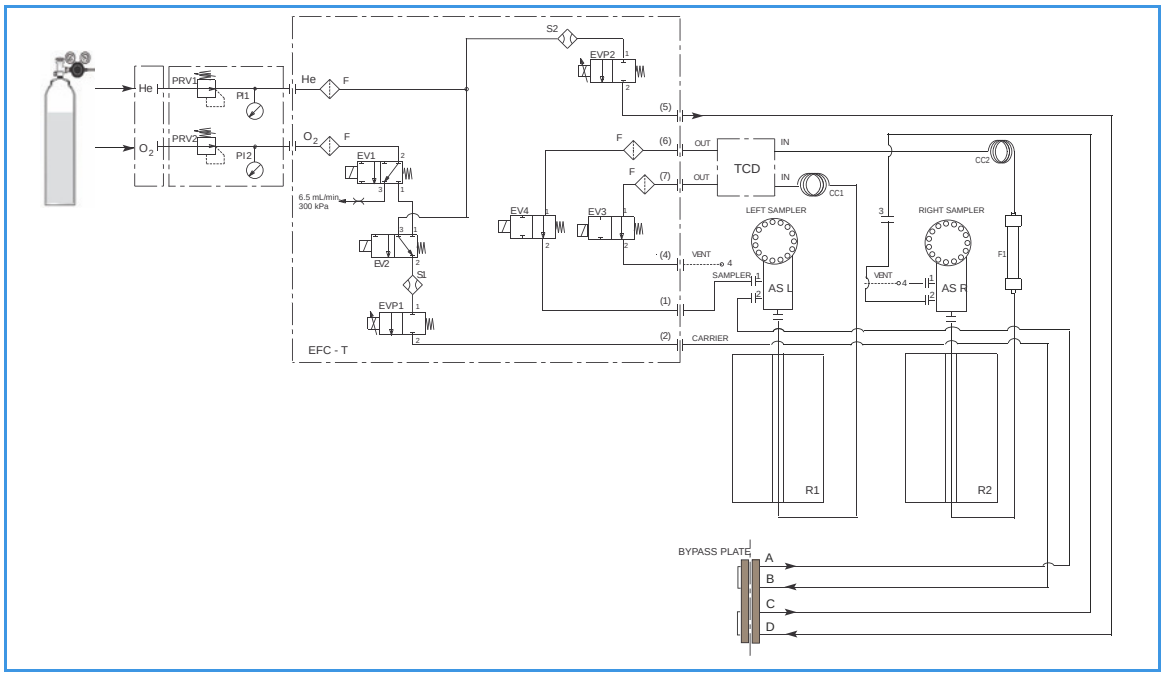



**Figure 4-12.** Instrument Parts Diagram and Pneumatic Diagram for CHNS/O Configuration with Two Samplers

**Table 4-6.** Components of the Pneumatic Circuit for CHNS/O Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| 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 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.

**Tip** 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 $\mathbf{O_2}$ is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

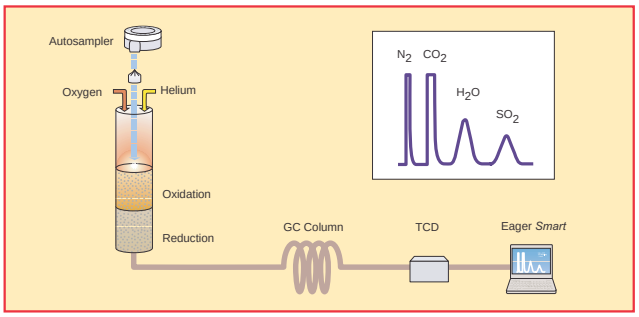



**Figure 4-13.** Schematic of CHNS/O Configuration (CHNS Determination)

**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** switches from point 2-3 to point 2-1, 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 ($N_2$, $CO_2$, $H_2O$ and $SO_2$) flows into the gas chromatographic column **CC1** where separation occurs.

The eluted gases are conveyed to the thermal conductivity detector **TCD**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the nitrogen, carbon, hydrogen, and sulfur percentages contained in the sample.

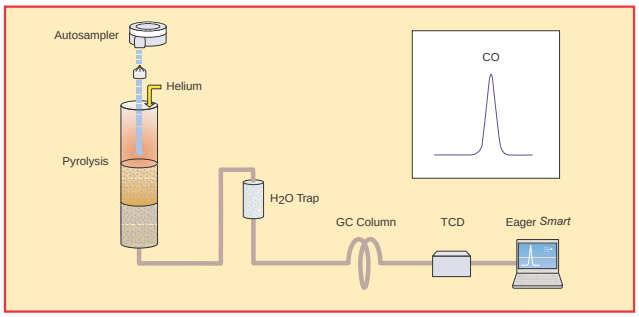



**Figure 4-14.** Schematic of CHNS/O Configuration (O Determination)

**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, $N_2$, CO, and $H_2$ form. The pyrolysis products cross the adsorption filter **F** where halogenated compounds (chlorine, bromine, and so on) are retained.

The gas mixture flows into the chromatographic columns **CC2** where carbon monoxide is separated from the other gases. Next, the eluted gases are conveyed to the thermal conductivity detector **TCD**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software provides the oxygen percentage.

# Analytical Principle for S (Sulfur) Configuration

An autosampler **AS** is connected to a reactor **R1** placed in an furnace at the temperature of **950 °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**.

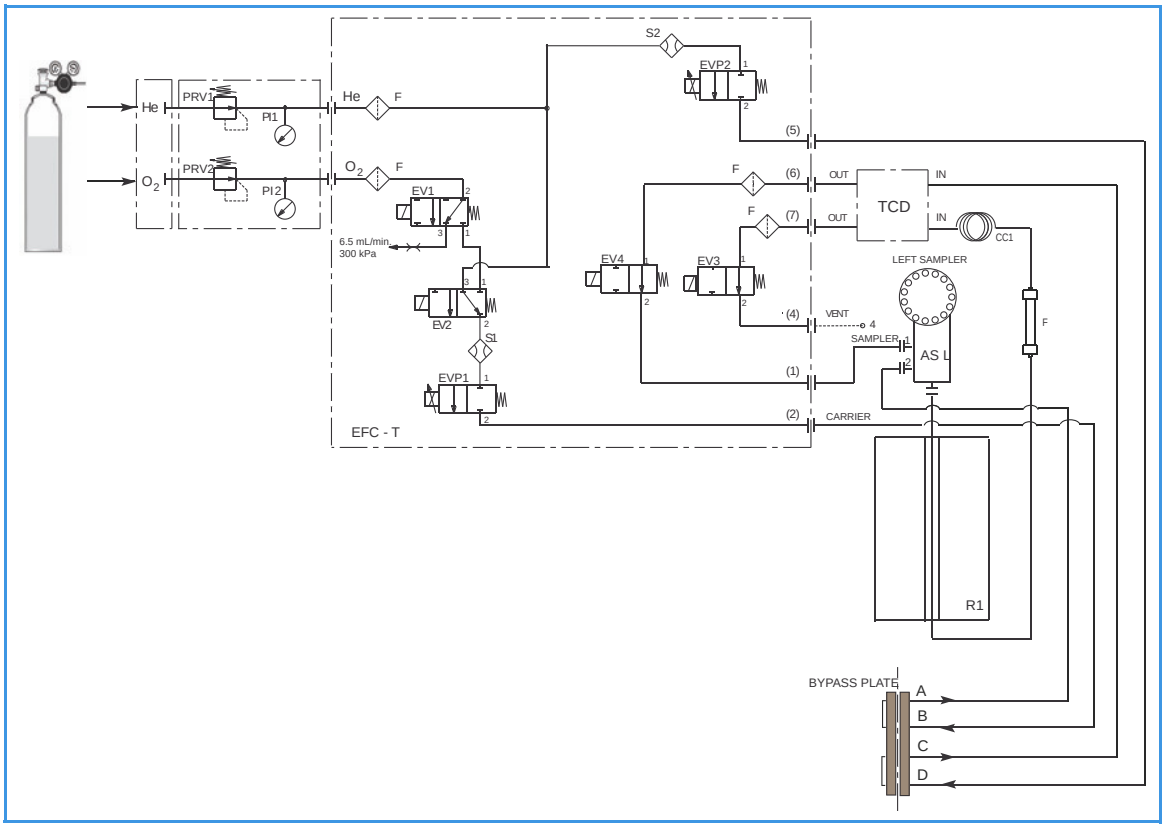



**Figure 4-15.** Instrument Parts Diagram and Pneumatic Diagram for S (Sulfur) Configuration

**Table 4-7.** Components of the Pneumatic circuit for S (Sulfur) Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Reactor |
| F1 | Adsorption filter |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

### 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 $\mathbf{O_2}$ is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

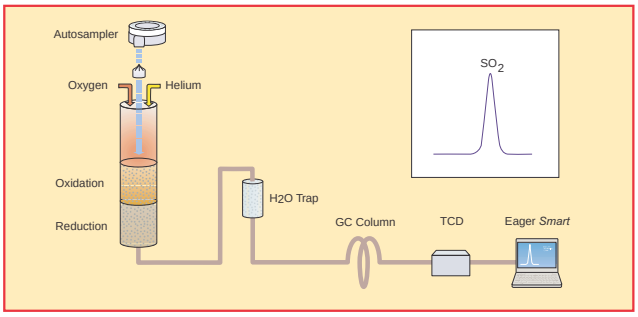



**Figure 4-16.** Schematic of S Configuration

During **pre-analysi**s 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** switch from point 2-3 to point 2-1, 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.

Next, the gas mixture ($N_2$, $CO_2$, $H_2O$ and $SO_2$) 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**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the sulfur percentage.

# Analytical Principle 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**.

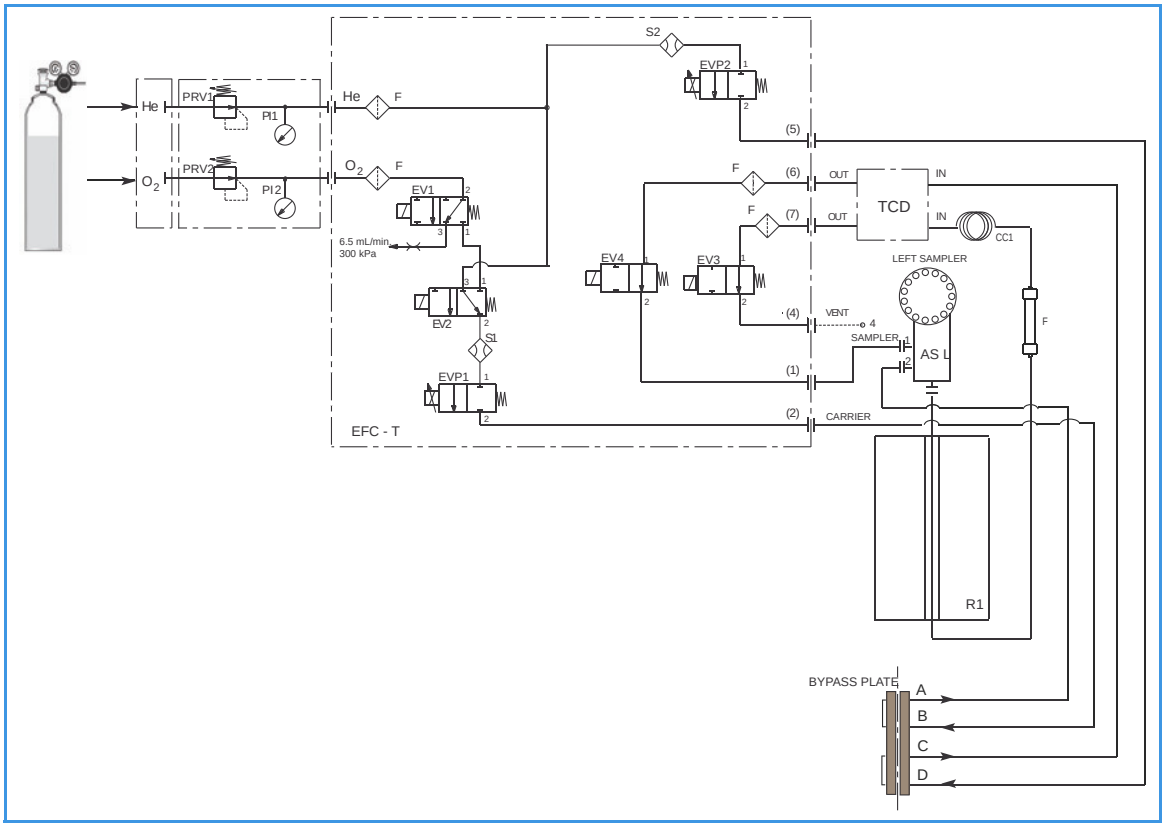



**Figure 4-17.** Instrument Parts Diagram and Pneumatic Diagram for O (Oxygen) Configuration

**Table 4-8.** Components of the Pneumatic Circuit for O (Oxygen) Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Reactor |
| F1 | Adsorption filter |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

### 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 **$O_2$** is connected to the solenoid valve **EV1**.

### Sequence of the Method Stages

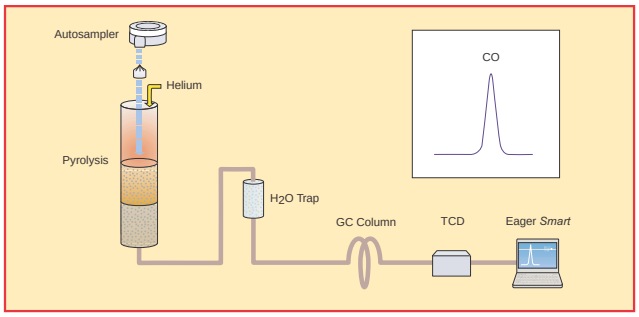



**Figure 4-18.** Schematic of O Configuration

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 **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, and so on) are retained. The gas mixture flows into the chromatographic column **CC** where carbon monoxide is separated from the other gases.

Next, the eluted gases are conveyed to the thermal conductivity detector **TCD**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the oxygen percentage.

# Analytical Principle for N (Nitrogen) Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of **950 °C**. This reactor, on its turn, is connected in series to a second reactor **R2** placed in an furnace at the temperature of **840 °C**. To the reactor **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**.

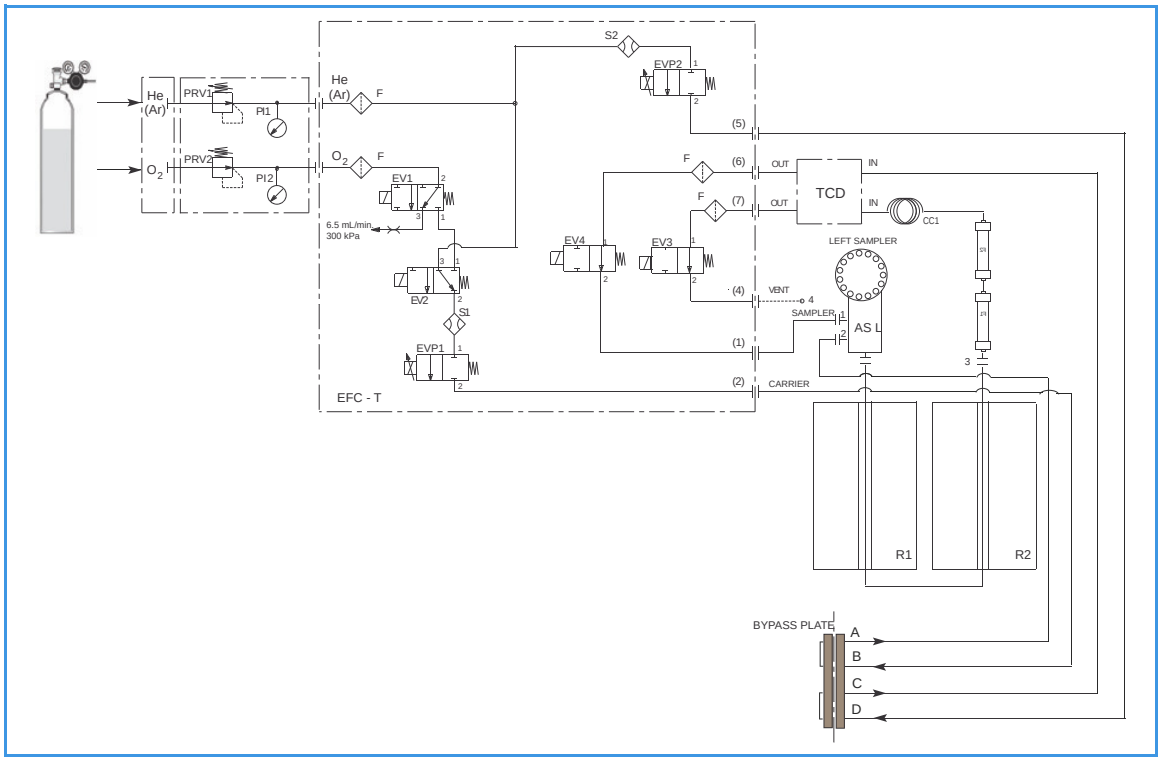



**Figure 4-19.** Instrument Parts Diagram and Pneumatic Diagram for N (Nitrogen) Configuration

**Table 4-9.** Components of the Pneumatic Circuit for N (Nitrogen) Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Oxidation reactor |
| R2 | Reduction reactor |
| F1 | Adsorption filter for carbon dioxide |
| F2 | Adsorption filter for water |

**Table 4-9.** Components of the Pneumatic Circuit for N (Nitrogen) Determinations, continued

| Component | Description |
|---|---|
| CC | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

**Tip** The pressure stabilizing cylinder is not present in the N configuration.

### 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 $\mathbf{O_2}$ is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

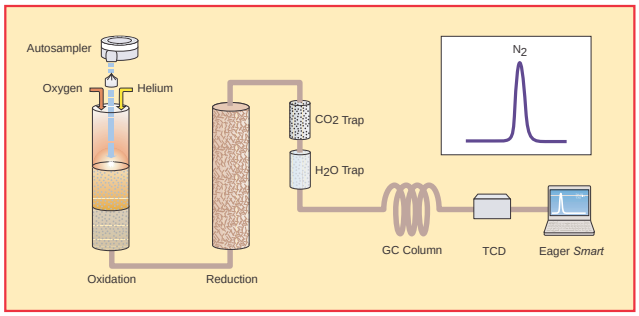



**Figure 4-20.** Schematic of N Configuration

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** switches from point 2-3 to point 2-1, 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. The 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 ($N_2$, $CO_2$, $H_2O$ and $SO_2$) generated by combustion is conveyed across the reactor **R1** where the oxidation of components is completed. Next, the mixture crosses the reactor **R2** where nitrogen oxides possibly formed are converted into elemental nitrogen, and the oxygen excess is retained.

Next, the gas mixture passes across the two adsorption filters **F1** and **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**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the nitrogen percentage.

# Analytical Principle for NC Configuration

An autosampler **AS** is connected to a quartz reactor **R1** placed in an furnace at the temperature of **950 °C**. This reactor, on its turn, is connected in series to a second reactor **R2** placed in an furnace at the temperature of **840 °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**.

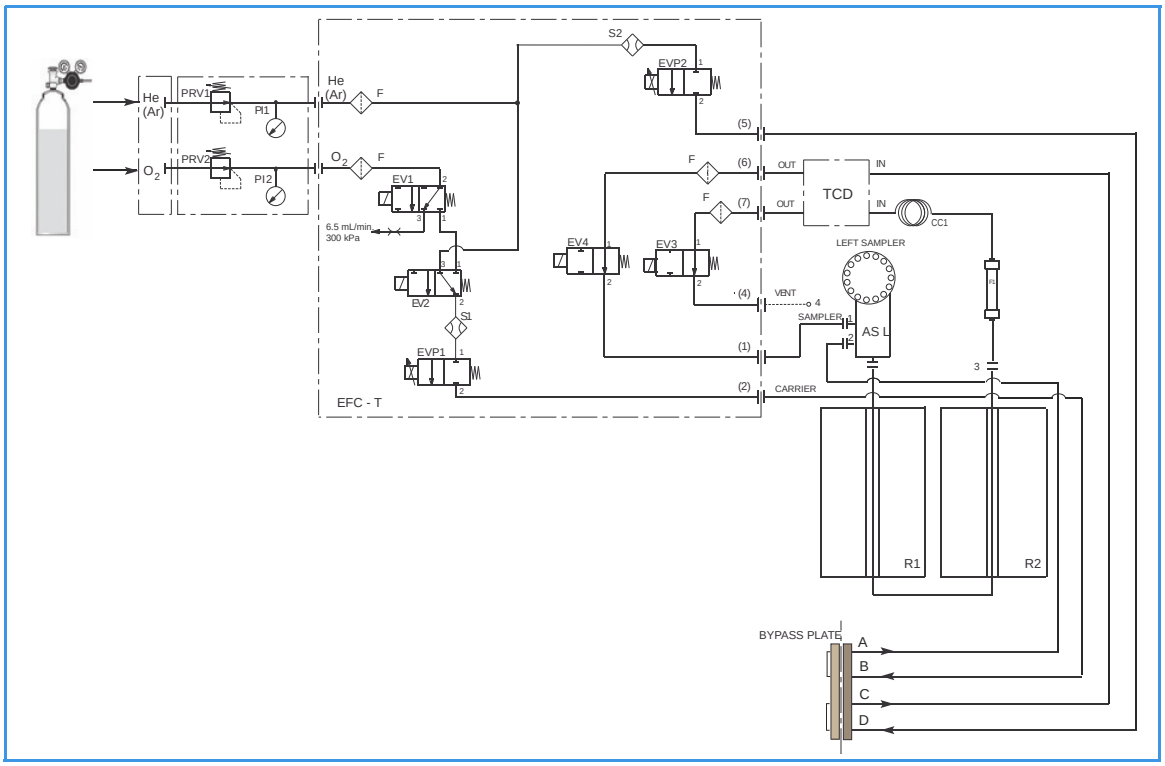



**Figure 4-21.** Instrument Parts Diagram and Pneumatic Diagram for NC Configuration

**Table 4-10.** Components of the Pneumatic Circuit for NC Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Oxidation reactor |
| R2 | Reduction reactor |
| F1 | Adsorption filter for water |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

### 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 **$O_2$** is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

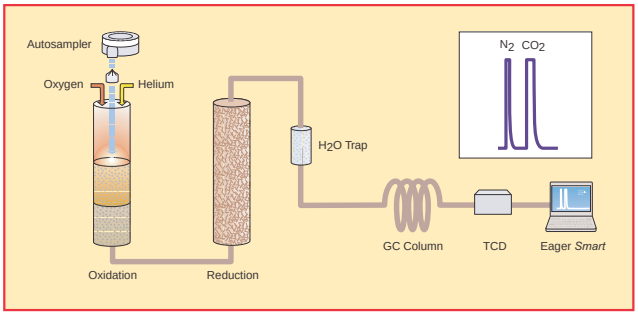



**Figure 4-22.** Schematic of NC Configuration

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** switches from point 2-3 to point 2-1, 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 ($N_2$, $CO_2$, $H_2O$ and $SO_2$) 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.

**Tip** Sulfur and halogenated compounds (chlorine, bromine, and so on), possibly present in the sample, do not affect analysis, because the silver-plated cobalt oxide catalyst holds back both $SO_2$ and halogens.

Next, the gas mixture 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**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the nitrogen and carbon percentages.

# Analytical Principle for NCS Configuration

An autosampler **AS** is connected to a reactor **R1** placed in an furnace at the temperature of **950 °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**.

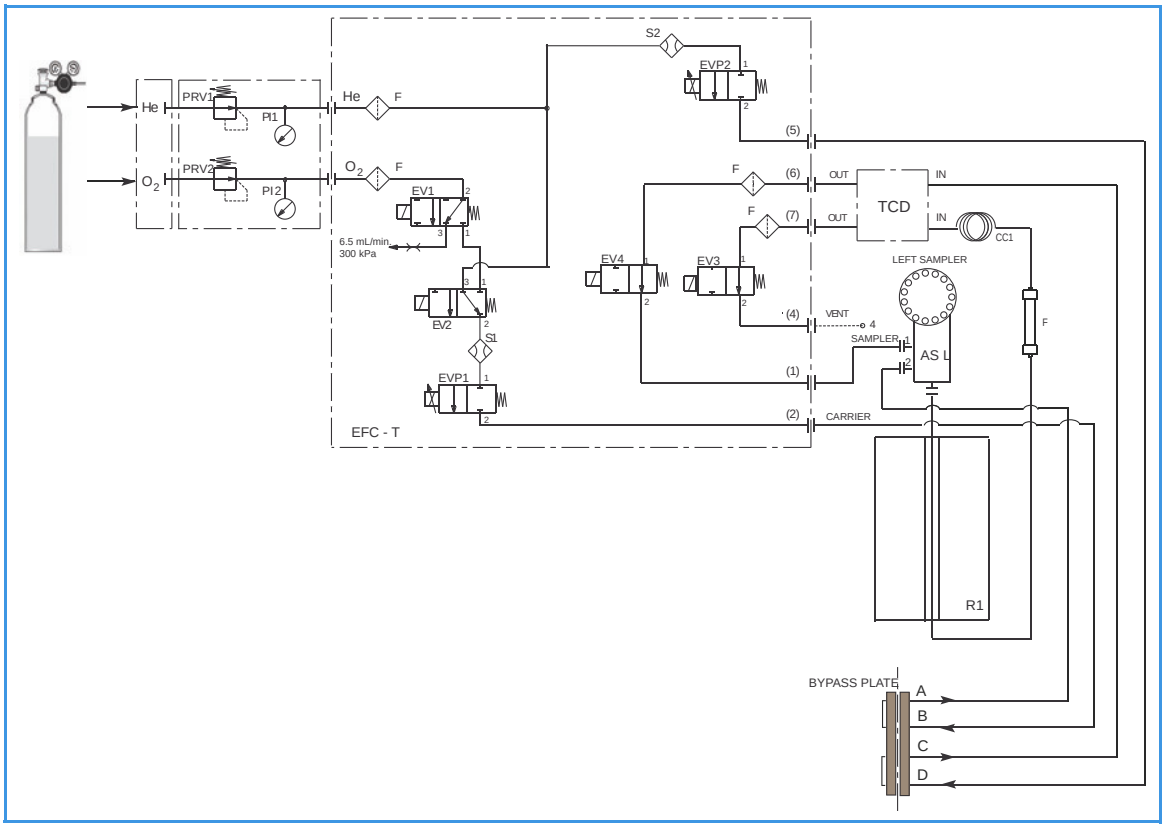



**Figure 4-23.** Instrument Parts Diagram and Pneumatic Diagram for NCS Configuration

**Table 4-11.** Components of the Pneumatic Circuit for NCS Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Reactor |
| F1 | Adsorption filter |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

## 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 **$O_2$** is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

## Sequence of the Method Stages

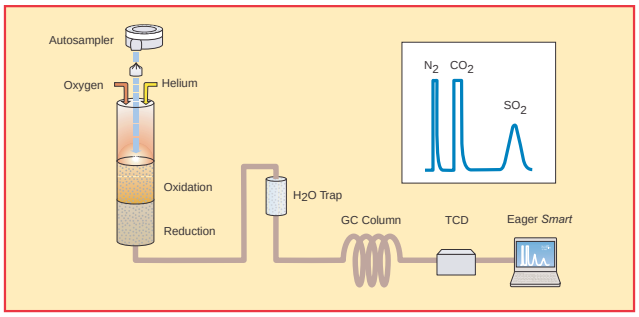



**Figure 4-24.** Schematic of NCS Configuration

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** switches from point 2-3 to point 2-1, 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 ($N_2$, $CO_2$, $H_2O$ and $SO_2$) 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.

Next, the gas mixture ($N_2$, $CO_2$, $H_2O$ and $SO_2$) 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**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing nitrogen, carbon, and sulfur percentages.

# Analytical Principle for NC Soils 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 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**.

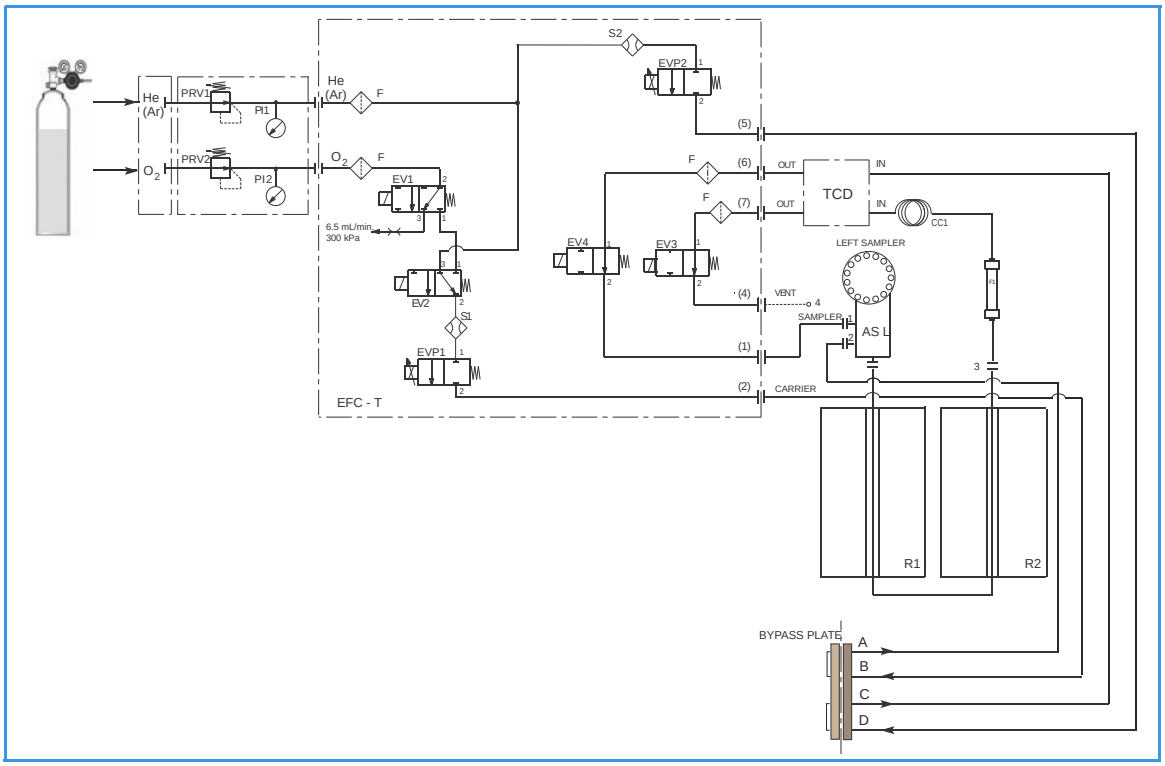



**Figure 4-25.** Instrument Parts Diagram and Pneumatic Diagram for NC-Soils Configurations

**Table 4-12.** Components of the Pneumatic Circuit for NC Soils

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Oxidation reactor |
| R2 | Reduction reactor |
| F1 | Adsorption filter for water |
| CC1 | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |

### Pneumatic Diagram Description

Helium **He** (argon **Ar**) 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 (argon) flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts helium (argon) to the atmosphere through **Vent 4**.
The proportional valve **EVP2**, connected to the detector reference channel **TCD**, controls the helium (argon) flow as far as the solenoid valve **EV4**. This valve, normally open, allows helium (argon) to reach point 1 of the autosampler and purge the zone where the sample is housed. The oxygen line **$O_2$** is connected to the solenoid valve **EV1**. This valve controls the oxygen inlet.

### Sequence of the Method Stages

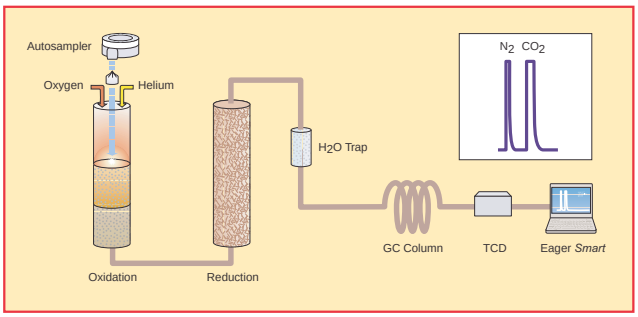



**Figure 4-26.** Schematic of NC Soils Configuration

During **pre-analysis**, the solenoid valve **EV1** shuts off oxygen, whereas the solenoid valve **EV2** allows helium (argon) to flow in the circuit.

When **Start Analysis** is pressed, the valve **EV1** switches from point 2-3 to point 2-1, 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 (argon) flow.

The gas mixture ($N_2$, $CO_2$, $H_2O$ and $SO_2$) 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. Next, 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**. The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing nitrogen and carbon percentages.

# Analytical Principle for N Lubricant, N Brew and N/Protein 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**.

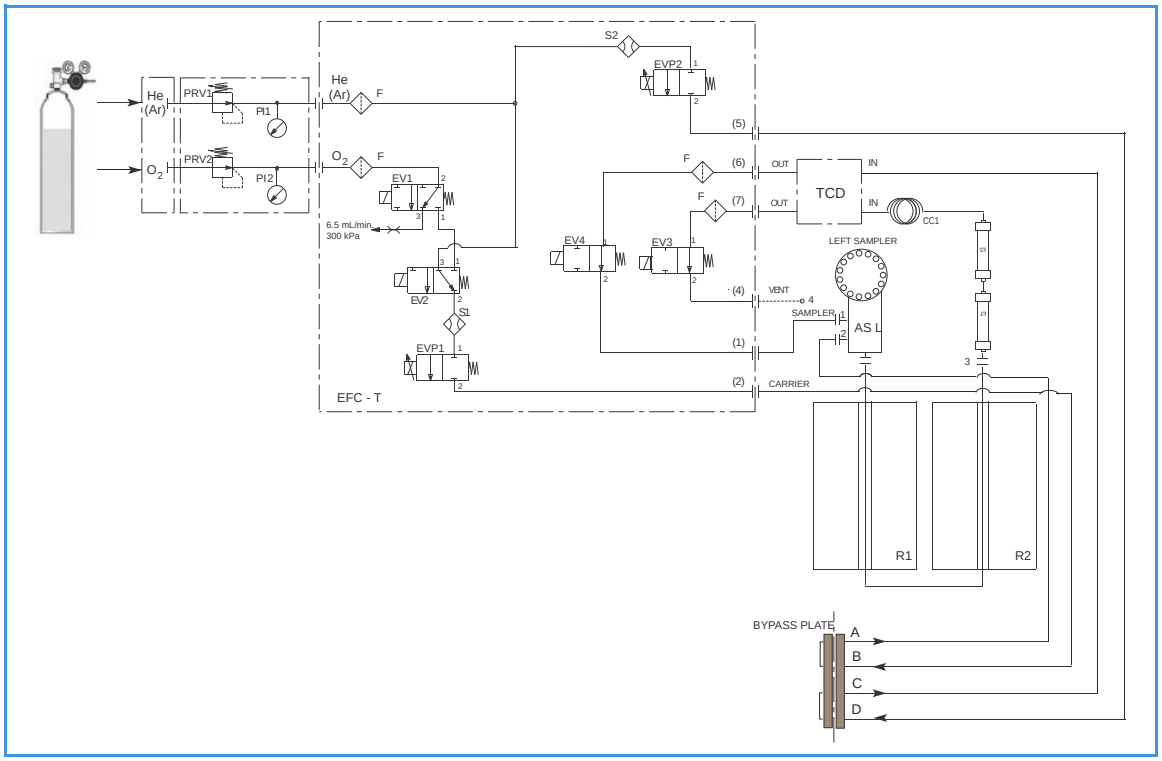



**Figure 4-27.** Instrument Parts Diagram and Pneumatic Diagram for N Lubricant, N Brew, and N/Protein Configurations

**Table 4-13.** Components of the Pneumatic Circuit for N Lubricant, N Brew, and N/Protein Determinations

| Component | Description |
|---|---|
| EV1-EV2 EV3-EV4<br>EVP1-EVP2<br>S1-S2 | They constitute the EFC-t module. See "Electronic Flow Controller (EFC-t) Module" on page 4-3. |
| AS | Autosampler |
| R1 | Oxidation reactor |
| R2 | Reduction reactor |
| F1 | Adsorption filter for carbon dioxide |
| F2 | Adsorption filter for water |

**Table 4-13.** Components of the Pneumatic Circuit for N Lubricant, N Brew, and N/Protein Determinations, continued

| Component | Description |
|---|---|
| CC | Gas chromatographic column |
| TCD | TCD thermal conductivity detector |
| CP | Pressure stabilizing cylinder |

**Tip** The pressure stabilizing cylinder CP avoids the introduction of air from the point 4 during the extended combustion of samples with very high weigh.

### Pneumatic Diagram Description

Helium **He** (argon **Ar**) 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 (argon) flow in the whole pneumatic circuit as far as the solenoid valve **EV3**. This valve, normally open, exhausts helium (argon) to the atmosphere through **Vent 4**. The proportional valve **EVP2**, connected to the detector reference channel **RF**, controls the helium (argon) flow as far as the solenoid valve **EV4**.
This valve, normally open, allows helium (argon) 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

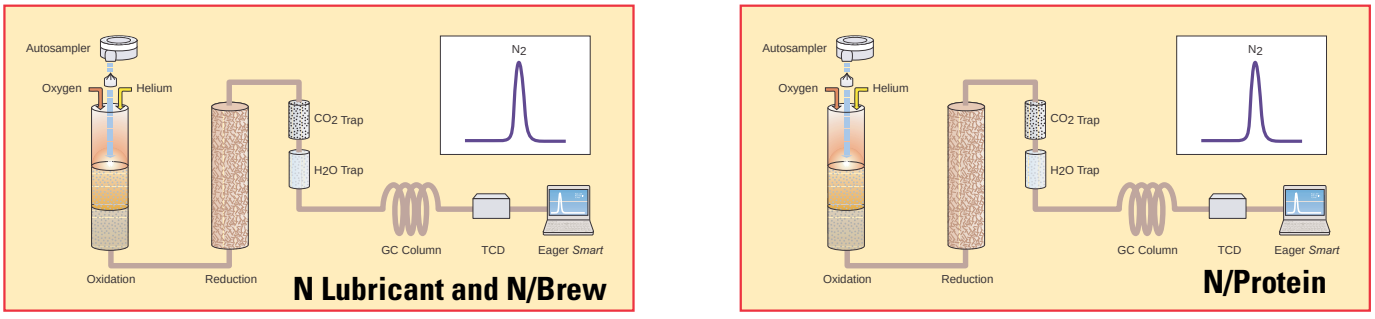



**Figure 4-28.** Schematics of N Lubricant, N Brew and N/Protein Configurations

During **pre-analysis**, the solenoid valve **EV1** shuts off oxygen, whereas the solenoid valve **EV2** allows helium (argon) to flow in the circuit.

When **Start Analysis** is pressed, the valve **EV1** switches from point 2-3 to point 2-1, 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 (argon) flow. The gas mixture ($N_2$, $CO_2$, $H_2O$ and $SO_2$) 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 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**.

The electrical signals generated by the detector are properly processed by the Eager*Smart* software providing the nitrogen-protein percentage.

5

# Installing the Flash*Smart* Elemental Analyzer

This chapter provides the instruction for installing the Flash*Smart* ™ Elemental Analyzer.

**Contents**

# Introduction

This section contains the information for installing and connecting the Flash*Smart* Elemental Analyzer, and for the electrical requirements.

## Who Performs the Installation

Your Flash*Smart* Elemental Analyzer will be installed by an authorized Thermo Fisher Scientific engineer (FSE), who will verify the instrument operation. If, for any reason, your system is not installed by a Thermo Fisher Scientific FSE, make sure that the following operations are performed.

## Standard Outfit

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

## Verifying the Site Preparation

Before you install the Flash*Smart* Elemental Analyzer, your laboratory must be in compliance with the guidelines and requirements described in the *FlashSmart Preinstallation Requirements Guide*.

## Unpacking the Instrument

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

❖ **To unpack the instrument**

1. Inspect the exterior of the shipping container for damage.
2. Carefully unpack the instrument.
3. Check the contents of each box against the packing list to verify the shipment is complete.
4. Inspect each item for damage.
    a. If equipment is damaged, keep the boxes and their equipment in their existing condition and immediately notify the carrier.
    b. Submit a damage claim directly to the carrier, and send a copy (including any shortage claim) to your authorized Thermo Fisher Scientific sales representative.

c. Do not return any equipment to the dealer or the factory without prior Thermo Fisher Scientific authorization.

## Placing the Instrument

Place the Flash*Smart* Elemental Analyzer on the workbench, allowing free access to electrical connections and gas lines.

**⚠CAUTION**

**Lifting Hazard.** The Flash*Smart* Elemental Analyzer weighs approximately 65 kg (145 lb) when unpacked. Pay attention when you lift the instrument onto the workbench. At least *two people* should perform this operation, each standing on the left/right side of the instrument and putting their hands near its supporting feet.

**Figure 5-1.** Placing the instrument

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

## Environmental Conditions

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

# Making the Gas Supply Plumbing Connections

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

## Building the Gas Lines

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

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

- 1/8-in. diameter (gas lines longer than 3 m [10 ft]
- 1/8-in. stainless steel tubing, properly cleaned
- a tubing cutter
- two wrenches

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

❖ **To connect the regulators and the tubing to the gas supply tank**

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

## Purging the Gas Lines

We recommend to purge the lines each time you make a cut in the tubing during the gas line assembly process. This will clear them of any debris from the cut. You should also purge the completely assembled gas lines before you connect the gas supply to the Flash*Smart* Elemental Analyzer.

❖ **To purge the gas lines**

1. Turn on the gas supply, and set the pressure to 35 kPa (0.35 bar, 5 psig).

2. Let the line purge for 10 minutes.
3. Turn off the gas supply.

## Connecting the Gas Lines

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

**NOTICE** The maximum pressures of the gases to supply the Flash*Smart* Elemental Analyzer is 700 kPa (7 bar).

❖ **To connect the gas lines**

1. Connect the helium (argon) gas line to the inlet labeled **He** (**Ar**) on the instrument rear panel. Gas inlet pressure must be adjusted through the reducing valves at 50 kPa higher than the nominal pressure.
2. Connect the oxygen gas line to the inlet labeled $\mathbf{O_2}$ on the instrument rear panel. Gas inlet pressure must be set to **400-500 kPa** (4-5 bar, 58-73 psig) according to the instrument configuration.
3. Use the pressure regulators and the pressure gauges located in the detector compartment of the instrument to set the pressure of the gases as follows:
   - **250 kPa** (2.5 bar, 36 psig) for helium (He), argon (Ar) (in N/Protein configuration), and nitrogen ($N_2$)
   - **400-450 kPa** (4.0-4.5 bar, 26-31 psig) for argon (in NC Soils configuration)
   - **250-300 kPa** (2.5-3 bar, 36-44 psig) for oxygen ($O_2$) according to the instrument configuration.

**Tip** Nitrogen ($N_2$) can be used for saving helium when the Flash*Smart* Elemental Analyzer is not used for a prolonged period of time, for example, overnight, or on weekends.

# Electrical Connections

This section explains the electrical connections of the Flash*Smart* Elemental Analyzer, and helps you to install and configure the peripheral devices and the Eager*Smart* Data Handling Software.

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

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

Unpack the peripheral devices that are shipped with the system and follow the instructions included with them. Follow the instructions in the paragraphs below to connect your peripheral devices to the Flash*Smart* Elemental Analyzer.

❖ **To connect the autosampler cable**

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

❖ **To connect the RS 232 cable**

1. Connect the RS 232 cable supplied in the standard outfit between the **COM1** or **COM2** ports of your computer and the 9-pin connector labeled **RS 232** on the instrument connection panel.

   If your computer is equipped with USB ports, a Serial-to-USB adapter is required to properly connect the cable.

2. Plug in the power cables for the instrument and the computer.

# Installing the Eager*Smart* Data Handling Software

The required software package includes the following items:

- Yellow pen driver (USB stick) containing the Eager*Smart* software
- *EagerSmart Software Manual* included in the green pen driver (USB stick) “OEA Documents”

For instructions about installing the Eager*Smart* software, see page 11-3 and refer to the *EagerSmart Data Handling Software Manual* for further details.

6

# Installing the MAS Plus Autosampler

This chapter provides the instruction for installing the MAS Plus autosampler for solid samples on the Flash*Smart* Elemental Analyzer.

**Contents**

# MAS Plus Autosampler Overview

The MAS Plus autosampler for solid samples is provided with the Elemental Analyzer. See Figure 6-1.

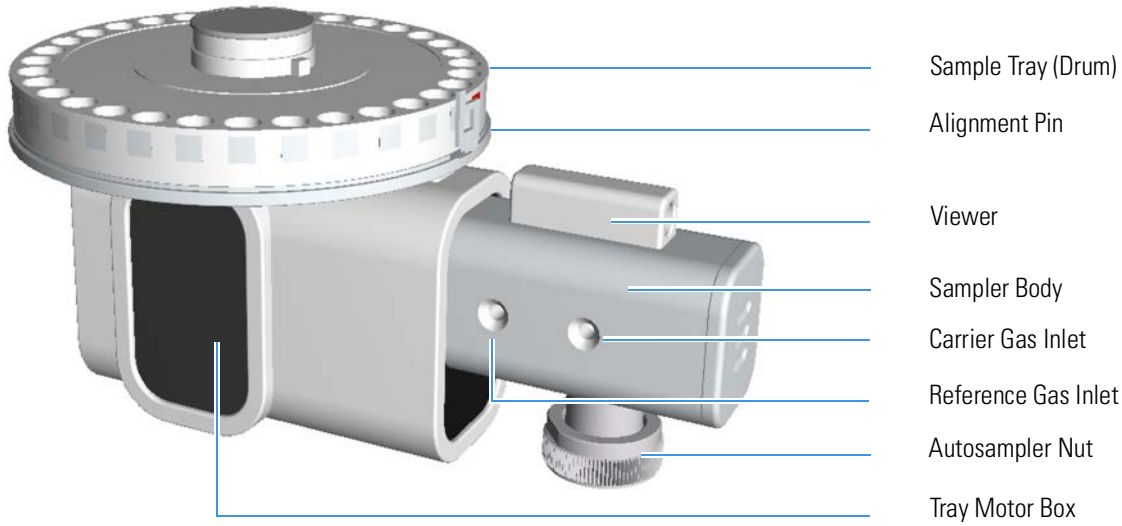



**Figure 6-1.** MAS Plus Autosampler

It consists of:

- An anodized aluminum block (sampler body) provided on the left side with fittings for carrier gas and reference gas lines connection.
- A 32-position sample-holding tray numbered 1 to 32. It is provided with a reference pin to be introduced into the seat labeled 1, which has a locating mark. See Figure 6-2.

  The modular design of the MAS Plus allows adding up to three additional 32-position sample trays to reach a capacity of 125 samples. Each sample tray is installed in a specific position defined by the numbering. They are not interchangeable. See Figure 6-3.

  The sample numbering is detailed in Table 6-1.

**Table 6-1.** Sample Tray Numbering

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

The correct alignment of the locating mark is important for the installation of the sample tray on the MAS Plus autosampler. See Figure 6-4.

- A motor for the movement of the sample tray (drum).
- A viewer on the sampler body.

**Tip** The viewer on the sampler body allows observation of the Flash combustion. See Figure 6-5.

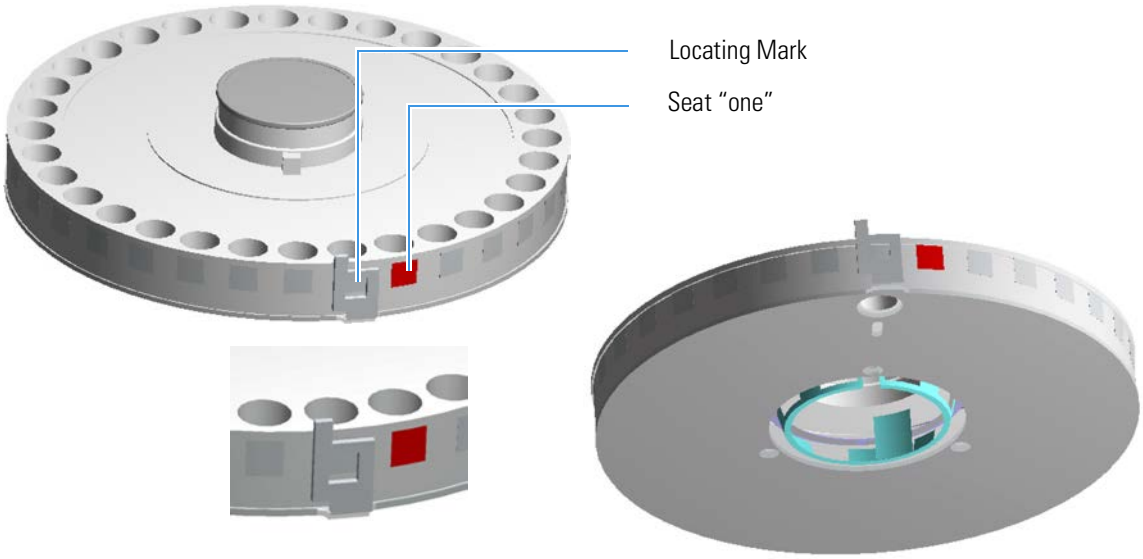



**Figure 6-2.** Sample Tray (Drum)

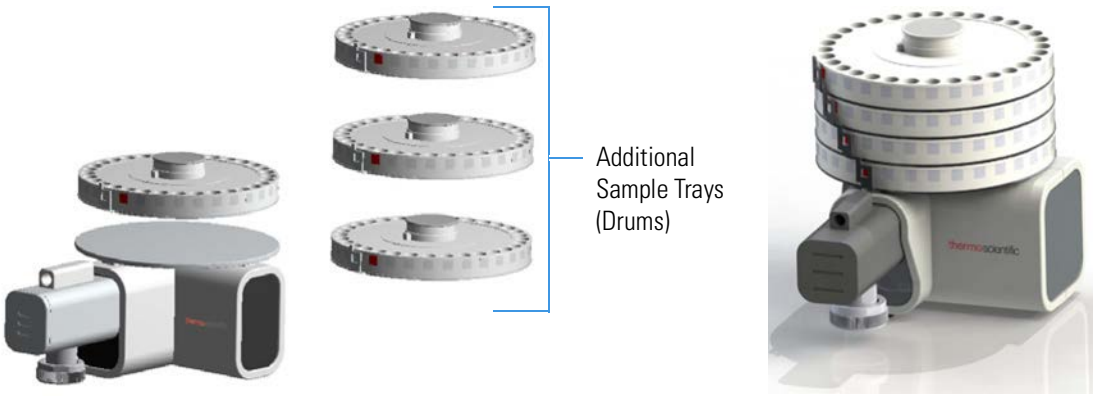
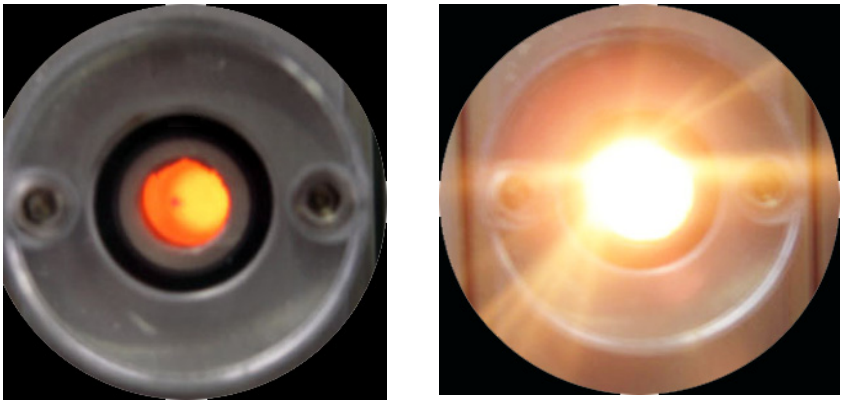



**Figure 6-3.** Additional Sample Trays


**Figure 6-4.** Sample Trays (Drums)



Before Sampling Flash Combustion

**Figure 6-5.** Viewer inside MAS Plus autosampler

# Installing the MAS Plus Autosampler

The installation sequence is common to all instrument configurations. The MAS Plus autosampler is normally installed on the left channel, but it can be installed on the right channel in the same way. Particular configurations of the Flash*Smart* Elemental Analyzer can require the use of two MAS Plus autosamplers; for example, the Flash*Smart* EA with MultiValve Control (MVC) Module.

**⚠CAUTION** **High Voltage.** Before you start, make sure that the Flash*Smart* Elemental Analyzer is powered off and that the reactors required for your analyses are installed in their corresponding furnace.

❖ **To install a MAS Plus autosampler**

| Material Required |
| --- |
| 8 mm wrench, open-ended |

1. Place the autosampler on the connecting nut of the concerned channel.
2. Manually screw the autosampler nut on the concerned channel.

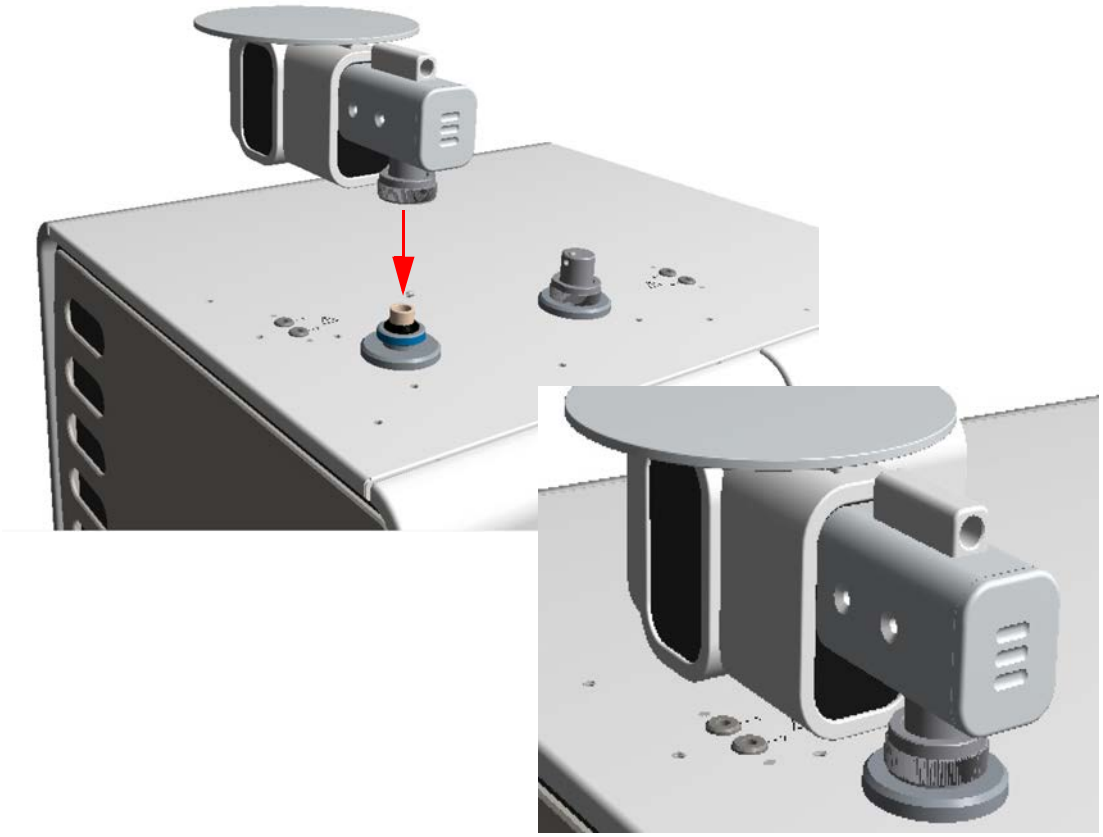
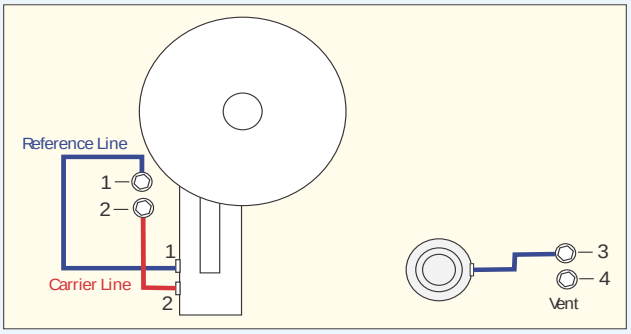


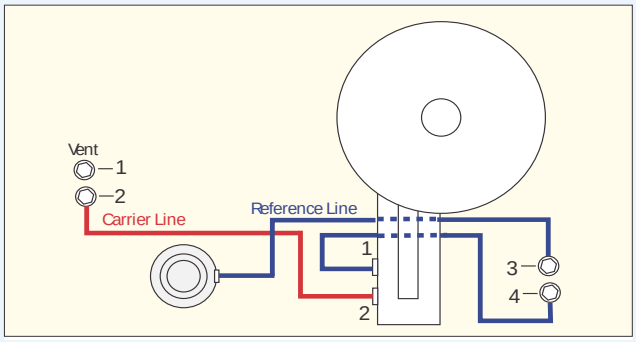
**Figure 6-6.** Installing a MAS autosampler

3. Connect the tubings coming from the gas connections, located on the analyzer, to the relevant connections of the autosampler.

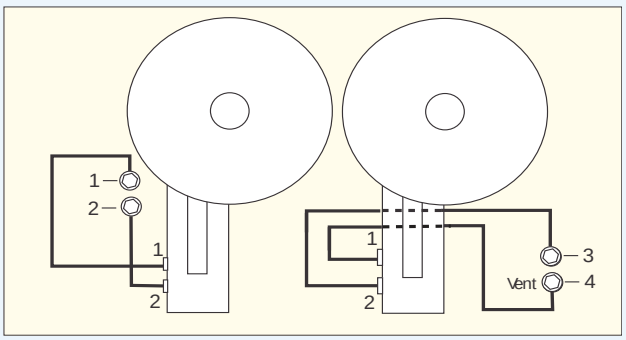
**Tip** If you are installing a MAS Plus autosampler on the **left channel**, the tubings coming from the gas connections must be connected as follows:




**Tip** If you are installing a MAS Plus autosampler on the **right channel**, the tubings coming from the gas connections must be connected as follows:




**Tip** If you are installing **two** MAS Plus autosamplers, the tubings coming from the gas connections must be connected as follows:




4. Connect the signal cable of the MAS Plus autosampler to the 2-pin connector labeled **Autosampler** on the back panel of the analyzer.

**Tip** When two MAS Plus autosamplers are installed, you cannot connect the signal cables of both autosamplers simultaneously. This is allowed only when the MultiValve Control (MVC) Module is used.

5. Install the samples tray (drum).

    a. Manually rotate the toothed wheel clockwise until the guide located on its rim is perfectly aligned with the metal pin of the autosampler body.

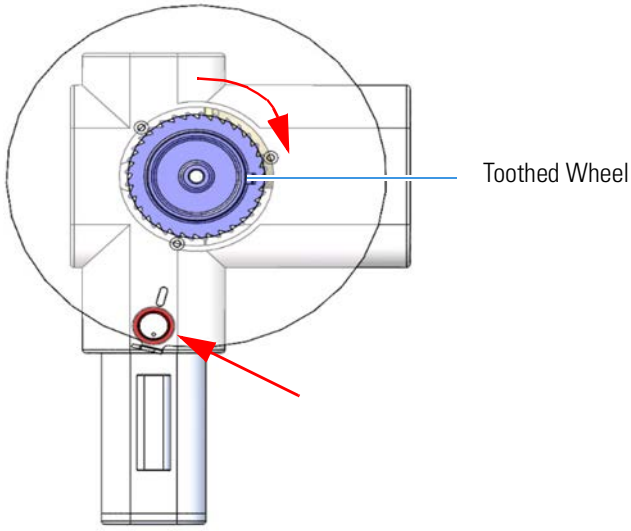



**Figure 6-7.** Rotating the toothed wheel

    b. Check that the sample tray (drum) reference pin is in correspondence with the seat labeled "**1**."

    c. Place the sample tray, with the reference pin in correspondence with the "**1**" seat, onto the toothed wheel. Make sure that the base matches with the guides.

**Figure 6-8.** Placing the sample tray onto the toothed wheel

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

6. If additional sample trays are required, install them in the correct order one over the other. Make sure that the relevant locating marks are in correspondence with the relevant seats labeled `0` (zero). Make sure that you place the samples properly.

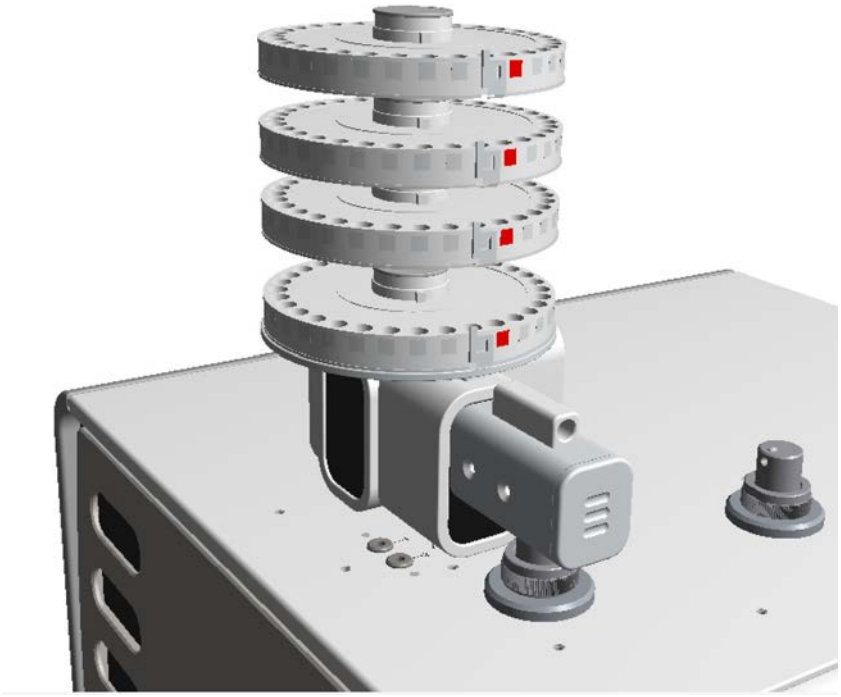


**Figure 6-9.** Installing additional sample trays

**Tip** Before you install an additional sample tray, make sure that the samples to analyze are placed in all the seats of the previous tray installed.

**NOTICE**

Before you start the samples analyses, make sure that the protection cover is positioned over the top sample tray. A complete de-aeration of the area where samples are housed is only possible if the cover is in place. Do not invert the cover. The surface labeled "*Side-up*" must be turned towards you.

7

# Installing the AI 1310/AS 1310 Autosampler

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

**Contents**

# Introduction

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

## Who Performs the Installation

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

## Electrical Requirements

The instrument has the following power supply rating:

- 24 Vdc through a portable external power supply, level VI efficiency

  Electrical characteristics of the supply:

  - input 100–240 Vac; 50–60 Hz
  - output 24 Vdc; 3 A–3.5 A

**⚠WARNING** Use only the portable external power supply that is supplied with the instrument by Thermo Fisher Scientific.

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

## Lifting and Carrying the Sampling Unit

Lift and carry the sampling unit by hand. See Figure 7-1.

**Figure 7-1.** How to Lift and Carry the Sampling Unit

# Installing the Direct Injection Device to the Flash*Smart* Elemental Analyzer

This device is installed in replacement of the MAS Plus autosampler for solids, when present. See Figure 7-2.

**Figure 7-2.** Direct Injection Device (1)

❖ **To install the direct injection device**

1. If it is present, disconnect the MAS Plus autosampler from the reactor, and place the autosampler nut on the stainless steel plate. If the MAS Plus autosampler is not present, remove the reactor fitting by unscrewing the relevant fixing nut.
2. Disconnect the gas connection.
3. Install the direct injection device over the reactor. See Figure 7-3.
    a. Mount the septum with the septum holder provided in the standard outfit.
    b. Connect the gas line to the direct injection device.

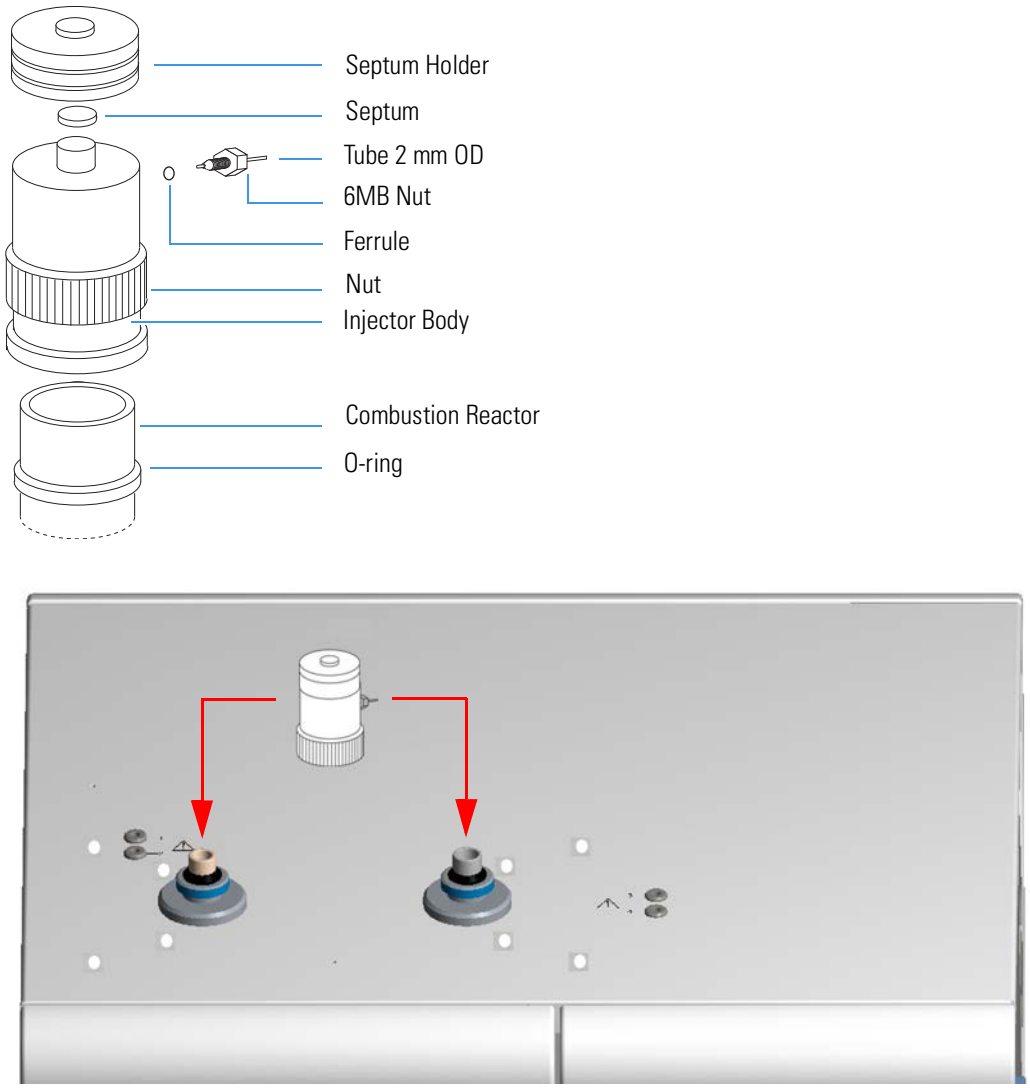



**Figure 7-3.** Direct Injection Device Installation

# Installing the Sampler Support on the Flash*Smart* Elemental Analyzer

The AI 1310/AS 1310 sampling system is installed on the Flash*Smart* Elemental Analyzer with the appropriate support provided. See Figure 7-4.

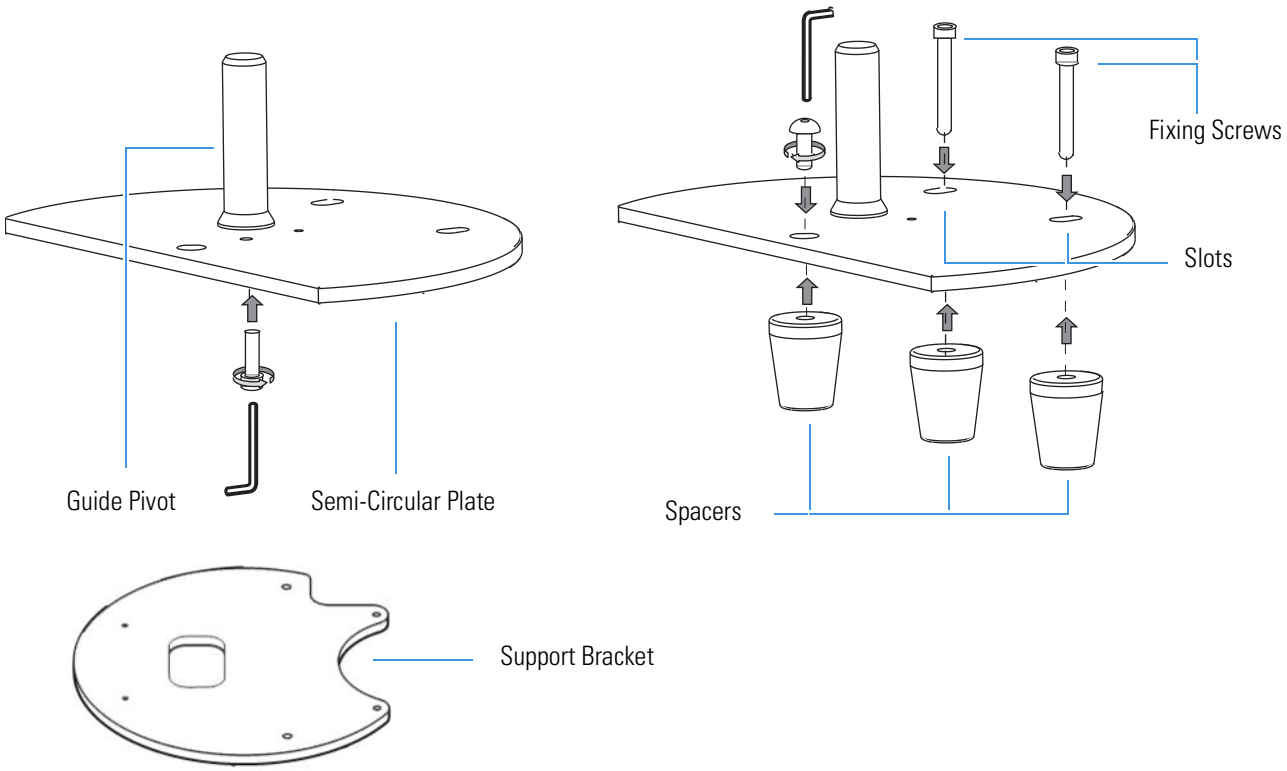



**Figure 7-4.** Sampler Support

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

❖ **To install the sampler support on the Flash*Smart* Elemental Analyzer**

1. Mount the support bracket.

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

   a. From the top panel of the Flash*Smart* Elemental Analyzer remove the four plastic caps covering the corresponding fixing holes. See Figure 7-5.

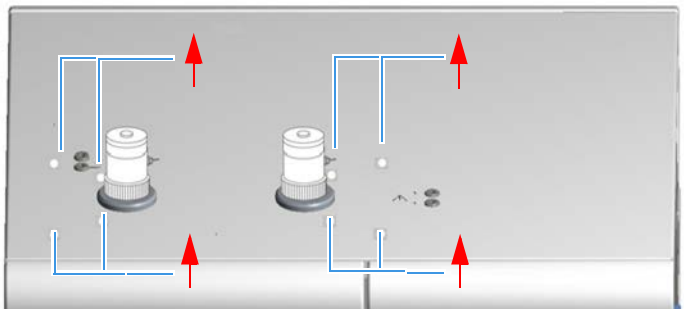


**Figure 7-5.** Plastic Caps Removal

b. Mount and fix the support bracket on the top panel of the Flash*Smart* Elemental Analyzer with the provided fixing screws. See Figure 7-6.

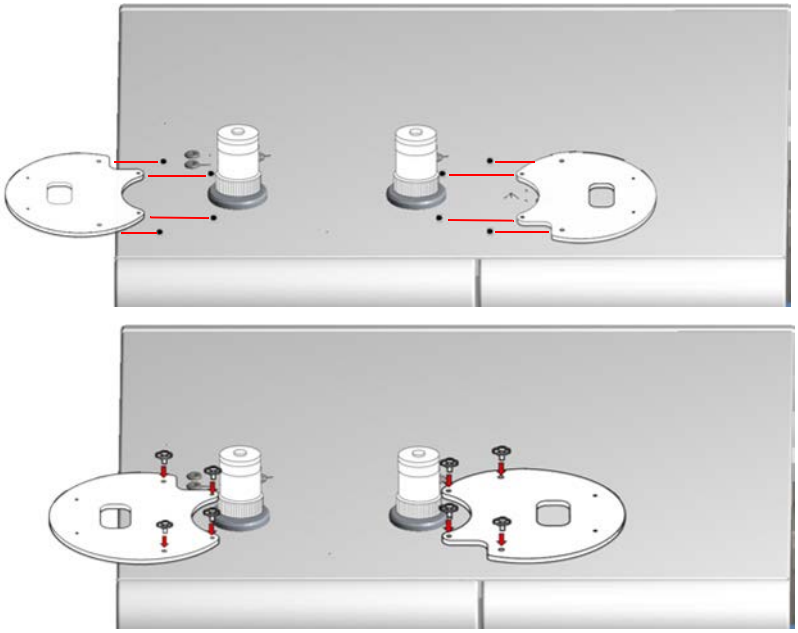


**Figure 7-6.** Mounting Support Bracket

2. Mount the sampler support. See Figure 7-7.

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

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

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

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

   e. Loosely tighten the screws.

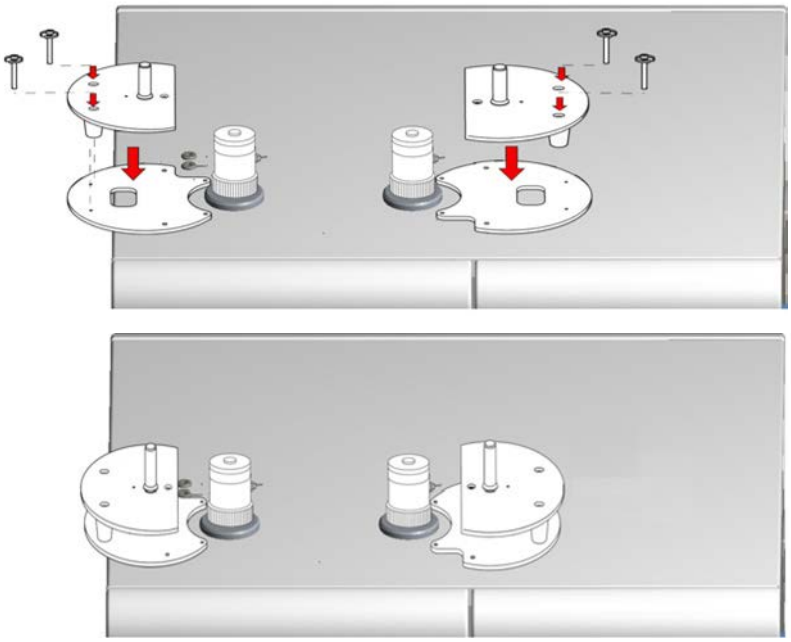


**Figure 7-7.** Mounting Sampler Support

f. If you must install an **AS 1310** autosampler, screw the support pin into the dedicated hole on the top plate. See Figure 7-8.

**Tip** The support pin is NOT required if you must install an AI 1310 autosampler.

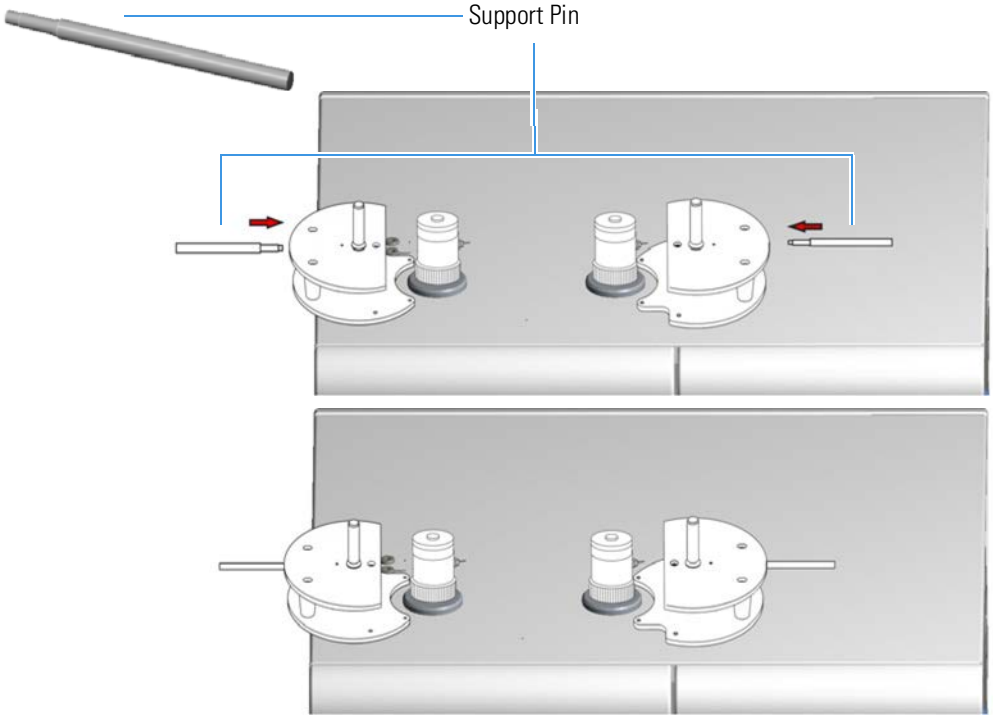



**Figure 7-8.** Mounting the Support Pin

# Installing the AI 1310/AS 1310 Autosampler on the Flash*Smart* Elemental Analyzer

This section provides the instruction for installing the AI 1310/AS 1310 autosampler on the Flash*Smart* Elemental Analyzer. See Figure 7-9.

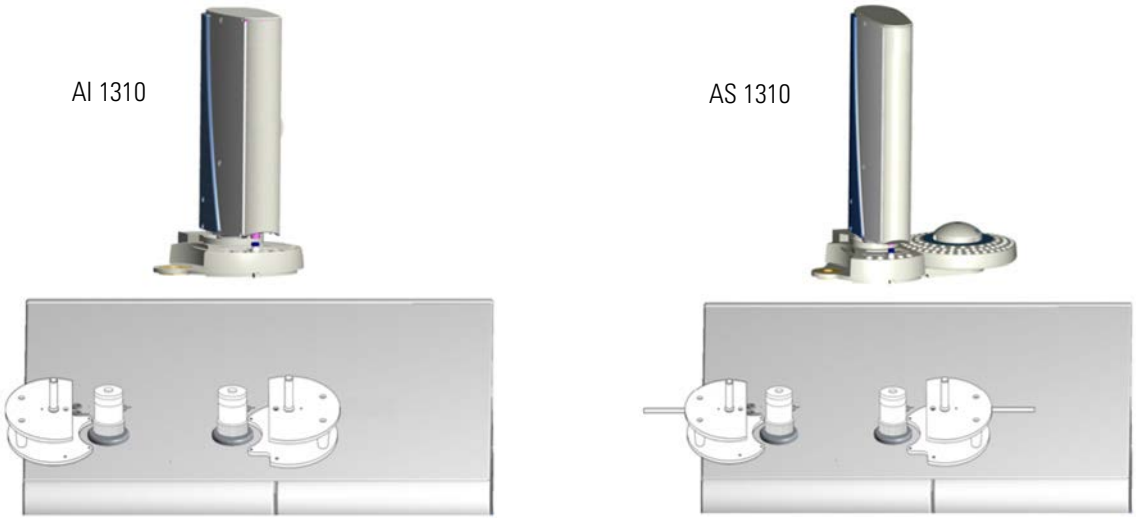



**Figure 7-9.** AI 1310/AS 1310 Installation

The installation procedure includes these steps:

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

## Installing the Sampling Unit

❖ **To install the sampling unit**

1. Lift the sampling unit and insert it into the guide pivot located on the sampling system support. Introduce the guide pivot into the hole provided on the bottom of the base. See Figure 7-10.

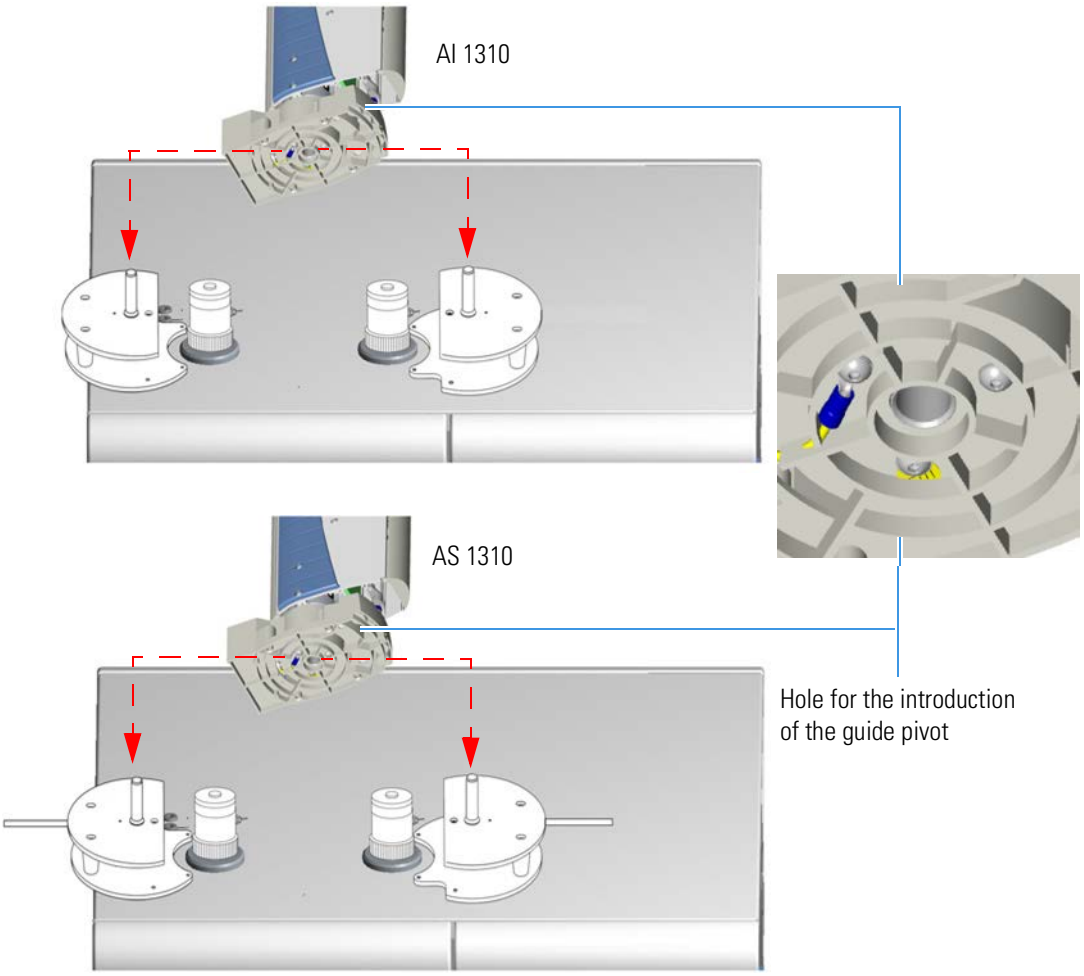



**Figure 7-10.** Installation of the Sampling Unit

2. Open the safety door and remove the protection of the injection assembly. See Figure 7-11.

3. Insert the centering plate into its seat located in the **right** side section of the sampling unit base. Make sure that the guide hole, present on the arm of the centering plate, correctly fits the injector nut. See Figure 7-12.

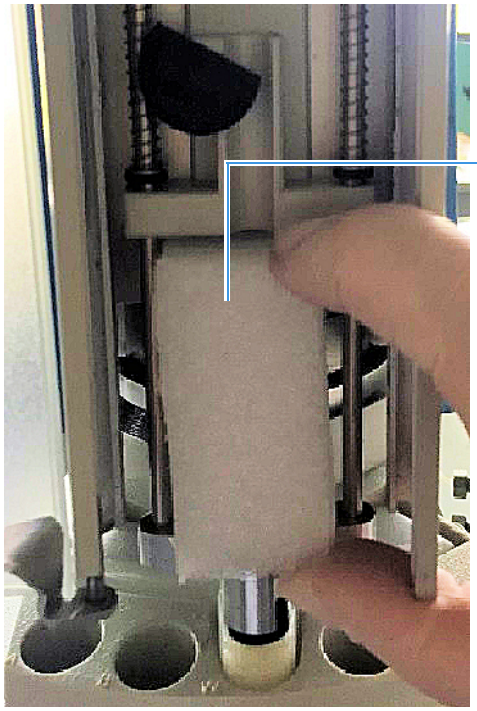



**Figure 7-11.** Remove Protection

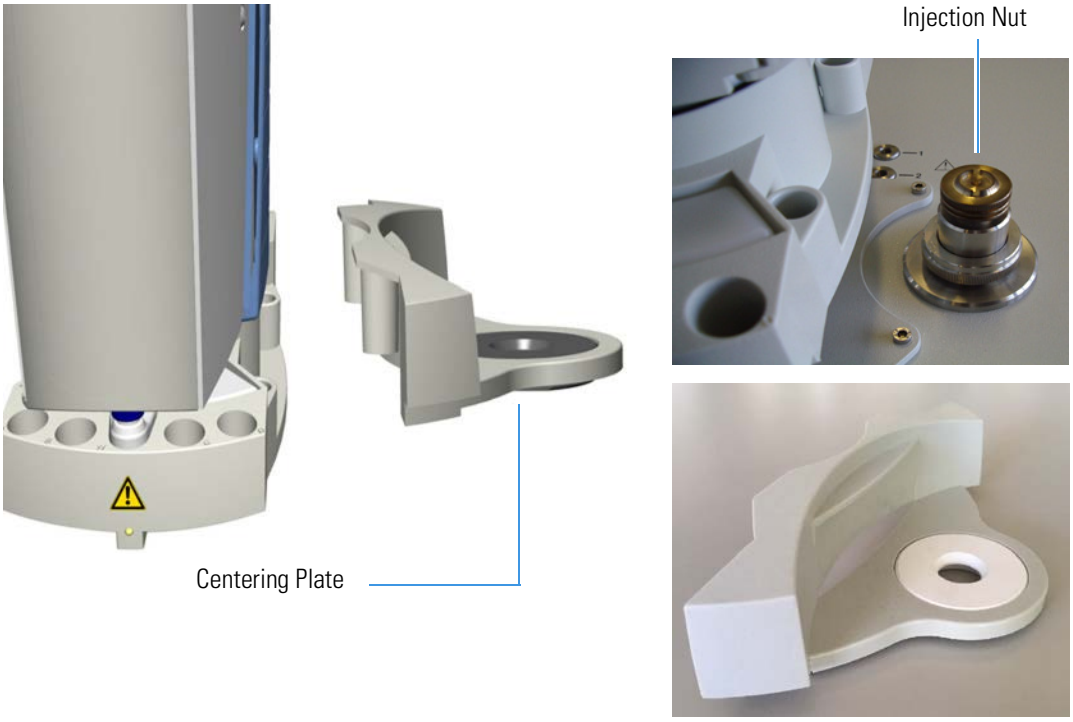



**Figure 7-12.** Centering Plate

4. Check the correct alignment of the sampling system support, then fix it by tightening the proper fixing screws.
5. Insert the sample tray into the sampling unit base.
   - **AI 1310** — Insert the 8-position sample tray into the appropriate housing of the sampling unit base. See Figure 7-13.

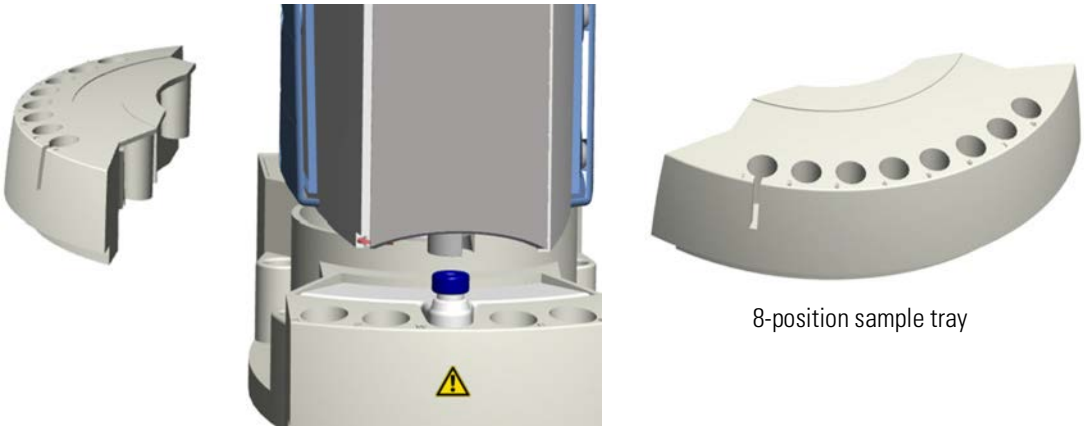



**Figure 7-13.** 8-position Sample Tray

- **AS 1310** — Insert the dedicated support plate into its appropriate housing of the sampling unit base. Place the 105-position sample tray on the hub located on the support. The system will automatically recognize the sample tray at the instrument power on. See Figure 7-14.

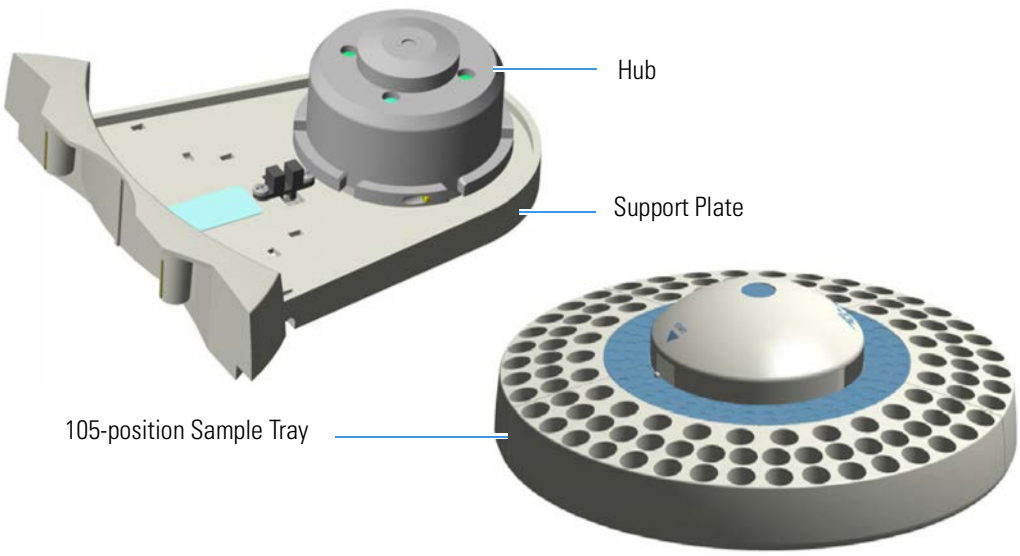



**Figure 7-14.** 105-position Sample Tray

## Installing the Syringe

The installation of the syringe must be performed with caution to avoid causing damage to the syringe needle and to ensure an optimal performance of the injection device.

The standard syringes have 10 µL and 250 µL capacity with a 50 mm needle. It is also possible to install 50 µL and 100 µL syringes with needles of 50 mm.

### ❖ To install the syringe

1. Open the safety door of the turret.
2. Insert the syringe needle into the vial capture device. See Figure 7-15.
3. Accommodate the syringe body into its seat paying attention to insert the flange and the head of the syringe plunger simultaneously into their relevant guides.
4. Turn the lock knob by approximately 180° clockwise to lock the syringe. See Figure 7-16.
5. Close the safety door of the rotating turret.

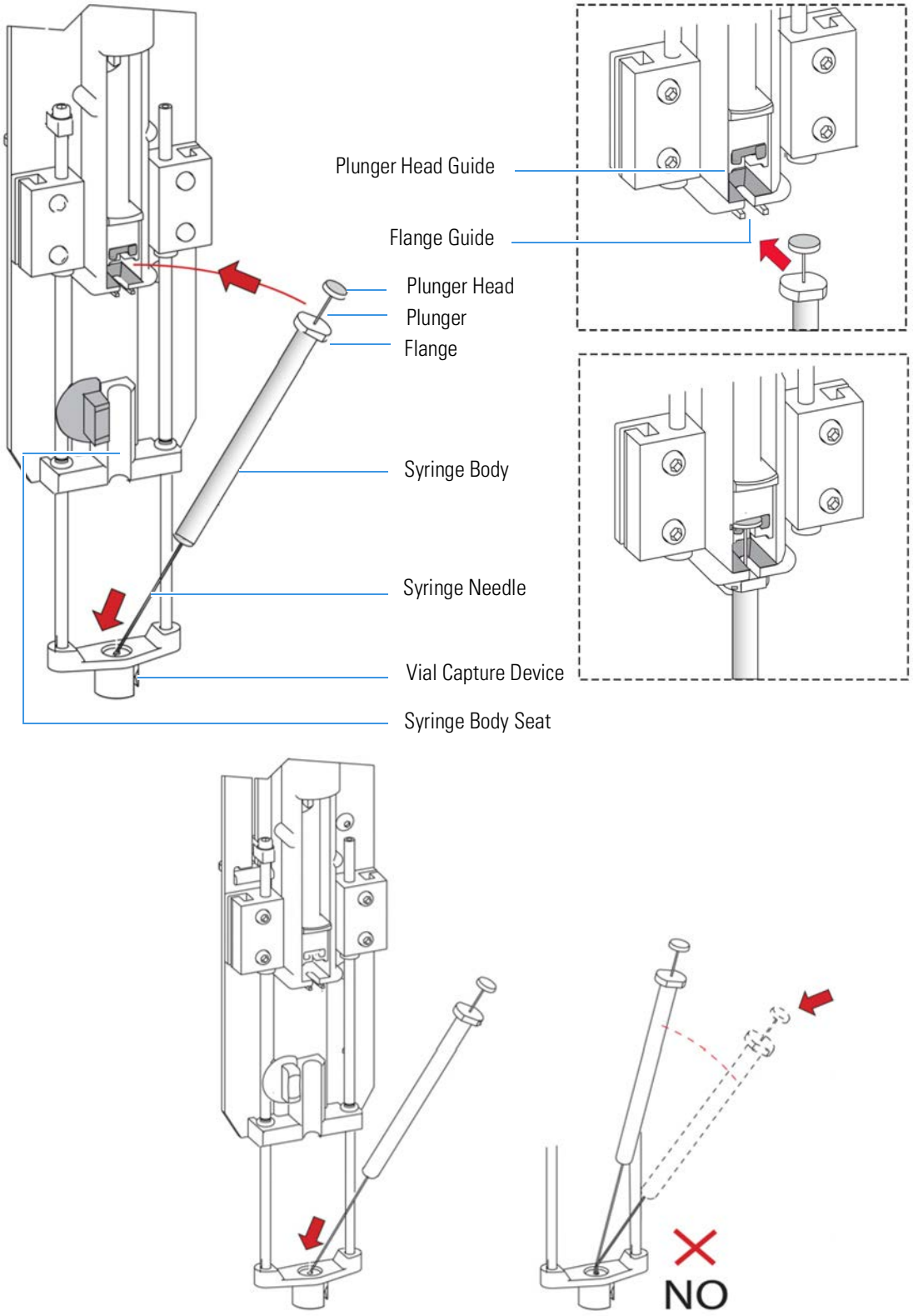



**Figure 7-15.** Syringe Installation (1)

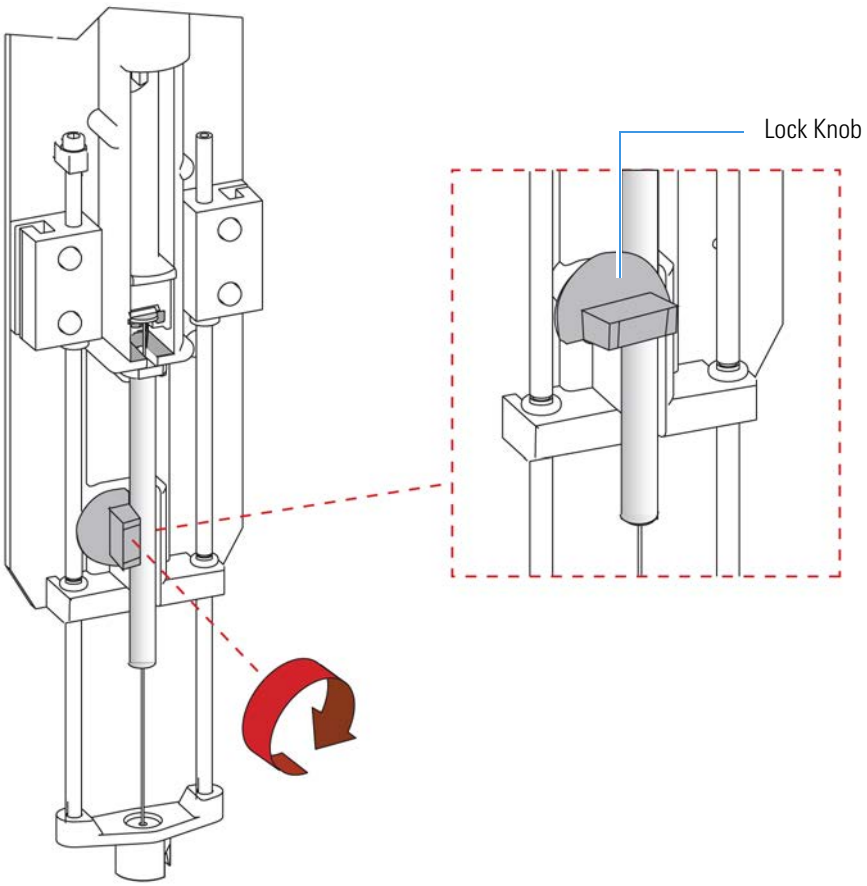



**Figure 7-16.** Syringe Installation (2)

## Installing the Electrical Connections

❖ **To install the electrical connections**

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

## Starting up

**NOTICE** You must only use the portable external power supply supplied with the instrument by Thermo Fisher Scientific.

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

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

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

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

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

8

# Preparing the Reactors and the Adsorption Filters

This chapter provides instructions for preparing the reactors and the adsorption filters for all the configurations of the Flash*Smart* Elemental Analyzer.

**Contents**

# 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 you.

## Reactors

The reactors can be quartz tubes or alloy steel tubes (HPAR):

- The quartz reactors and the alloy steel reactors (HPAR) have a conical bottom end.
- The alloy steel reactors (HPAR) have their top end provided with two through-holes.

The filling materials depend on the required analytical determination. See "Filling Materials" on page 8-5.

**Tip** The alloy steel reactors (HPAR), used for combustion, require the presence of a crucible. For more details, see page 8-40.

## Adsorption Filters

The adsorption filters can be Pyrex™ or Plexiglas™ filters. The filling materials depend on the required analytical determination. See "Filling Materials" on page 8-5.

## Gas Chromatographic Columns

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

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

Table 8-1 lists the characteristics of reactors, filters, and gas chromatographic columns required for each analytical determination.

**Table 8-1.** 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 Soils (argon carrier gas) |
| Reactors | Quartz | 45 | 18 | 14 | × | × | × | × | × | × | × | | | | × | × | |
| | Alloy Steel (HPAR) | 45 | 25 | 23 | | | | | | | | × | × | × | | | × |
| Filters | Pyrex™ | 11 | 10 | 8 | | | × | × | | × | | | | | × | × | × |
| | Plexiglas™ | 23 | 30 | 22 | | | | | | | × | × | × | × | | | |
| Columns | Stainless Steel | 100 | 6 | 5 | | | | | | × | | | | | | | |
| | | 200 | 6 | 5 | | | | | | | | | | | × | × | |
| | | 300 | 6 | 5 | | | | | | | | | | | | | |
| | | 500 | 6 | 5 | | | | | | | | | | | | | × |
| | PTFE | 15 | 6 | 4 | | | | | × | | | | | | | | |
| | | 50 | 8 | 6 | | | | | | × | × | | | | | | |
| | | 80 | 6 | 4 | | | | × | | | | | | | | | |
| | | 100 | 8 | 6 | | | | | | | | × | | × | | | |
| | | 200 | 6 | 5 | × | × | × | | | | | | | | | | |

# Filling Materials

Table 8-2 lists the materials used to fill reactors, adsorption filters, and gas chromatographic columns.

**Table 8-2.** Material Required for Filling Reactors, Filters and Chromatographic Columns

| | Characteristics | Analytical Determination | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Filling Material | CHNS | CHN | NCS | S (TCD) | S (FPD) | O | N | N Lubricant | N/Protein | N/Brew | NC | NC Soils |
| **Reactors** | Quartz Wool | × | × | × | × | × | × | × | × | × | × | × | × |
| | Electrolytic Copper | × | | × | × | × | | | | | | | |
| | Copper Oxide | × | | × | × | × | | × | | | | × | |
| | Reduced Copper | | × | | | | | × | × | × | × | × | × |
| | Chromium Oxide | | × | | | | | | | | | | |
| | Silvered Cobaltous/Cobaltic Oxide | | × | | | | | × | | | | × | |
| | Quartz Chips | | | | | | × | | | | | | |
| | Nickel Plated Carbon | | | | | | × | | | | | | |
| | Oxidation Catalyst | | | | | | | | × | × | × | | × |
| **Filters** | Quartz Wool | | | × | × | × | × | × | × | × | × | × | × |
| | Soda Lime | | | | | | × | × | × | × | × | | |
| | Molecular Sieves 3 Angstrom | | | | | | | × | × | × | × | | |
| | Magnesium Perchlorate (Anhydrone) | | | × | × | × | | | | | | × | × |
| | Silica Gel | | | | | | | × | × | × | × | | |
| **Columns** | Multi-separation Column (PTFE) | | × | | | | | | | | | | |
| | Multi-separation Column (Stainless Steel) | | | | | | | | | | | × | × |
| | Oxygen Separation Column | | | | | | × | | | | | | |
| | Nitrogen Separation Column (50 cm) | | | | | | | × | | × | | | |
| | Nitrogen Separation Column (100 cm) | | | | | | | | × | | × | | |
| | CHNS/NCS Packed Column | × | | × | | | | | | | | | |
| | Sulfur Separation Column | | | | × | × | | | | | | | |

# 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.

See the following sections:

# Filling Materials Colors Convention

Table 8-3 provides the colors convention used for graphical convenience to identify the materials required for filling the reactors and the filters according to the configuration of the Flash*Smart* Elemental Analyzer.

**Tip** The conventional colors DOES NOT RESPECT the natural color of the filling materials.

**Table 8-3.** Filling Materials and Flash*Smart* Elemental Analyzer Configurations

| Filling Material | Location | Part Number | Qty. | Flash*Smart* Elemental Analyzer Configuration |
|---|---|---|---|---|
| Quartz Wool | Reactor | 338 22200 | 5 g | All |
| Chromium Oxide | Reactor | 338 22900 | 25 g | CHN; CHN-O; |
| Copper Reduced | Reactor | 338 35312 | 50 g | CHN; CHN-O; N; N/Brew; N/Protein; N Lubricant; NC; NC Soils |
| Silvered Cobaltous-Cobaltic Oxide | Reactor | 338 24500 | 25 g | CHN; CHN-O; NC |
| Nickel Plated Carbon | Reactor | 338 23800 | 5 g | CHN-O; CHNS-O; O |
| Quartz Turning | Reactor | 338 22300 | 50 g | CHN-O; CHNS-O; O |
| Copper Oxide | Reactor | 338 21730 | 25 g | CHNS; CHNS-O; N; S; NC; NCS |
| Electrolytic Copper | Reactor | 338 35314 | 80 g | CHNS; CHNS-O; NCS: S |
| Oxidation Catalysts | Reactor | 338 40000 | 40 g | N/Brew; N/Protein; NC Soils; N Lubricant |
| Quartz Wool | Filter | 338 22200 | 5 g | NCS; S; O; N; N/Brew; N/Protein; NC; NC Soils; N Lubricant |
| Soda Lime | Filter | 338 35235 | 100 g | CHN/O; CHNS-O; O; N; N/Brew; N/Protein; N Lubricant |
| Magnesium Perchlorate (Anhydrone) | Filter | 338 21900 | 100 g | CHNS-O; O; S, NC; NC Soils; NCS |
| Molecular Sieves | Filter | 338 01801 | 100 g | N; N/Brew; N/Protein; N Lubricant |
| Silica Gel | Filter | 338 40035 | 100 g | N; N/Brew; N/Protein; N Lubricant |

# CHN Configuration

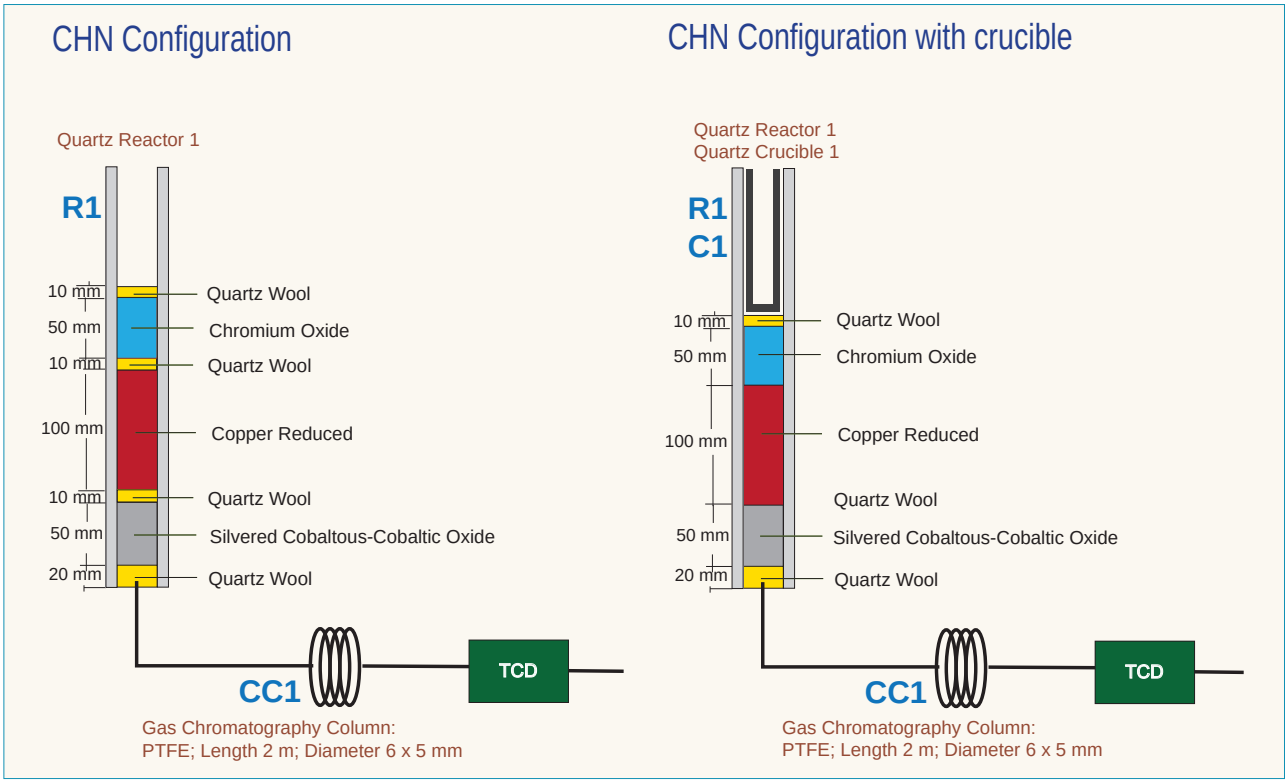
Table 8-4 lists the characteristics of the components required for **CHN determination**, and the type and size of the filling materials for a proper preparation of the reactor.

**Table 8-4.** Components Required for CHN Determinations

| Reference | Component | Characteristic | Filling material |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Reduced<br>Chromium Oxide<br>Silvered Cobaltous/Cobaltic Oxide |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 meters<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Quartz | |

**Tip** If the sample has a 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 high quality **copper reduced** and **cobaltous/cobaltic oxide**.

**Size of the Filling Material**



# CHN/CHN Configuration with MultiValve Control (MVC) Module

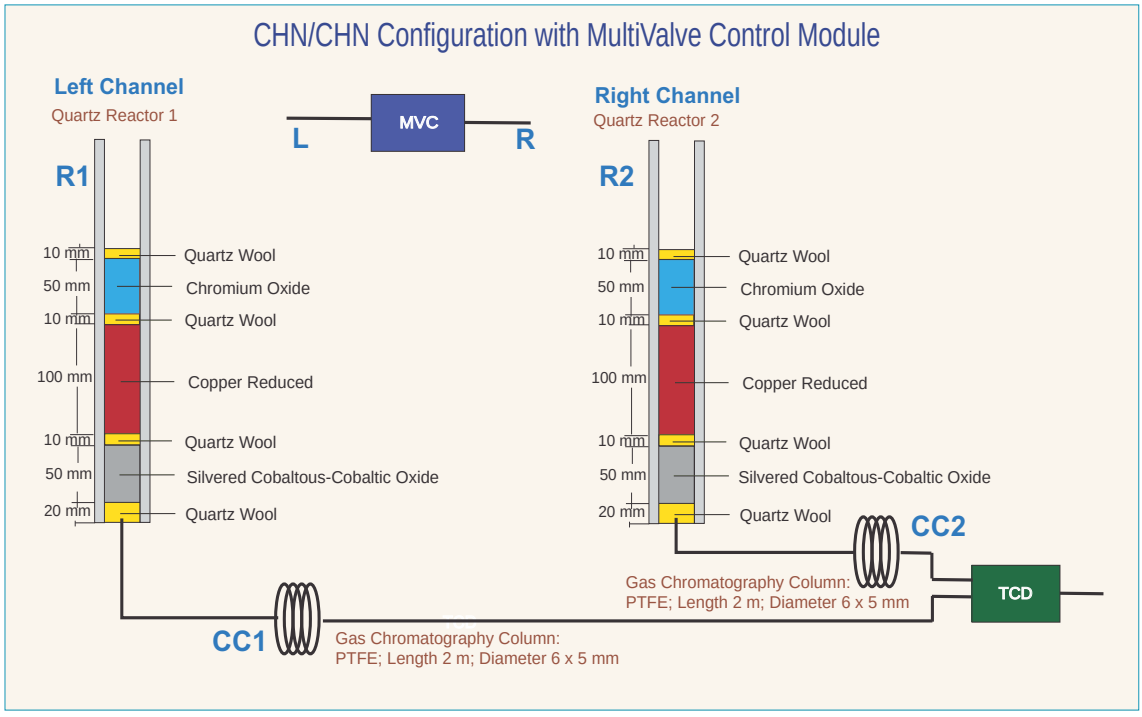
Table 8-4 lists the characteristics of the components required for **CHN/CHN determination**, and the type and size of the filling materials for a proper preparation of the reactors.

**Table 8-5.** Components Required for CHN Determinations

| Reference | Component | Characteristic | Filling material |
|---|---|---|---|
| R1-R2 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Reduced<br>Chromium Oxide<br>Silvered Cobaltous/Cobaltic Oxide |
| CC1-CC2 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 meters<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Quartz | |

**Tip** If the sample has a 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 high quality **copper reduced** and **cobaltous/cobaltic oxide**.

## Size of the Filling Material




**Tip** The crucible can be used as in the CHN Configuration.

# CHN/O Configuration

Table 8-6 lists the characteristics of the components required for **CHN/O determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filters.

**Table 8-6.** Components Required for CHN/O Determinations

| Reference | Component | Characteristic | Determination | Filling material |
|---|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | CHN | Quartz Wool<br>Chromium Oxide<br>Copper Reduced<br>Silvered Cobaltous/Cobaltic Oxide |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | CHN | --- |
| C1 | Crucible | *Material:* Quartz | --- | --- |
| R2 | Reactor | *Material:* Quartz | Oxygen | Quartz Wool<br>Nickel Plated Carbon<br>Quartz Turnings |
| F | Adsorption filter | *Material:* Pyrex™ | Oxygen | Quartz Wool<br>Soda Lime<br>Magnesium Perchlorate (Anhydrone) |
| CC2 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 1 m<br>*Diameter:* 6 × 5 mm | Oxygen | --- |
| C2 | Crucible | *Material:* Quartz | --- | --- |

**Tip** In some cases, it is suggested to use **nickel wool** in the top layer up the **nickel plated carbon** instead of **quartz wool**.

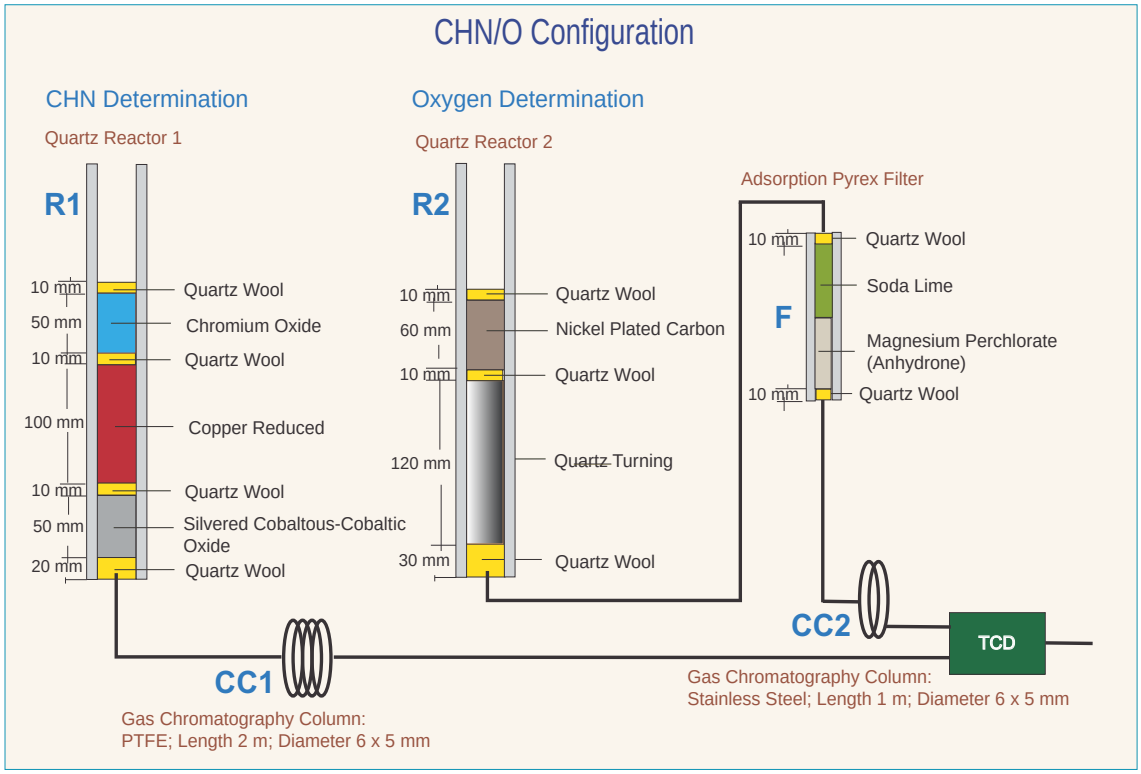
## CHN Determination

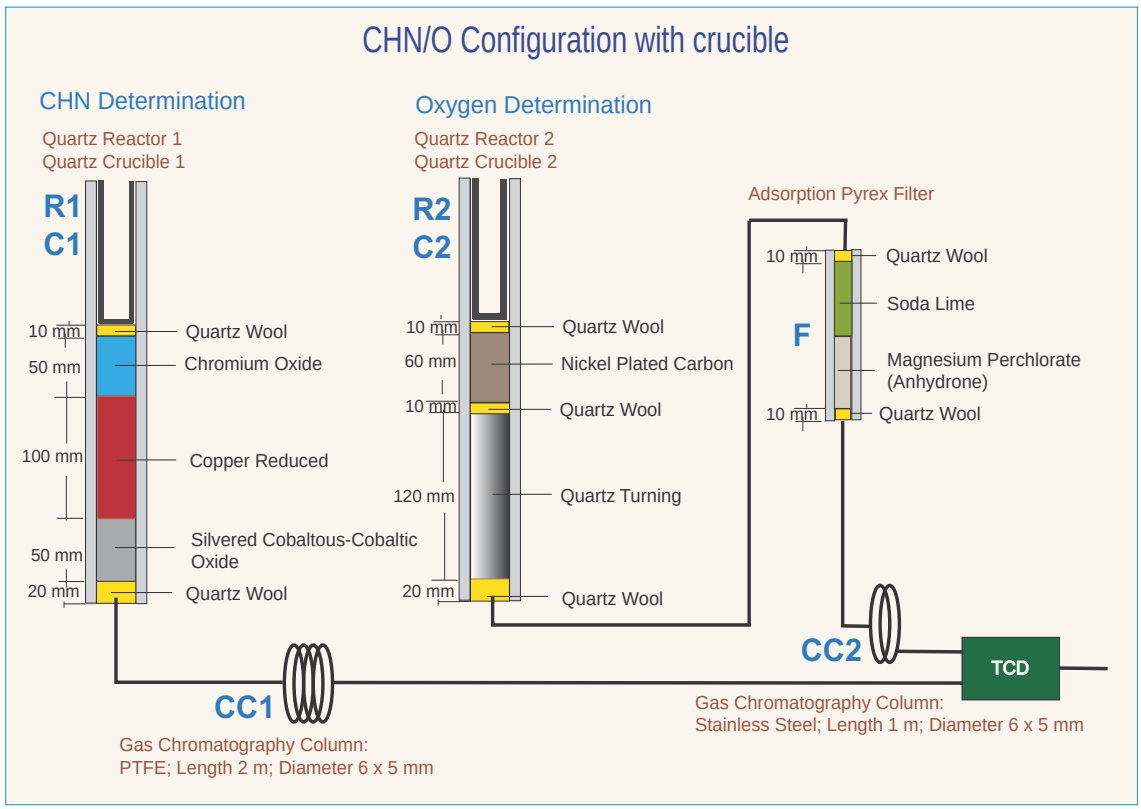
**Tip** If the sample has a high presence of inorganic material, we suggest to insert the **quartz crucible** into the oxidation/reduction reactor. In this case, it is necessary to eliminate the **quartz wool** between **chromium oxide** and high quality **copper reduced**, and between high quality **copper reduced** and **cobaltous/cobaltic oxide**.

## Oxygen Determination

**Tip** If the sample has a 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 the **quartz wool** from **30 mm** to **20 mm** in the lower section of the reactor.

## Size of the Filling Material



# CHN/O Configuration with MultiValve Control (MVC) Module

Table 8-6 lists the characteristics of the components required for **CHN/O determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filter.

**Table 8-7.** Components Required for CHN/O Determinations

| Reference | Component | Characteristic | Determination | Filling material |
|---|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | CHN | Quartz Wool<br>Chromium Oxide<br>Copper Reduced<br>Silvered Cobaltous/Cobaltic Oxide |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | CHN | --- |
| C1 | Crucible | *Material:* Quartz | --- | --- |
| R2 | Reactor | *Material:* Quartz | Oxygen | Quartz Wool<br>Nickel Plated Carbon<br>Quartz Turnings |
| F | Adsorption filter | *Material:* Pyrex™ | Oxygen | Quartz Wool<br>Soda Lime<br>Magnesium Perchlorate (Anhydrone) |
| CC2 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 1 m<br>*Diameter:* 6 × 5 mm | Oxygen | --- |
| C2 | Crucible | *Material:* Quartz | --- | --- |

**Tip** In some cases, it is suggested to use **nickel wool**, in the top layer up the **nickel plated carbon**, instead of **quartz wool**.

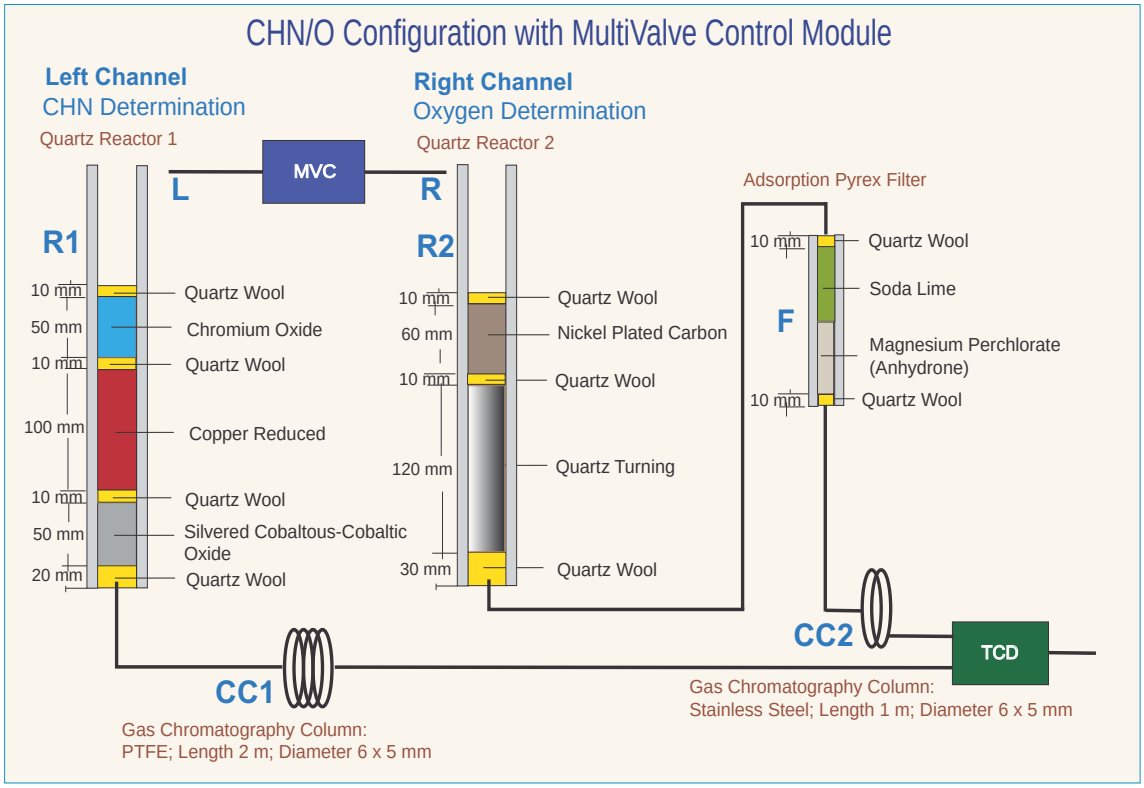
## CHN Determination

**Tip** If the sample has a 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 high quality **copper reduced** and **cobaltous/cobaltic oxide**.

## Oxygen Determination

**Tip** If the sample has a 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 the **quartz wool** from **30 mm** to **20 mm** in the lower section of the reactor.

### Size of the Filling Material




**Tip** The crucibles can be used as in the CHN/O Configuration.

# CHNS Configuration

Table 8-8 lists the characteristics of the components required for **CHNS determination**, and the type and size of the filling materials for a proper preparation of the reactor.

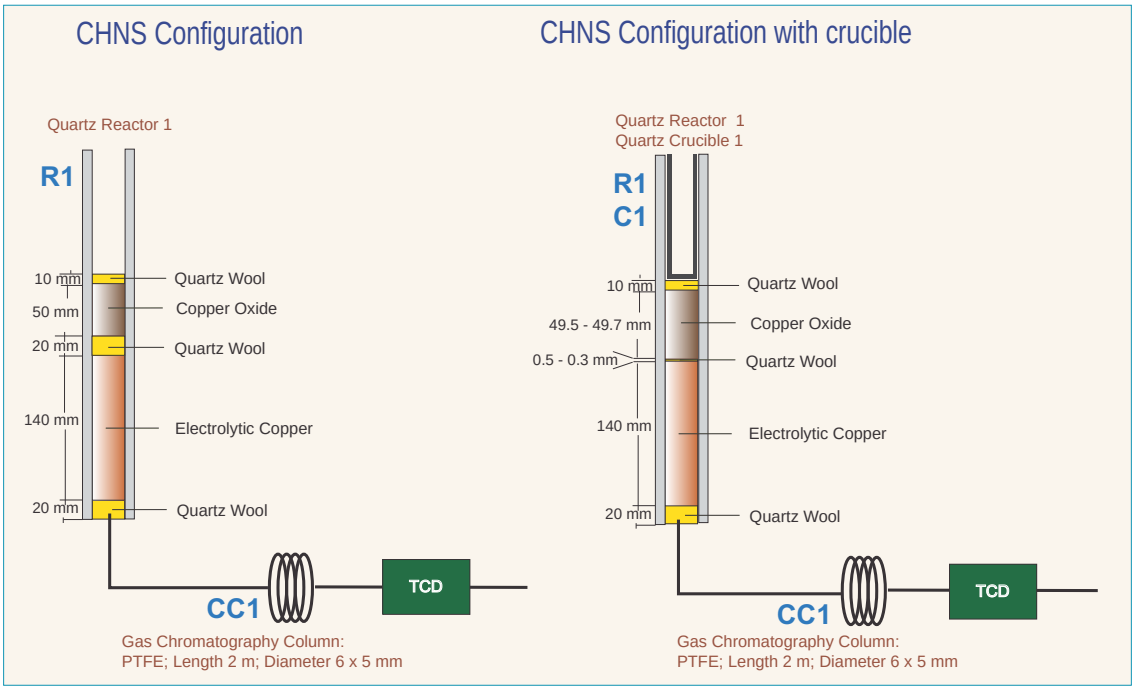
**Table 8-8.** Components Required for CHNS Determinations

| Reference | Component | Characteristic | Filling material |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Oxide<br>Electrolytic Copper |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Quartz | --- |

**Tip** **Vanadium pentoxide** ($V_2O_5$) is an oxygen donor. According to the sample nature, it is suggested to insert in the tin container with the sample for a proper oxidation of material and consequently a quantitative sulfur determination.

**Tip** If the sample has a 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.

**Size of the Filling Material**



# CHNS/CHNS Configuration with MultiValve Control (MVC) Module

Table 8-8 lists the characteristics of the components required for **CHNS/CHNS determination**, and the type and size of the filling materials for a proper preparation of the reactors.

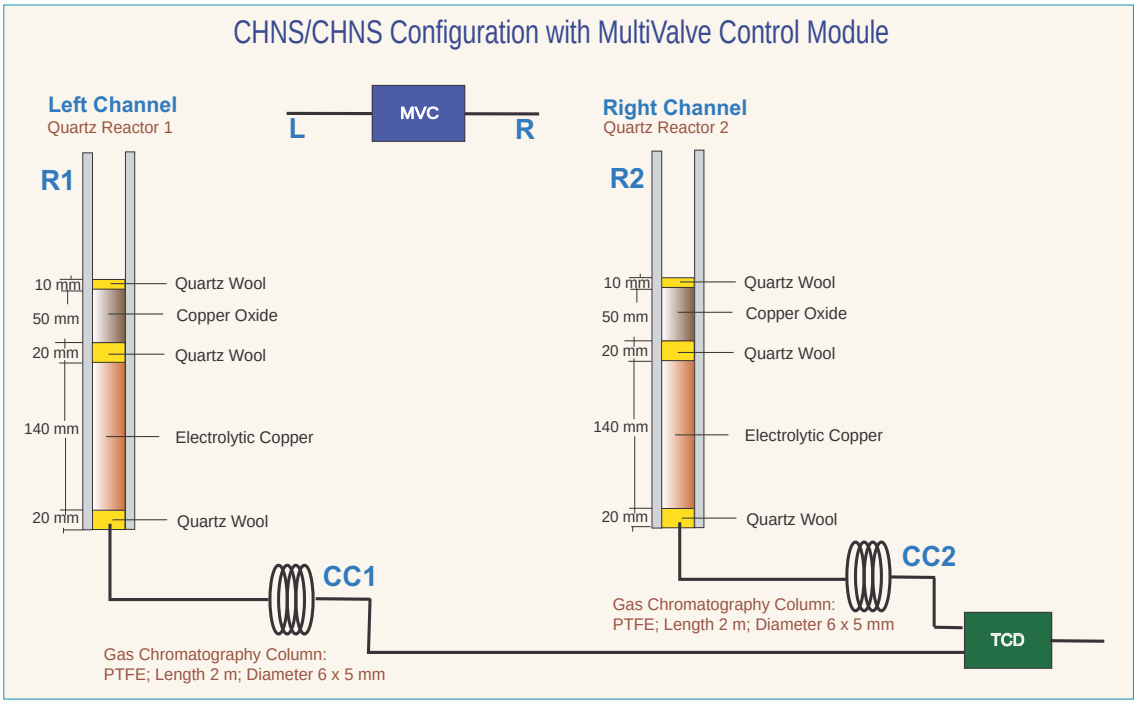
**Table 8-9.** Components Required for CHNS Determinations

| Reference | Component | Characteristic | Filling material |
|---|---|---|---|
| R1-R2 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Oxide<br>Electrolytic Copper |
| CC1-CC2 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Quartz | --- |

**Tip** **Vanadium pentoxide** ($V_2O_5$) is an oxygen donor. According to the sample nature, it is suggested to insert in the tin container with the sample for a proper oxidation of material and consequently a quantitative sulfur determination.

**Tip** If the sample has a 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.

**Size of the Filling Material**




**Tip** The crucible can be used as in the CHNS Configuration.

# CHNS/O Configuration

Table 8-10 lists the characteristics of the components required for **CHNS/O determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filter.

**Table 8-10.** Components Required for CHNS/O Determinations

| Reference | Component | Characteristic | Determination | Filling material |
|---|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | CHNS | Quartz Wool<br>Copper Oxide<br>Electrolytic Copper |
| C1 | Crucible | *Material:* Quartz | --- | --- |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | CHNS | --- |
| R2 | Reactor | *Material:* Quartz | Oxygen | Quartz Wool<br>Nickel Plated Carbon<br>Quartz Turning |
| F | Adsorption filter | *Material:* Pyrex™ | Oxygen | Quartz Wool<br>Soda Lime<br>Magnesium Perchlorate (Anhydrone) |
| CC2 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 1 m<br>*Diameter:* 6 × 5 mm | Oxygen | --- |
| C2 | Crucible | *Material:* Quartz | ---- | --- |

**Tip** **Vanadium pentoxide** ($V_2O_5$) is an oxygen donor. According to the sample nature, it is suggested to insert in the tin container with the sample, for a proper oxidation of material and consequently a quantitative sulfur determination.

**Tip** In some cases, it is suggested to use **nickel wool** in the top layer above the **nickel plated carbon** instead of quartz wool.

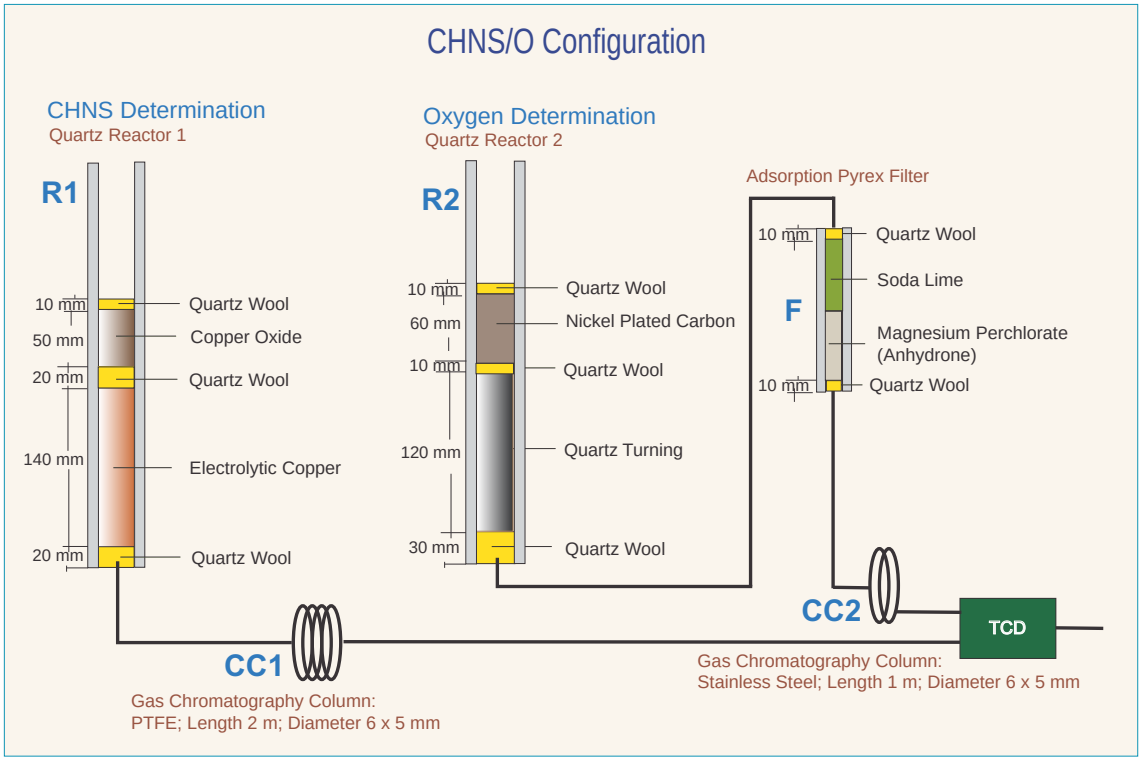
## CHNS Determination

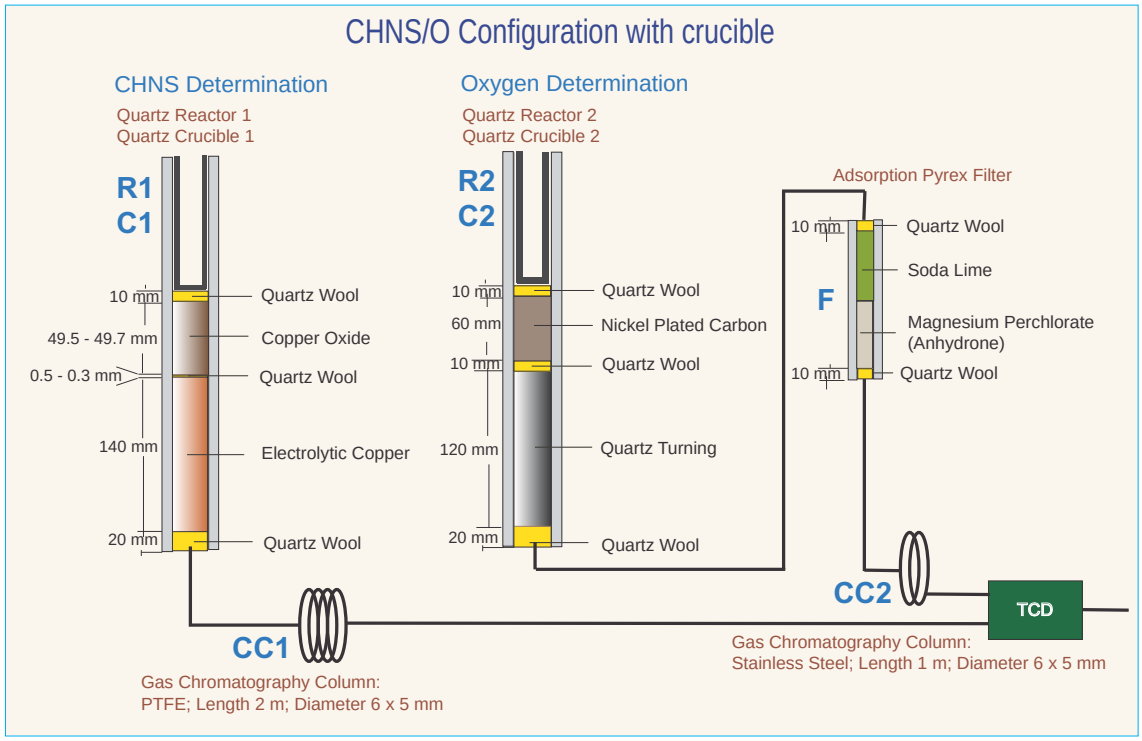
**Tip** If the sample has a high presence of inorganic material, we suggest to insert the **quartz crucible** into the oxidation/reduction reactor. In this case, it is necessary to reduce the **quartz wool** between **copper oxide** and **electrolytic copper** to obtain a thin layer and to reduce the **copper oxide** proportionally.

## Oxygen Determination

**Tip** If the sample has a 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 the **quartz wool** from **30 mm** to **20 mm** in the lower section of the reactor.

## Size of the Filling Material



# CHNS/O Configuration MultiValve Control (MVC) Module

Table 8-10 lists the characteristics of the components required for **CHNS/O determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filter.

**Table 8-11.** Components Required for CHNS/O Determinations

| Reference | Component | Characteristic | Determination | Filling material |
|---|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | CHNS | Quartz Wool<br>Copper Oxide<br>Electrolytic Copper |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | CHNS | --- |
| R2 | Reactor | *Material:* Quartz | Oxygen | Quartz Wool<br>Nickel Plated Carbon<br>Quartz Turning |
| F | Adsorption filter | *Material:* Pyrex™ | Oxygen | Quartz Wool<br>Soda Lime<br>Magnesium Perchlorate (Anhydrone) |
| CC2 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 1 m<br>*Diameter:* 6 × 5 mm | Oxygen | --- |
| C1 | Crucible | *Material:* Quartz | --- | --- |
| C2 | Crucible | *Material:* Quartz | ---- | --- |

**Tip** **Vanadium pentoxide** ($V_2O_5$) is an oxygen donor. According to the sample nature, it is suggested to insert in the tin container with the sample, for a proper oxidation of material and consequently a quantitative sulfur determination.

**Tip** In some cases, it is suggested to use **nickel wool** in the top layer above the **nickel plated carbon** instead of quartz wool.

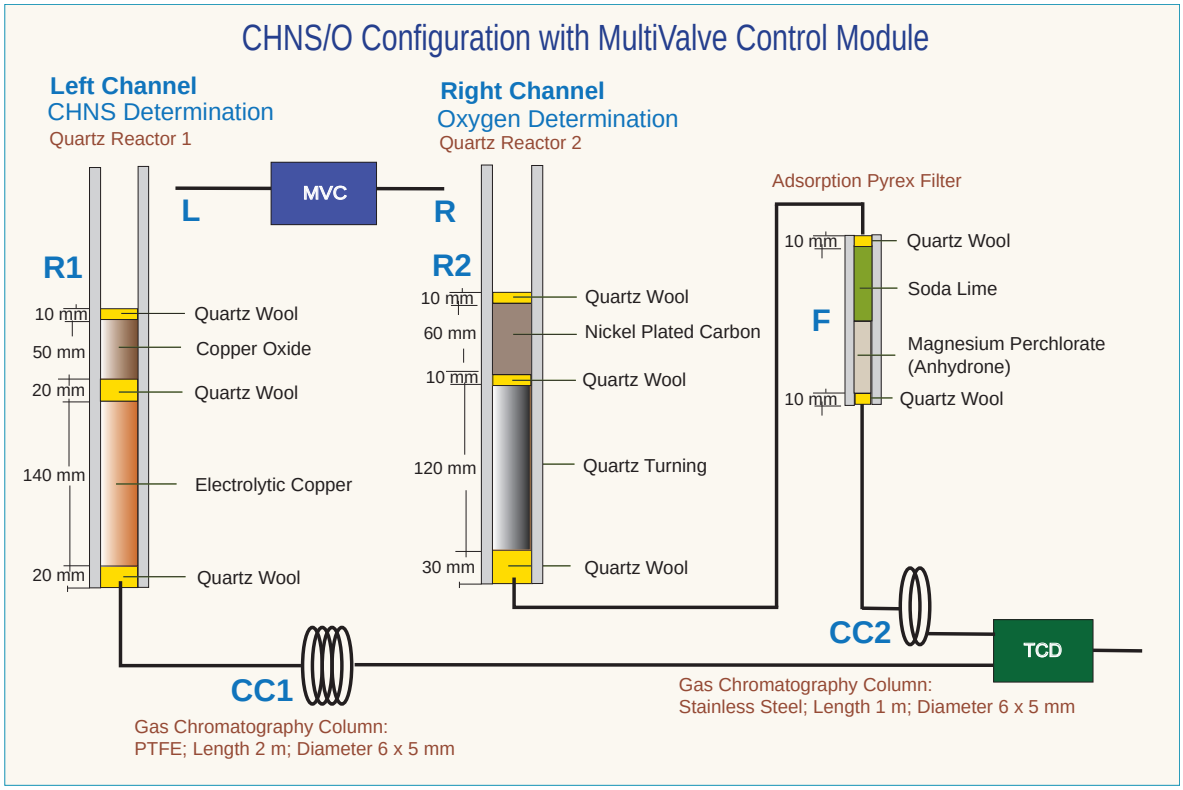
## CHNS Determination

**Tip** If the sample has a 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.

## Oxygen Determination

**Tip** If the sample has a 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 the **quartz wool** from **30 mm** to **20 mm** in the lower section of the reactor.

### Size of the Filling Material




**Tip** The crucible can be used as in the CHNS/O Configuration.

# S (Sulfur) Configuration

Table 8-12 lists the characteristics of the components required for **S determination**, and the type and size of filling materials for a proper preparation of reactor and adsorption filter.

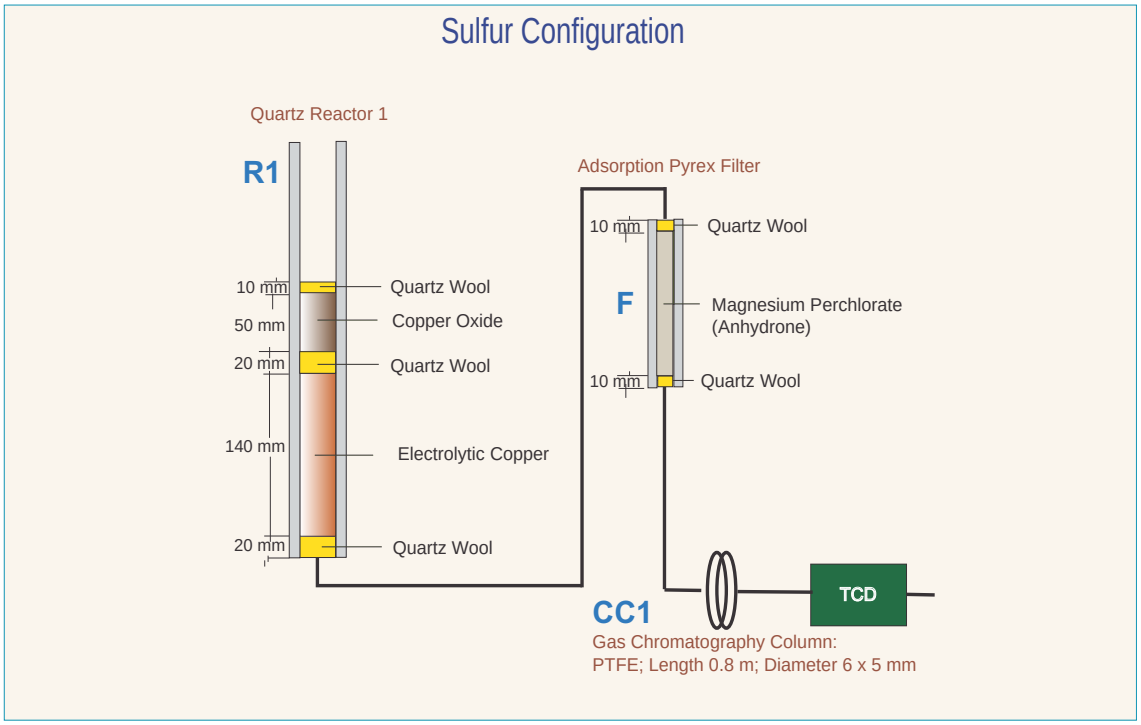
**Table 8-12.** Components Required for S (Sulfur) Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Oxide<br>Electrolytic Copper |
| C1 | Crucible | *Material:* Quartz | |
| F | Adsorption filter | *Material:* Pyrex™ | Quartz Wool<br>Magnesium Perchlorate (Anhydrone) |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 0.8 m<br>*Diameter:* 6 × 5 mm | --- |

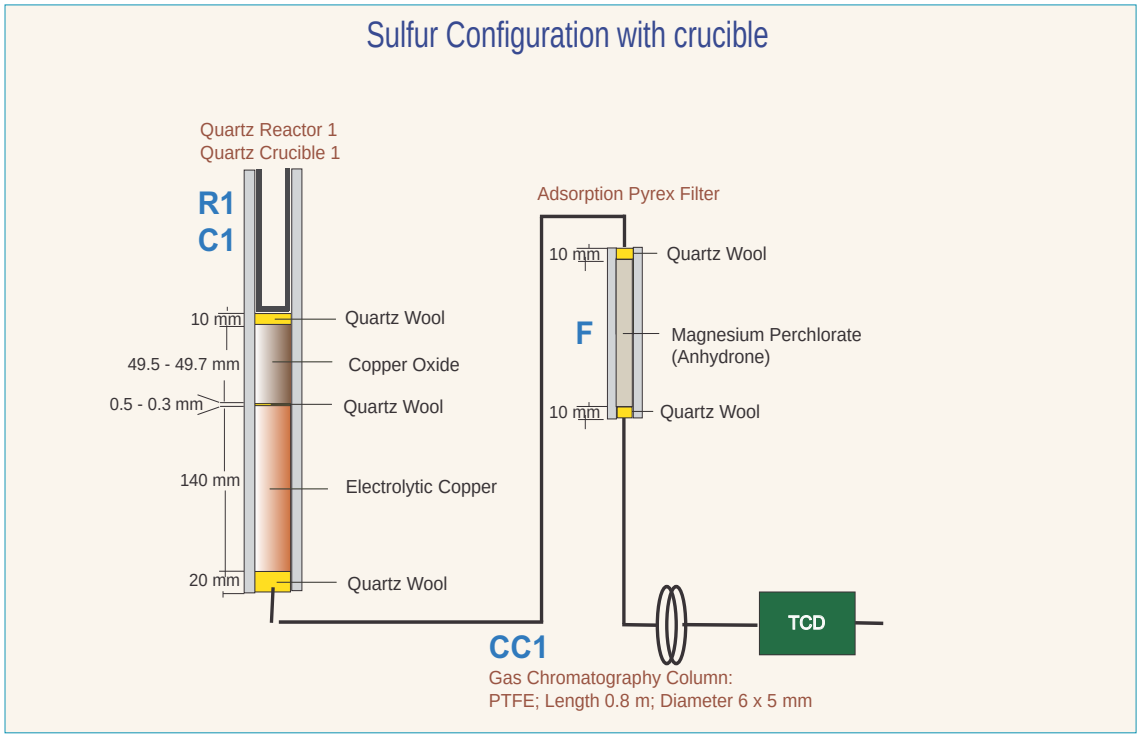
**Tip** If the sample has a high presence of inorganic material, we suggest to insert the **quartz crucible** into the oxidation/reduction reactor. In this case, it is necessary to reduce the **quartz wool** between **copper oxide** and **electrolytic copper** to obtain a thin layer and to reduce the **copper oxide** proportionally.

**Tip** **Vanadium pentoxide** ($V_2O_5$) is an oxygen donor. According to the sample nature, it is suggested to insert in the tin container with the sample for a proper oxidation of material and consequently a quantitative sulfur determination.

## Size of the Filling Material (1)




## Size of the Filling Material (2)



# O (Oxygen) Configuration

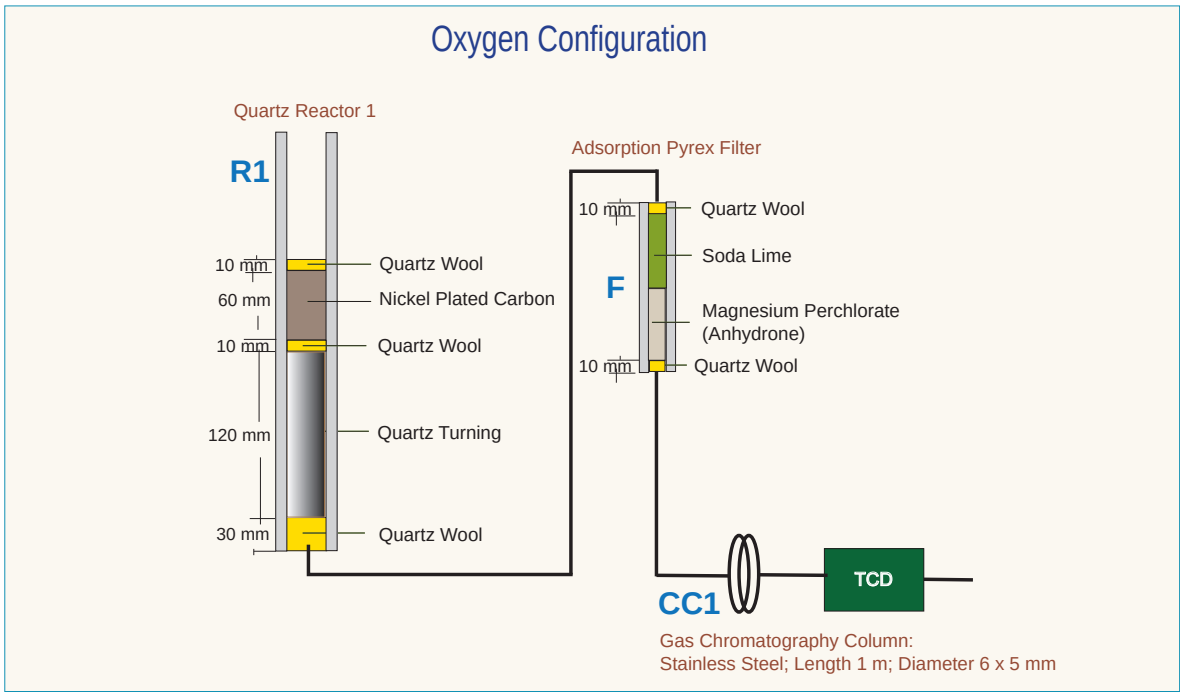
Table 8-13 reports the characteristics of the components required for **O determination**, and the type and size of the filling materials for a proper preparation of reactor and adsorption filter.

**Table 8-13.** Components Required for O (Oxygen) Determinations

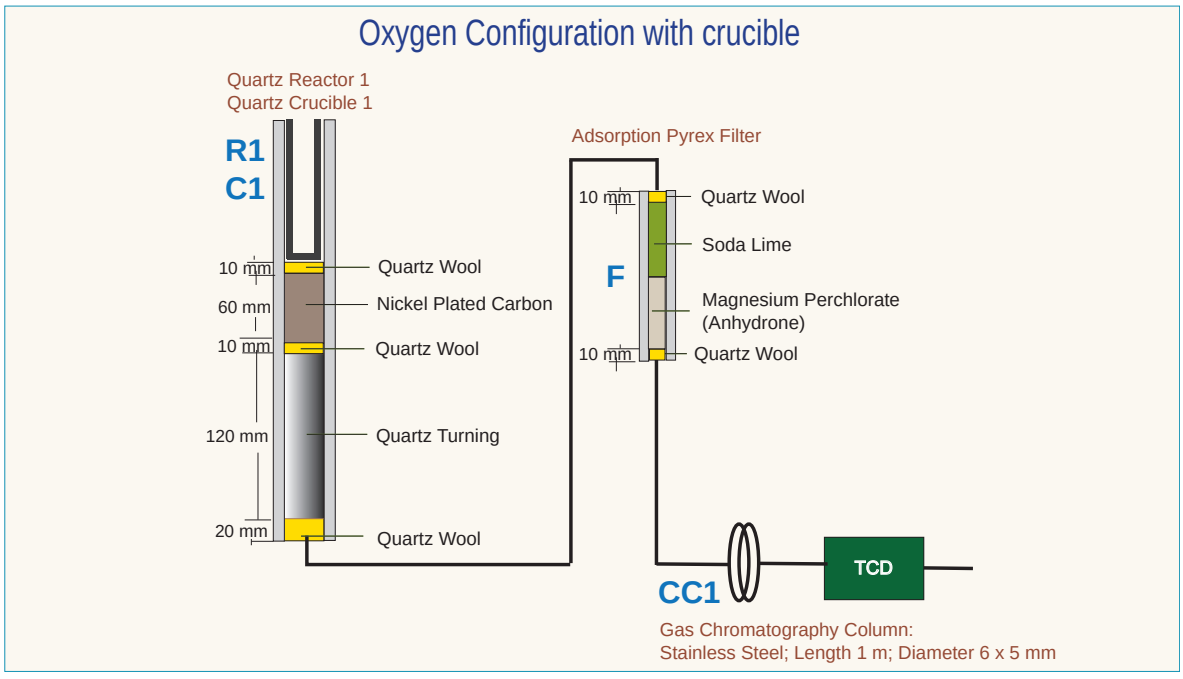
| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Nickel Plated Carbon<br>Quartz Turnings |
| F | Adsorption filter | *Material:* Pyrex™ | Quartz Wool<br>Soda Lime<br>Magnesium Perchlorate (Anhydrone) |
| CC1 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 1 m<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Quartz | |

**Tip** If the sample has a 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 the **quartz wool** from **30 mm** to **20 mm** in the lower section of the reactor.

## Size of the Filling Material (1)




## Size of the Filling Material (2)



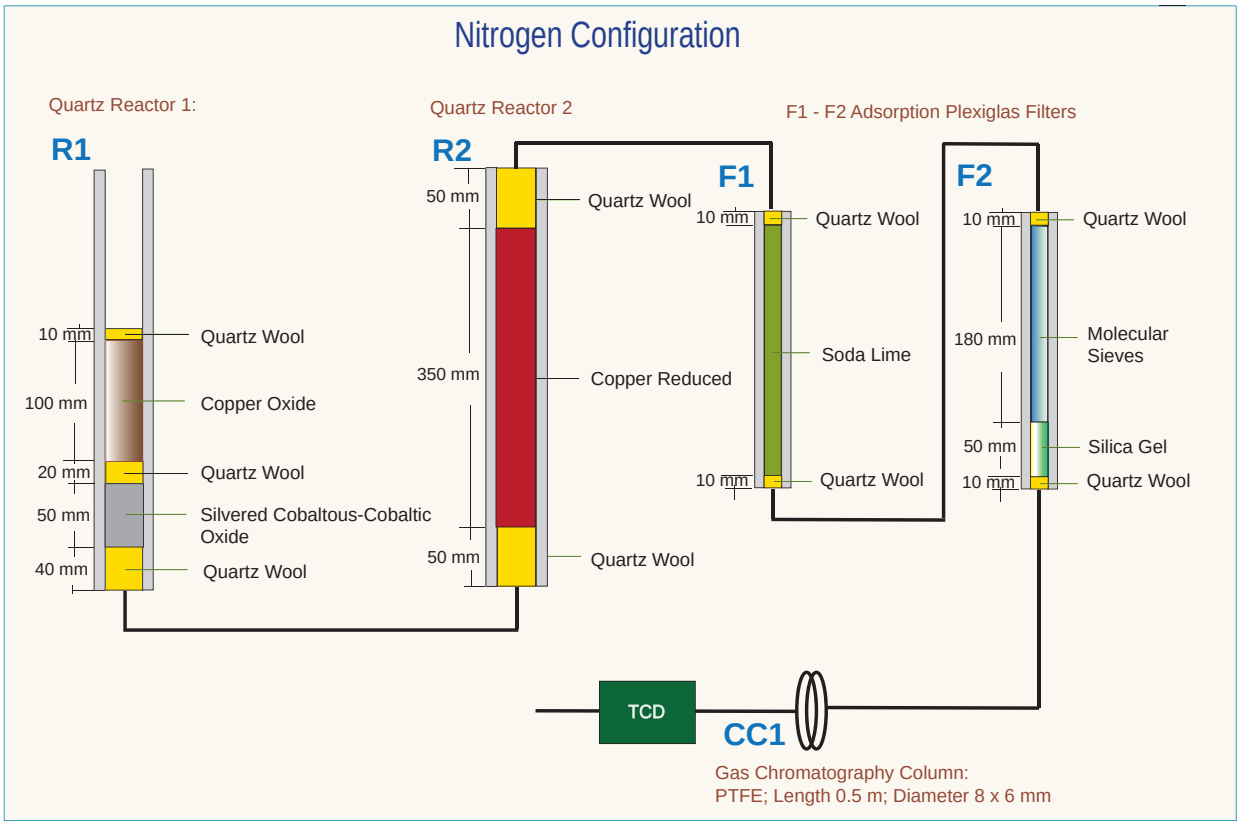
# N (Nitrogen) Configuration

Table 8-14 lists the characteristics of the components required for **N determination,** and the type and size of the filling materials for a proper preparation of reactors and adsorption filters.

**Table 8-14.** Components Required for N (Nitrogen) Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Oxide<br>Silvered Cobaltous/Cobaltic Oxide |
| R2 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Reduced |
| F1 | Adsorption filter | *Material:* Plexiglas™ | Quartz Wool<br>Soda Lime |
| F2 | Adsorption filter | *Material:* Plexiglas | Quartz Wool<br>Molecular Sieves<br>Silica Gel |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 0.5 m<br>*Diameter:* 8 × 6 mm | --- |

**Size of the Filling Material**



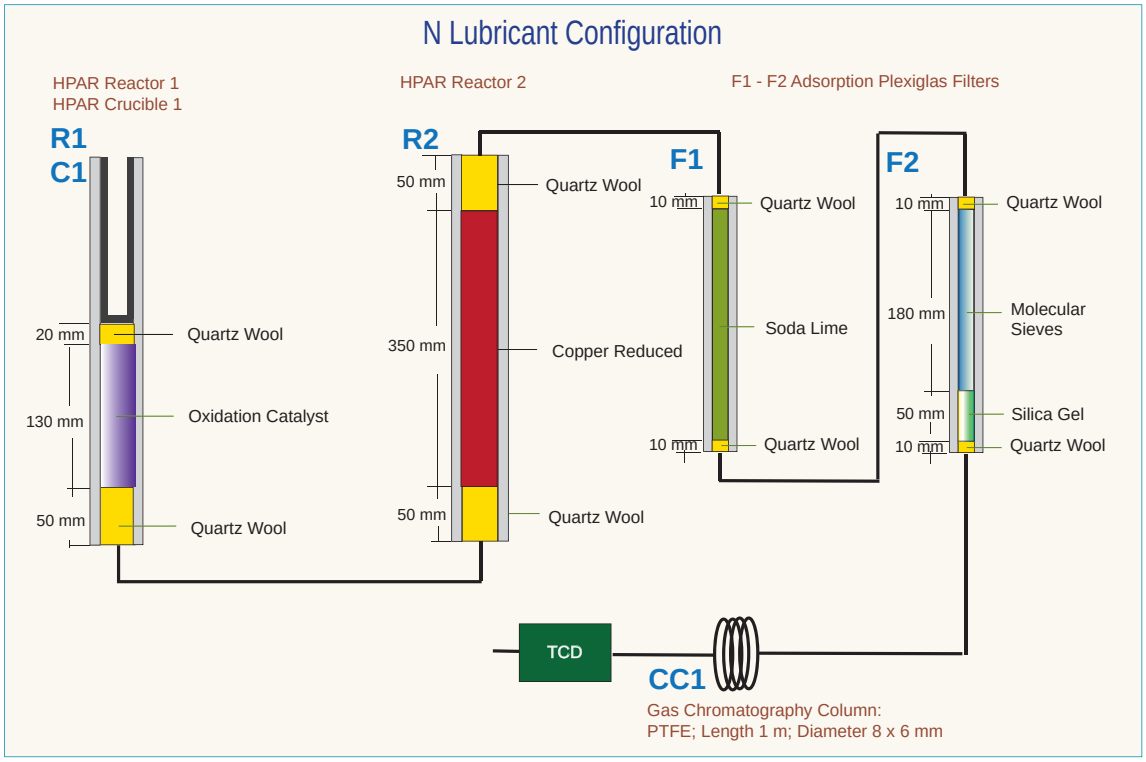
# N Lubricant Configuration

Table 8-15 lists the characteristics of the components required for **N Lubricant** determination, and the type and size of the filling materials for a proper preparation of reactors and adsorption filters.

**Table 8-15.** Components Required for N Lubricant Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor (See note below) | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Oxidation Catalyst |
| R2 | Reactor | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Copper Reduced |
| F1 | Adsorption filter | *Material:* Plexiglas™ | Quartz Wool<br>Soda Lime |
| F2 | Adsorption filter | *Material:* Plexiglas | Quartz Wool<br>Molecular Sieves<br>Silica Gel |
| CC | Gas chromatographic column | *Material:* PTFE<br>*Length:* 1 m<br>*Diameter:* 8 × 6 mm | --- |
| C1 | Crucible | *Material:* Alloy Steel (HPAR) | -- |
| **Tip** R1 combustion reactor requires the use of a crucible C1. | | | |

## Size of the Filling Material



# NC Configuration

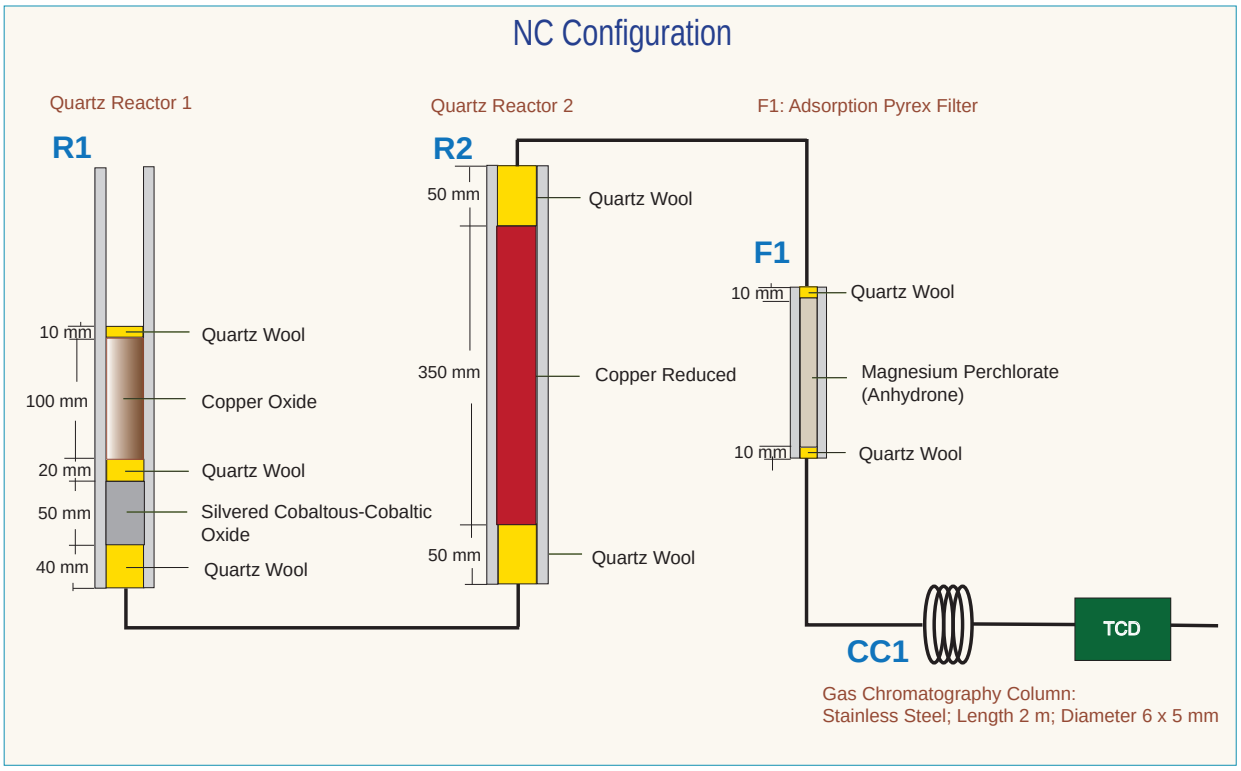
Table 8-16 lists the characteristics of the components required for **NC determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filter.

**Table 8-16.** Components Required for NC Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Oxide<br>Silvered Cobaltous/Cobaltic Oxide |
| R2 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Reduced |
| F1 | Adsorption filter | *Material:* Pyrex™ | Quartz wool<br>Magnesium perchlorate (Anhydrone) |
| CC1 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | --- |

**Tip** R1 combustion reactor requires the use of a crucible C1.

**Size of the Filling Material**



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# NCS Configuration

Table 8-17 lists the characteristics of the components required for **NCS determination**, and the type and size of the filling materials for a proper preparation of reactor and adsorption filter.

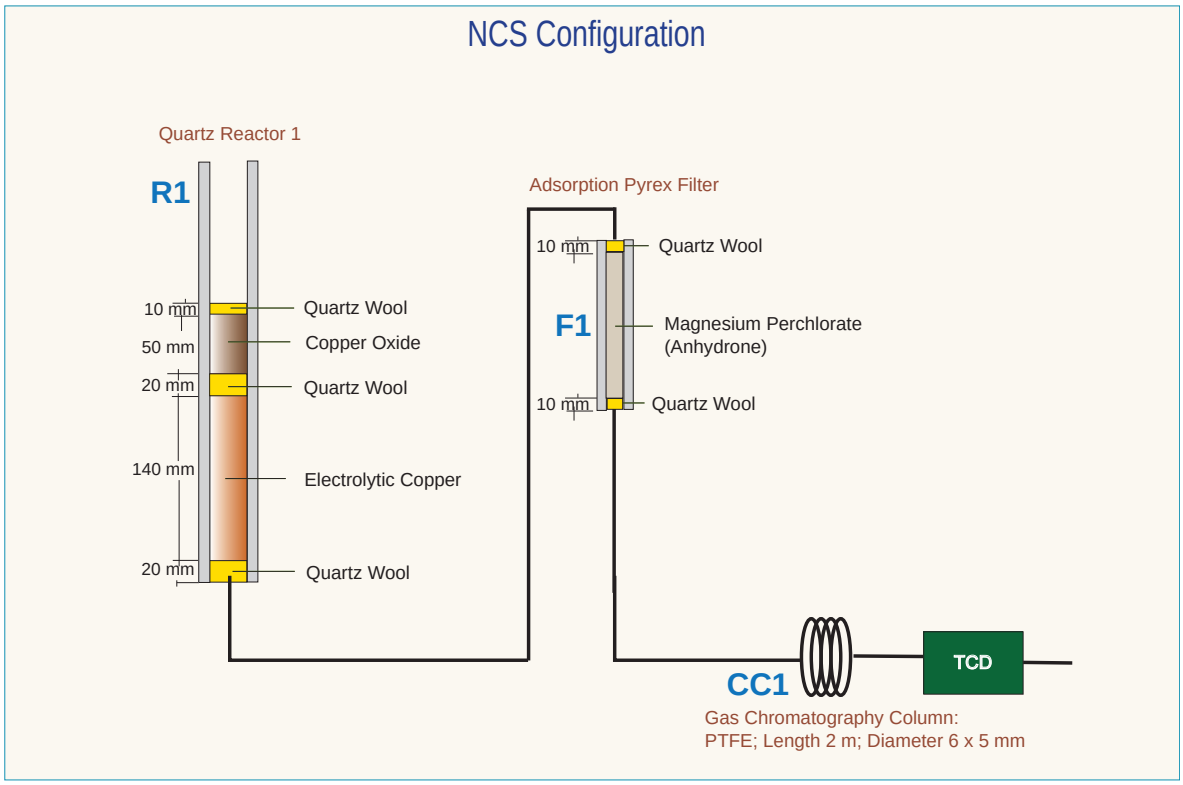
**Table 8-17.** Components Required for NCS Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Oxide<br>Electrolytic Copper |
| C1 | Crucible | *Material:* Quartz | --- |
| F1 | Adsorption filter | *Material:* Pyrex™ | Quartz wool<br>Magnesium Perchlorate (Anhydrone) |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | --- |

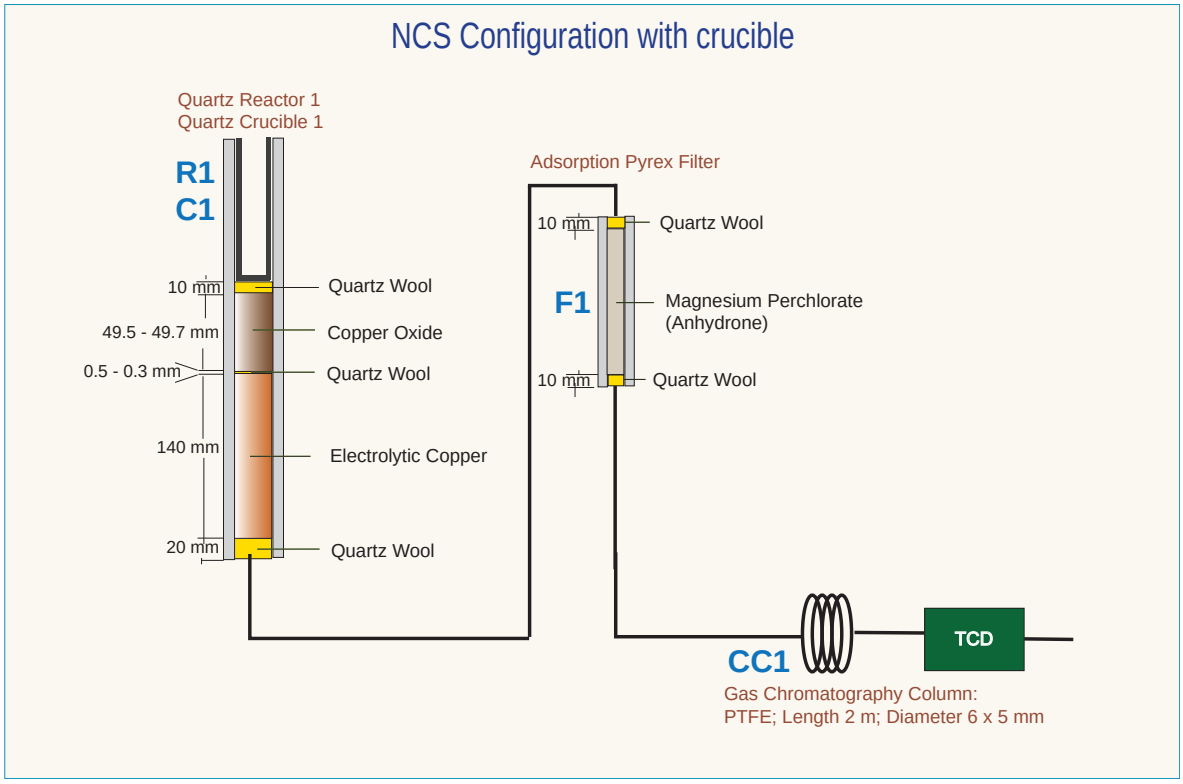
**Tip** **Vanadium pentoxide** ($V_2O_5$) is an oxygen donor. According to the sample nature, it is suggested to insert in the tin container with the sample for a proper oxidation of material and consequently a quantitative sulfur determination.

**Tip** If the sample has a 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** to obtain a thin layer and to reduce the **copper oxide** proportionally.

## Size of the Filling Material (1)




## Size of the Filling Material (2)



# NC Soils Configuration (Double Reactor)

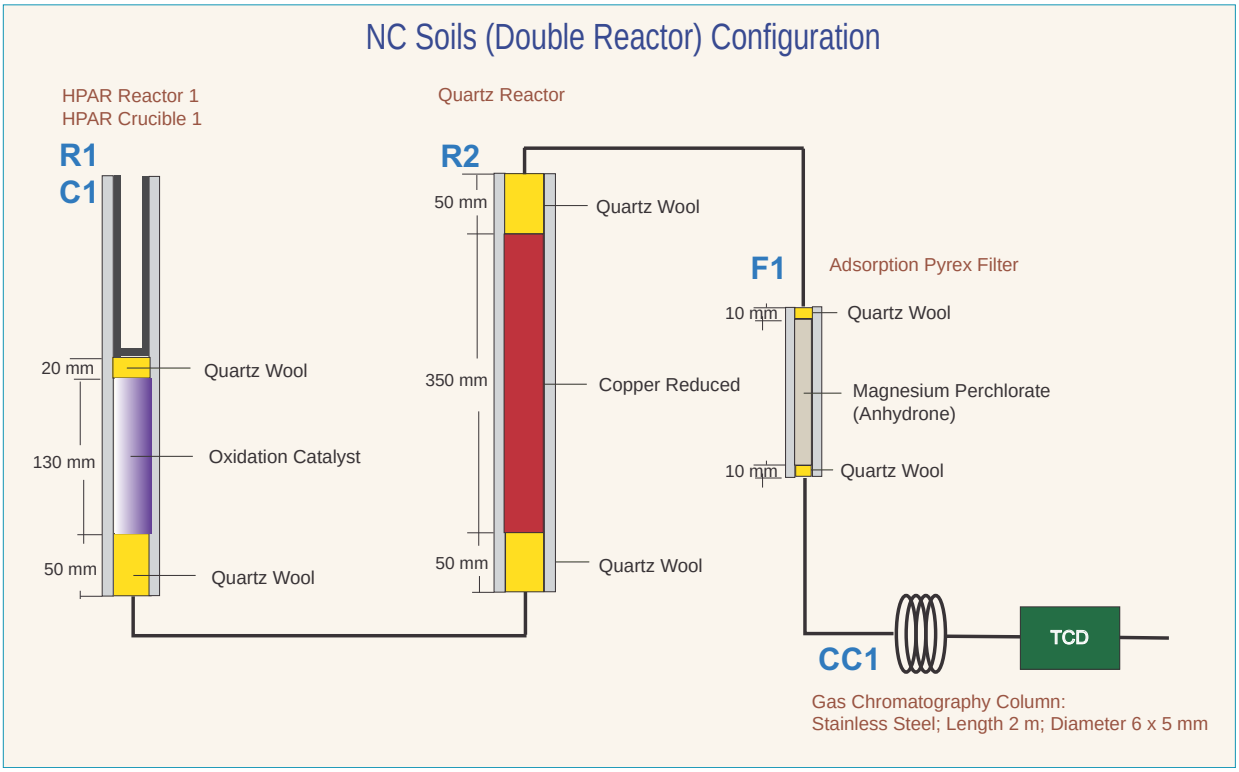
Table 8-18 lists the characteristics of the components required for **NC Soils (Double Reactor) determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filter.

**Table 8-18.** Components Required for NC Soils (Double Reactor) Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor (See note below) | *Material:* Alloy steel (HPAR) | Quartz Wool<br>Oxidation Catalyst |
| R2 | Reactor | *Material:* Quartz | Quartz Wool<br>Copper Reduced |
| F1 | Adsorption filter | *Material:* Pyrex™ | Quartz Wool<br>Magnesium Perchlorate (Anhydrone) |
| CC1 | Gas chromatographic column | *Material:* Stainless Steel<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Alloy Steel (HPAR) | --- |

**Tip** R1 combustion reactor requires the use of a crucible C1.

**Size of the Filling Material**



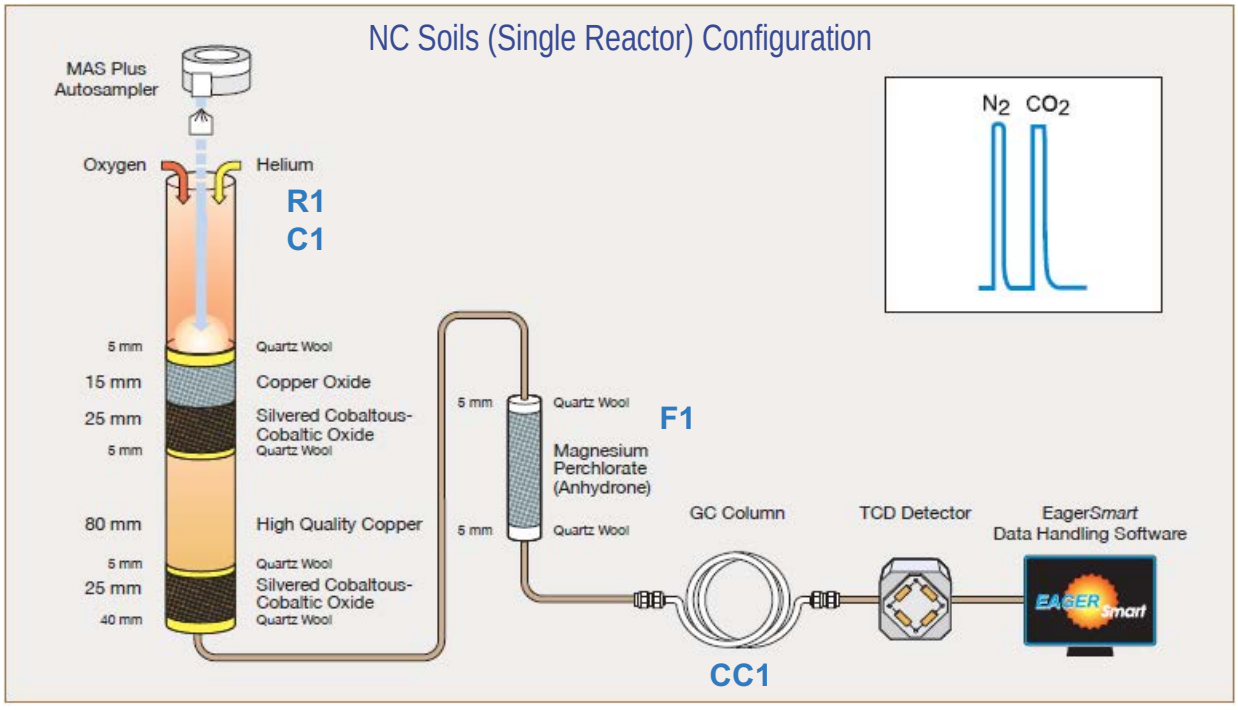
# NC Soils Configuration (Single Reactor)

Table 8-22 lists the characteristics of the components required for **NC Soils (Single Reactor) determination**, and the type and size of the filling materials for a proper preparation of reactor and adsorption filter.

**Table 8-19.** Components Required for NC Soils (Single Reactor) Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Copper Oxide<br>Silver Cobaltous - Cobaltic Oxide<br>High Quality Copper |
| F1 | Adsorption filter | *Material:* Plexiglas™ | Quartz Wool<br>Magnesium Perchlorate (Anhydrone) |
| CC1 | Gas chromatographic column | *Material:* Stainless steel<br>*Length:* 2 m<br>*Diameter:* 6 × 5 mm | --- |
| C1 | Crucible | *Material:* Alloy Steel (HPAR) | --- |

**Size of the Filling Material**



# N/Protein Configuration (Double Reactor)

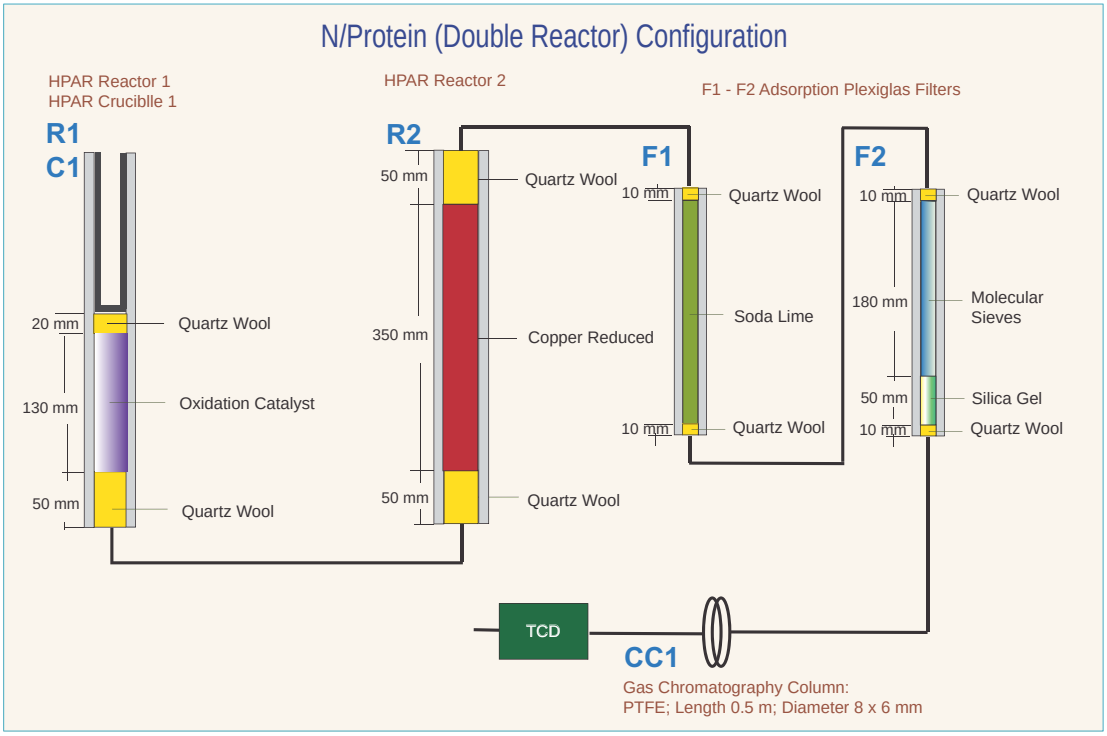
Table 8-20 lists the characteristics of the components required for **N/Protein (Double Reactor) determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filters.

**Table 8-20.** Components Required for N/Protein (Double Reactor) Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor (See note below) | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Oxidation Catalyst |
| R2 | Reactor | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Copper Reduced |
| F1 | Adsorption filter | *Material:* Plexiglas™ | Quartz Wool<br>Soda Lime |
| F2 | Adsorption filter | *Material:* Plexiglas | Quartz Wool<br>Molecular Sieves<br>Silica Gel |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 0.5 m<br>*Diameter:* 8 x 6 mm | N/Protein |
| C1 | Crucible | | |

**Tip** R1 combustion reactor requires the use of a crucible C1.

## Size of the Filling Material



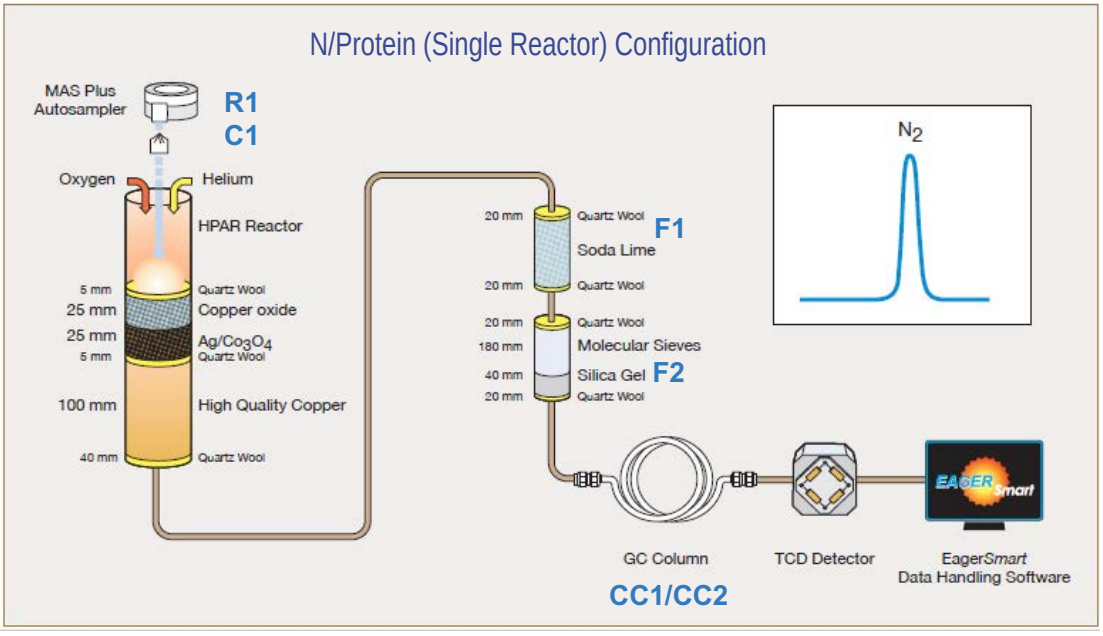
# N/Protein Configuration (Single Reactor)

Table 8-22 lists the characteristics of the components required for **N/Protein (Single Reactor) determination**, and the type and size of the filling materials for a proper preparation of reactor and adsorption filters.

**Table 8-21.** Components Required for N/Protein (Single Reactor) Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Copper Oxide<br>Silvered Cobaltous/Cobaltic Oxide<br>High Quality Copper |
| F1 | Adsorption filter | *Material:* Plexiglas™ | Quartz Wool<br>Soda Lime |
| F2 | Adsorption filter | *Material:* Plexiglas | Quartz Wool<br>Molecular Sieves<br>Silica Gel |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 1 m<br>*Diameter:* 8 × 6 mm | --- |
| C1 | Crucible | *Material:* Alloy Steel (HPAR) | --- |

**Size of the Filling Material**



# N/Brew Configuration

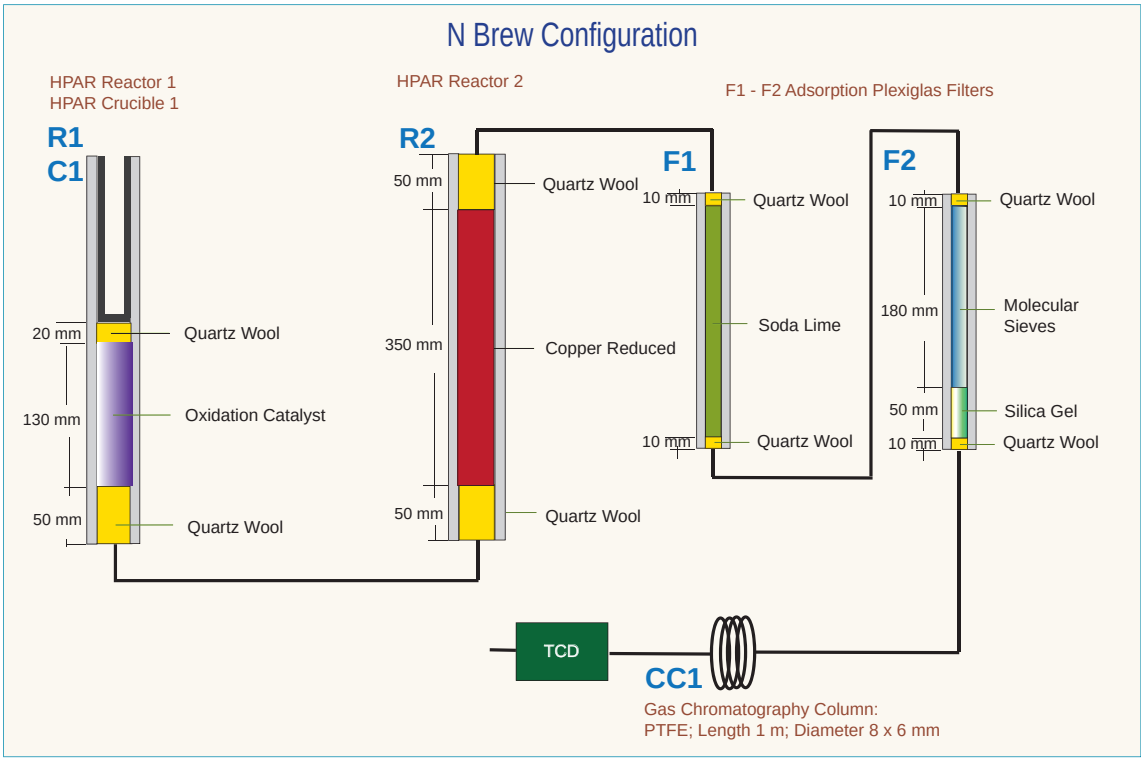
Table 8-22 lists the characteristics of the components required for **N/Brew determination**, and the type and size of the filling materials for a proper preparation of reactors and adsorption filters.

**Table 8-22.** Components Required for N Brew Determinations

| Reference | Component | Characteristic | Filling materials |
|---|---|---|---|
| R1 | Reactor (See note below) | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Oxidation Catalyst |
| R2 | Reactor | *Material:* Alloy Steel (HPAR) | Quartz Wool<br>Copper Reduced |
| F1 | Adsorption filter | *Material:* Plexiglas™ | Quartz Wool<br>Soda Lime |
| F2 | Adsorption filter | *Material:* Plexiglas | Quartz Wool<br>Molecular Sieves<br>Silica Gel |
| CC1 | Gas chromatographic column | *Material:* PTFE<br>*Length:* 1 m<br>*Diameter:* 8 × 6 mm | N-Brew |
| C1 | Crucible | | --- |

**Tip** R1 combustion reactor requires the use of a crucible C1.

## 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, see the filling diagram of the concerned instrument configuration, as described in "Introduction to the Preparation of Reactors and Filters" on page 8-6.

Remember that:

- all reactors have a conical bottom end.
- alloy steel reactors (HPAR) have the upper end provided with two through-holes.

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

The filling of reactors requires the use of quartz wool. When you handle quartz wool, we recommend that you wear gloves and face protection.

**NOTICE**

Always use original Thermo Fisher Scientific materials and products. The use of materials that do not meet the technical specifications of our products does not ensure a good operation of the instrument and might even cause damage to it.

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

- "Filling the Quartz Reactor" on page 8-35
- "Filling the Alloy Steel (HPAR) Reactor" on page 8-38
- "Preparing the Crucible" on page 8-40

## Filling the Quartz Reactor

❖ **To fill the quartz reactor**

| Material Required |
|---|
| Quartz reactor |
| Compression rod |
| Filling material |

1. Starting from the reactor conical bottom end, introduce a sufficient amount of quartz wool to form the required layer, as shown in Figure 8-1.

   **Tip** 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.

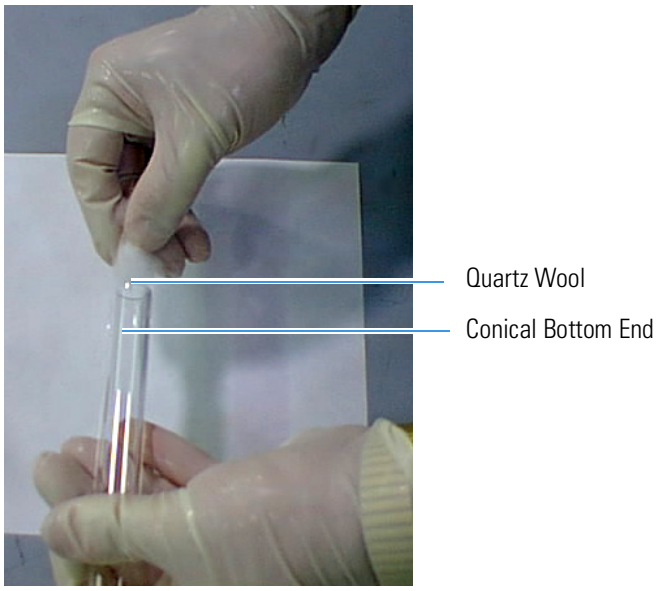



**Figure 8-1.** Introduction of Quartz Wool into the Conical End of the Reactor

2. Plug with your finger the mouth of the reactor conical end. Gently press the quartz wool with the provided rod, as shown in Figure 8-2.

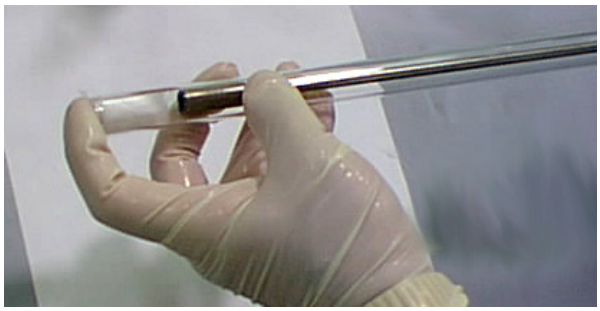


**Figure 8-2.** Compression of Quartz Wool into the Quartz Reactor

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 8-3, ensuring that each layer has the indicated size. At each step, gently press the quartz wool with the provided rod.

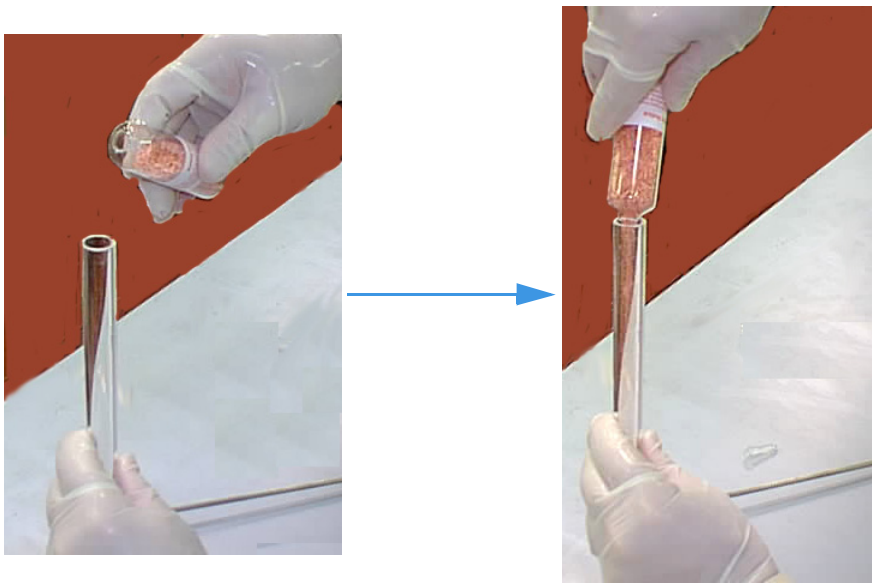


**Figure 8-3.** Filling of the Quartz Reactor

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 8-4.

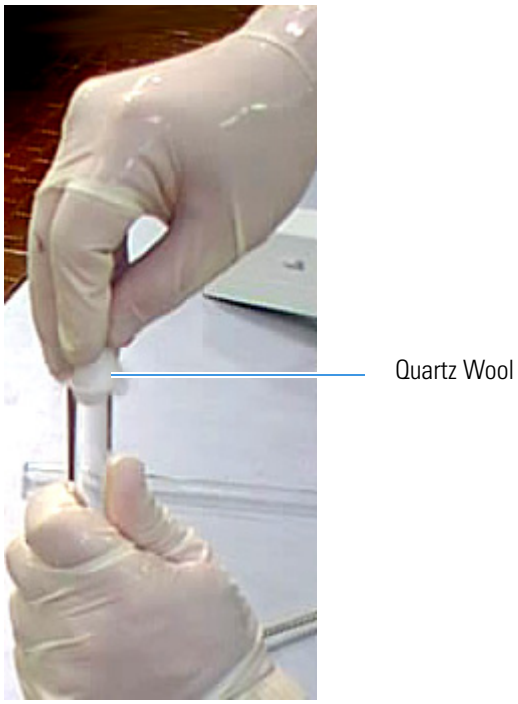



**Figure 8-4.** Introduction of Quartz Wool as Last Layer of the Sequence

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

# Filling the Alloy Steel (HPAR) Reactor

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

❖ **To fill the alloy steel reactor (HPAR)**

| Material required |
|---|
| Alloy steel reactor (HPAR) |
| Compression rod |
| Filling materials |

1. Introduce into the bottom end of the reactor a sufficient amount of quartz wool to form the required layer, as shown in Figure 8-5.

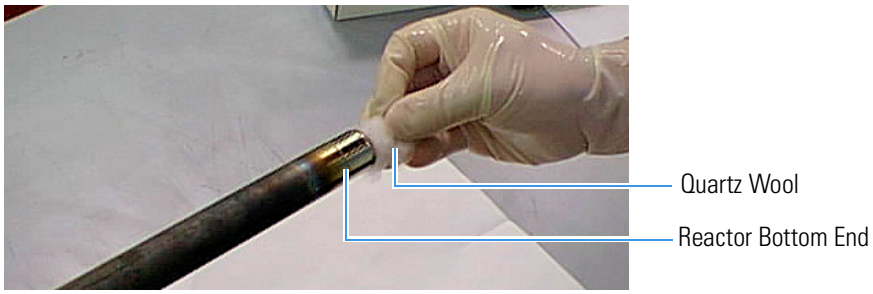



**Figure 8-5.** Introduction of Quartz Wool into the Bottom End of the Reactor

2. Plug with your finger the mouth of the reactor bottom end. Gently press the quartz wool with the provided rod, as shown in Figure 8-6.

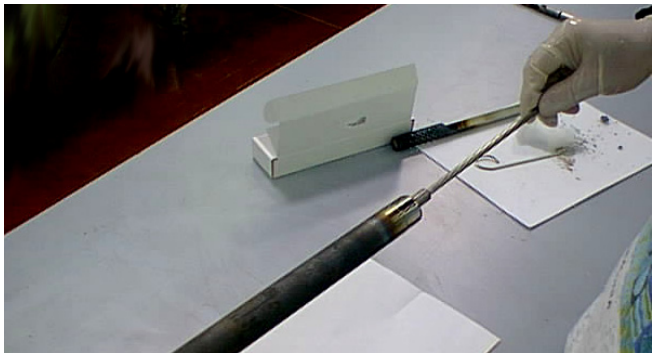


**Figure 8-6.** Compression of Quartz Wool into the Alloy Steel (HPAR) Reactor

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

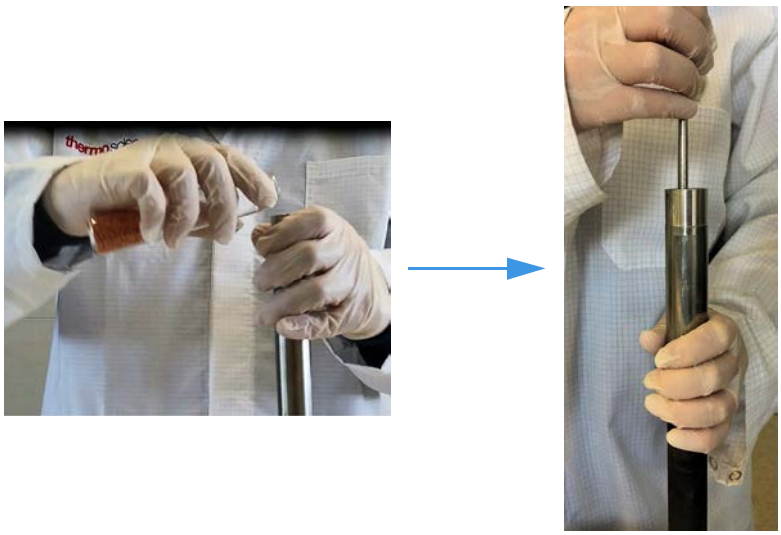


**Figure 8-7.** Filling of the Alloy Steel (HPAR) Reactor and Compression of Materials

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

   **Tip** When the oxidation catalyst is used, it must be introduced into the reactor homogeneously. See Table 8-2 on page 8-5.

   At each step, gently press the quartz wool with the provided rod.

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 8-8.

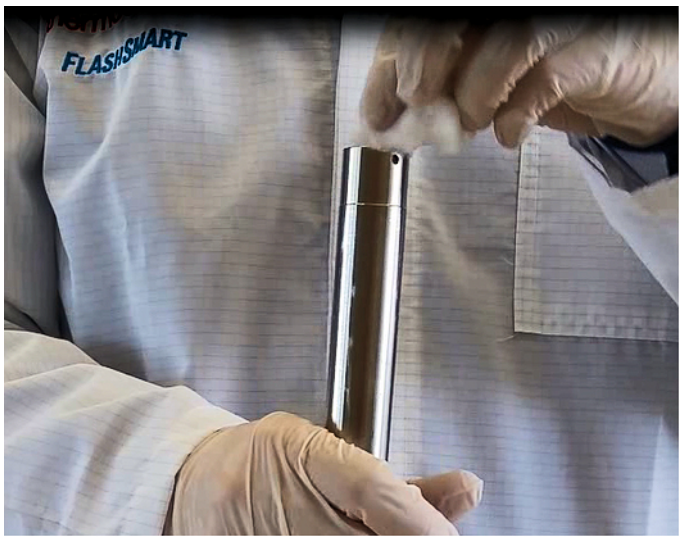



**Figure 8-8.** Introduction of Quartz Wool as Last Layer of the Sequence

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

# Preparing the Crucible

The following instructions describe the preparation of the crucible you will use with the alloy steel reactor (HPAR), which is required for combustion.

❖ **To prepare the crucible**

| Material required |
| --- |
| Quartz wool |
| Compression rod |

1. Hold the crucible as shown in Figure 8-9. Introduce into the bottom end of the crucible a sufficient quantity of quartz wool to form a 1 cm layer.

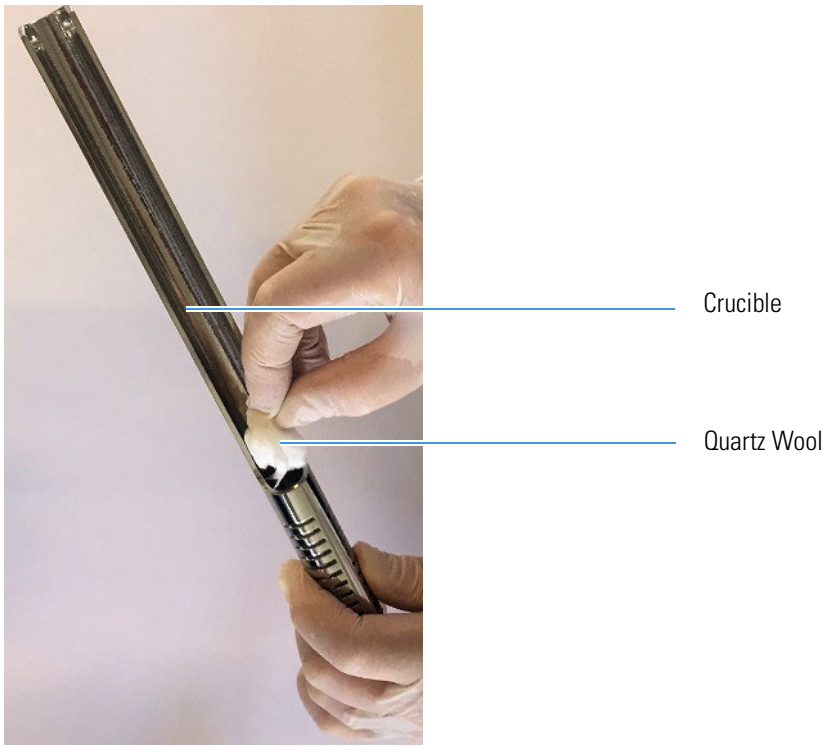



**Figure 8-9.** Introduction of Quartz Wool into the Crucible

2. Gently press the quartz wool with the provided rod, as shown in Figure 8-10.

**Figure 8-10.** 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 "Introduction to the Preparation of Reactors and Filters" on page 8-6.

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

- a large filter (Plexiglas™)
- a small filter (Pyrex™)

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

The filling of reactors requires the use of quartz wool. When you handle quartz wool, we recommend that you wear gloves and face protection.

**NOTICE**

Always use original Thermo Fisher Scientific materials and products. The use of materials that do not meet the technical specifications of our products does not ensure a good operation of the instrument and might even cause damage to it.

## Filling the Adsorption Filter

The filling procedure should be carried out on a wide and clean workbench.

❖ **To fill the adsorption filter**

| Material required |
|---|
| Pyrex™ or Plexiglas™ filter according to the instrument configuration |
| Compression rod |
| Filling materials |

1. Introduce into either of the tube ends a sufficient amount of quartz wool to form the required layer as shown in Figure 8-11.

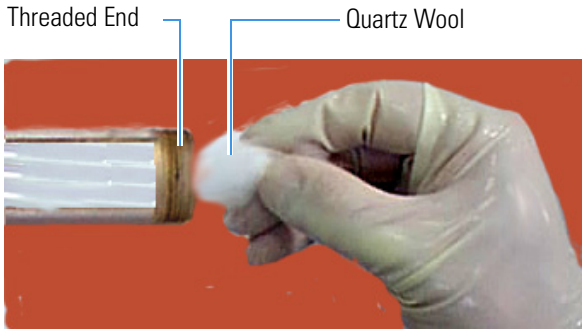



**Figure 8-11.** Introduction of Quartz Wool into the Tube

2. While plugging the tube mouth with your hand, gently press the quartz wool with the provided rod.
3. Screw the nut complete with its seal onto the threaded mouth, as shown in Figure 8-12.

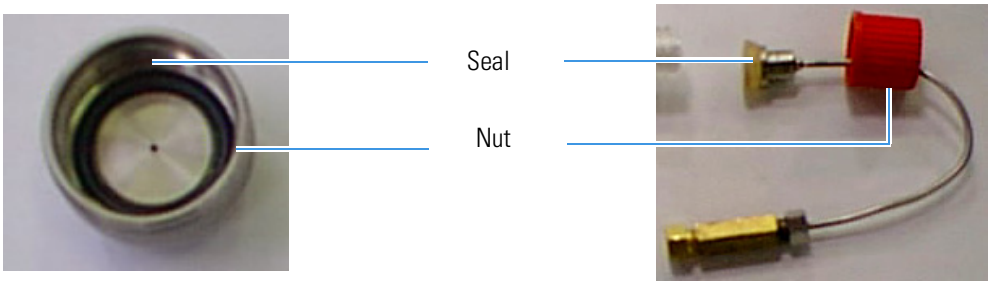



**Figure 8-12.** Nuts and Seals for Adsorption Filters

4. Pour sequentially the required filling materials into the adsorption filter. Make sure that each layer has the indicated size. At each step, gently press the quartz wool with the provided rod.

**NOTICE**

Soda lime must be wet before using. Pour 0.5 mL of water on the soda lime surface on the side that will be connected to the reduction reactor.

5. Do the last layer using a sufficient quantity of quartz wool to form the required layer.
6. Complete the procedure by screwing on the second nut complete with its seal, as shown in Figure 8-13.

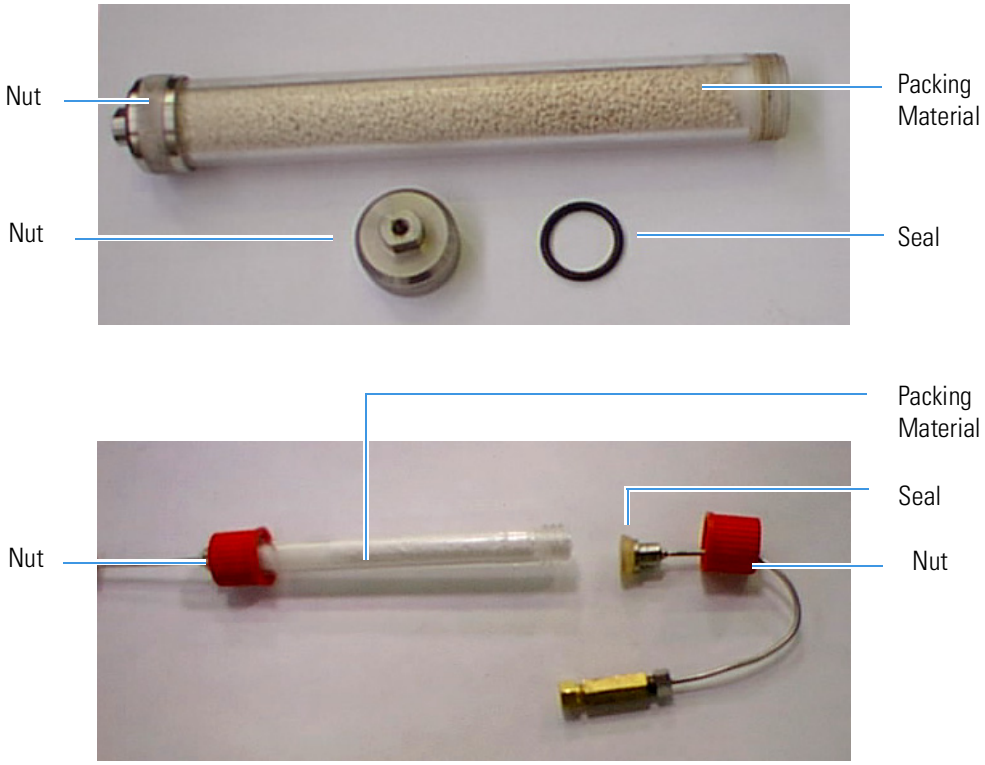



**Figure 8-13.** Preparation of the Adsorption Filters

Figure 8-14 shows the result of the filling operation of an adsorption filter.

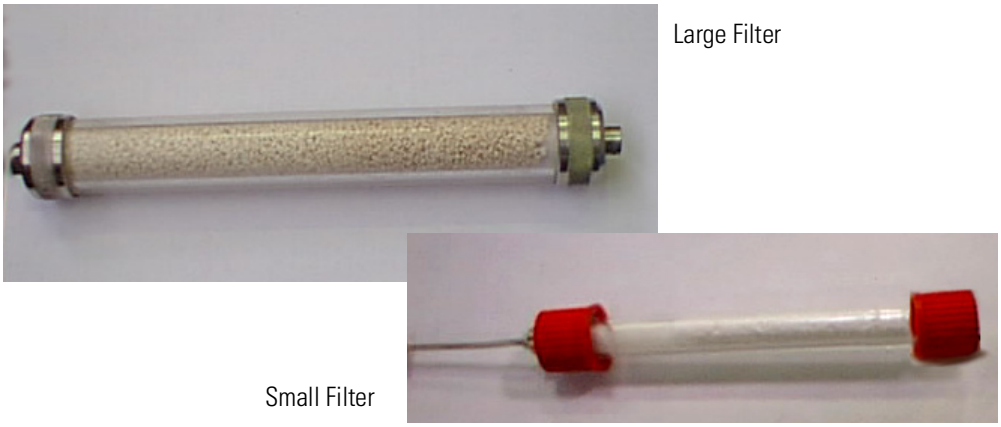



**Figure 8-14.** Result of the Filling of a Large and Small Adsorption Filter

9

# Connecting the Reactors and the Adsorption Filters

This chapter provides the instructions for installing/removing the reactors and the adsorption filters into the Flash*Smart* Elemental Analyzer.

**Contents**

# Installing the Reactors into the Furnaces

Table 9-1 summarizes the type of reactor to be used and the furnace where it must be installed according to your instrument configuration. See "Introduction" on page 8-3.

**Tip** The alloy steel reactors (HPAR) used for combustion require the use of a crucible.

**Table 9-1.** Reactors and Furnaces

| Determination | Left furnace | Right furnace |
|---|---|---|
| CHN | Quartz reactor | --- |
| CHN/CHN | Quartz reactor | Quartz reactor |
| CHN/O | Quartz reactor | Quartz reactor |
| CHNS | Quartz reactor + crucible (quartz) | --- |
| CHNS/CHNS | Quartz reactor + crucible (quartz) | Quartz reactor + crucible (quartz) |
| CHNS/O | Quartz reactor + crucible (quartz) | Quartz reactor |
| S (Sulfur) | Quartz reactor + crucible (quartz) | --- |
| O (Oxygen) | Quartz reactor | --- |
| N (Nitrogen) | Quartz reactor | Quartz reactor |
| N Lubricant | Alloy steel (HPAR) reactor + crucible (HPAR) | Alloy steel (HPAR) reactor |
| NC | Quartz reactor | Quartz reactor |
| NCS | Quartz reactor + crucible (quartz) | --- |
| NC Soils | Alloy steel (HPAR) reactor + crucible (HPAR) | Quartz reactor |
| N/Protein | Alloy steel (HPAR) reactor + crucible (HPAR) | Alloy steel (HPAR) reactor |
| N/Brew | Alloy steel (HPAR) reactor + crucible (HPAR) | Alloy steel (HPAR) reactor |

## Preparing the Installation of the Reactors

❖ **To prepare the installation of the reactors**

1. Make sure that the furnaces are at room temperature.
2. Open the furnaces compartment by lifting the cover and removing the protecting plate, see "Furnace Compartment" on page 3-13.
3. Remove the MAS Plus autosampler, if installed, by manually undoing the fixing nut counter-clockwise. See Figure 9-1.
4. Carefully put the MAS Plus autosampler on the top cover of the instrument.


**Figure 9-1.** Removing the MAS Plus Autosampler

## Installing the Quartz Reactor into the Furnace

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

**NOTICE**

The reactors must be installed with the furnaces at room temperature. Do not use mechanical tools to screw or unscrew the fixing nut.

❖ **To install the quartz reactor into the furnace**

| **Material required** |
| --- |
| O-ring |

1. Remove the fixing nut.
2. Carefully introduce the reactor into the furnace. Make sure that the conical end of the tube is turned downward. See Figure 9-2.

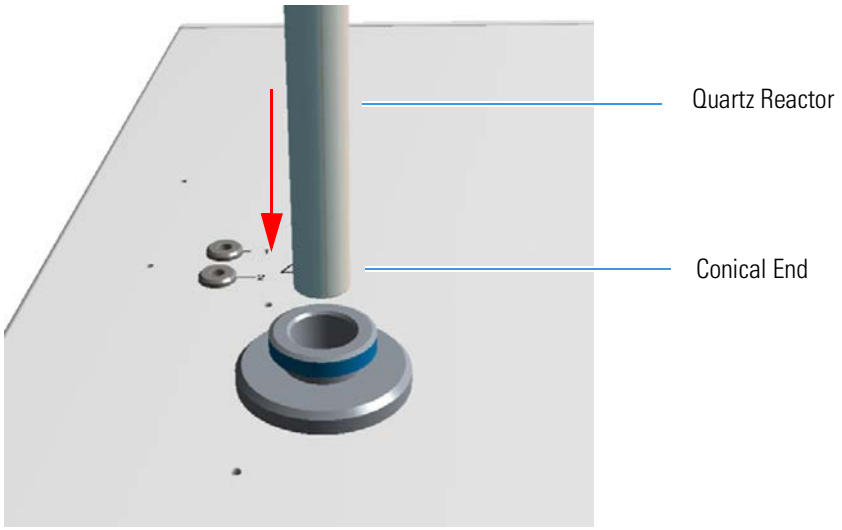



**Figure 9-2.** Introduction of the Quartz Reactor into the Furnace

3. Guide the quartz reactor inside the furnace. The reactor conical end must fit into the coupling union located on the base of the furnaces compartment. See Figure 9-3.

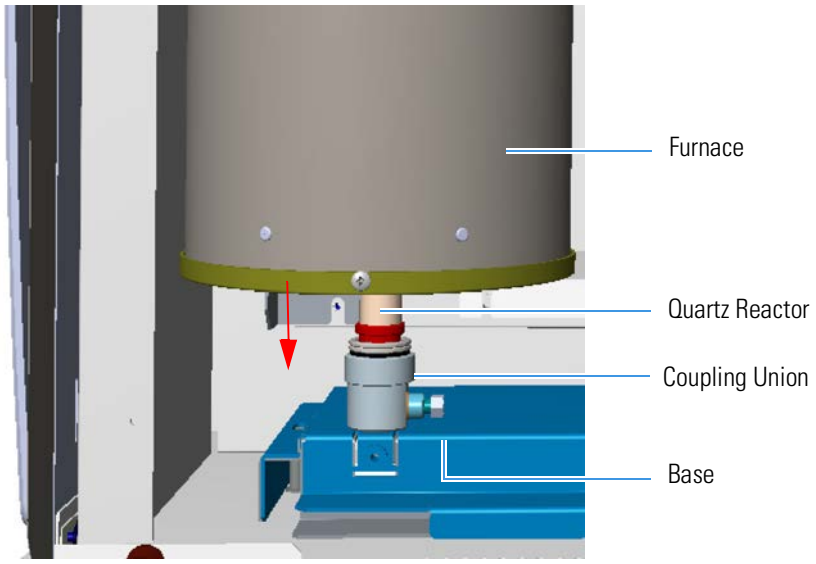



**Figure 9-3.** Driving the Reactor into the Furnace

4. Gently press the edge of the reactor until its introduction is complete. See Figure 9-4.

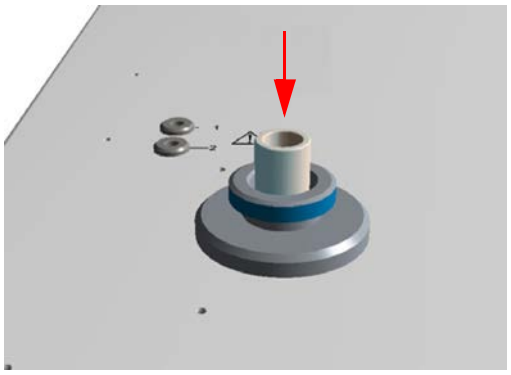


**Figure 9-4.** Complete the Introduction of the Reactor

5. Slip on the O-ring with its conical section turned upwards. See Figure 9-5.

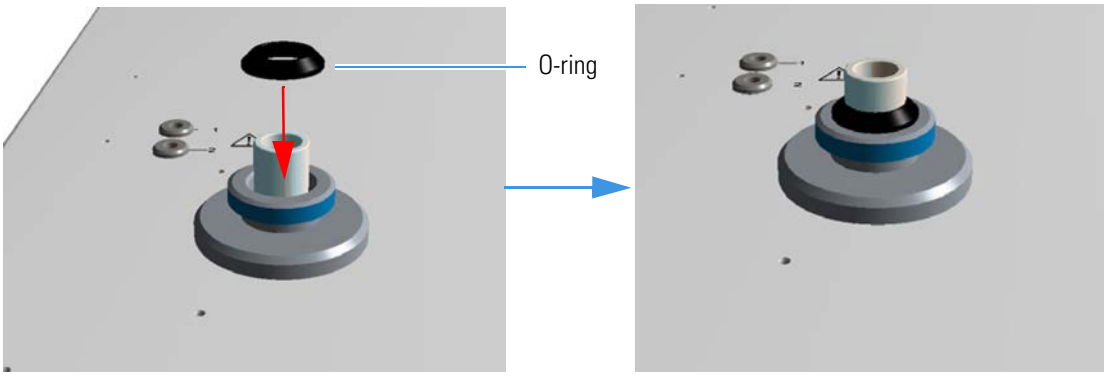



**Figure 9-5.** O-ring

6. Manually screw the autosampler fixing nut.

   **Tip** If it is required by your instrument configuration, install the reactor into the right furnace following the same instructions reported in this operating sequence. The MAS Plus 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 nut of the MAS Plus autosampler if installed.

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

## Installing the Alloy Steel (HPAR) Reactor into the Furnace

The following procedure provides the instructions for installing the alloy steel (HPAR) reactors into the left and right furnaces. The figures in this operating sequence show the installation of an alloy steel (HPAR) reactor into the left furnace.

**NOTICE** The reactors must be installed with the furnaces at room temperature. Do not use mechanical tools to screw or unscrew the fixing nut.

❖ **To install the alloy steel (HPAR) reactor into the furnace**

| Material required |
|---|
| Tool for steel tube |
| O-ring |

1. Remove the fixing nut.

2. Introduce the tool, provided in the standard outfit, into the holes located on the top end of the alloy steel (HPAR) reactor. See Figure 9-6.

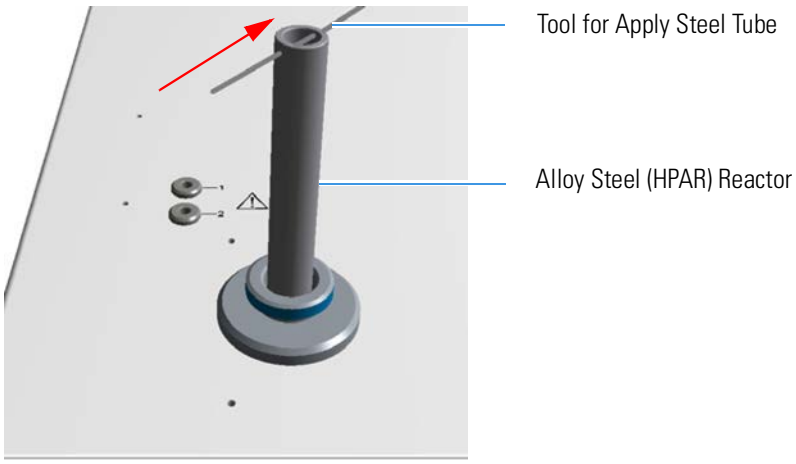



**Figure 9-6.** Introducing the Alloy Steel (HPAR) Reactor Into the Furnace

3. Guide the reactor into the furnace. The conical part should slide into the coupling union located on the base of the furnaces compartment. See Figure 9-7.

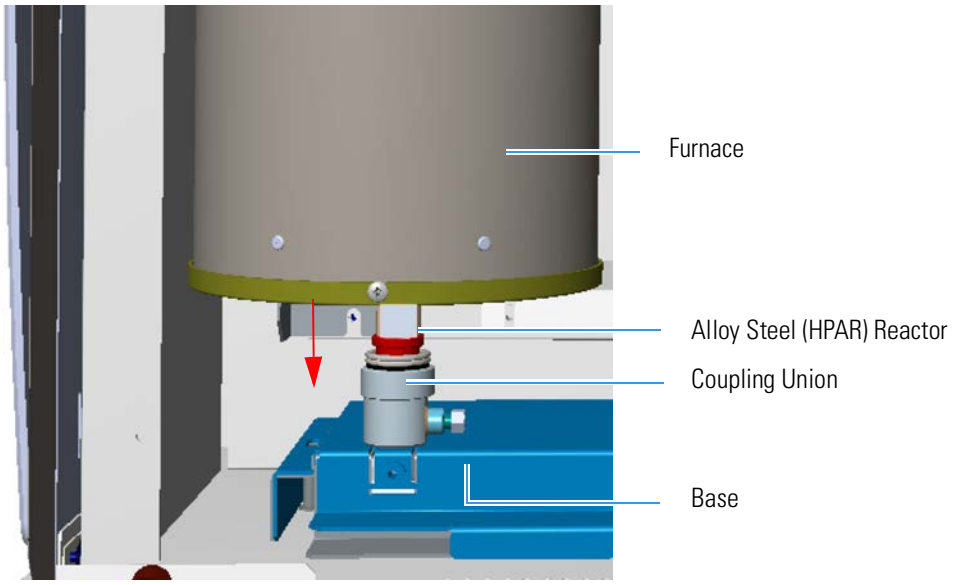



**Figure 9-7.** Driving the Reactor into the Furnace

4. Turn the alloy steel (HPAR) reactor clockwise, and push until completely in place. See Figure 9-8.

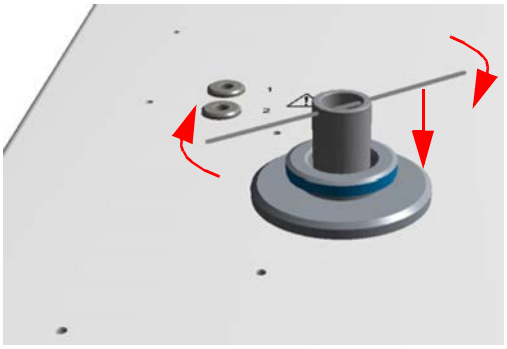


**Figure 9-8.** Reactor in Place

5. Slip on the O-ring. See Figure 9-9.

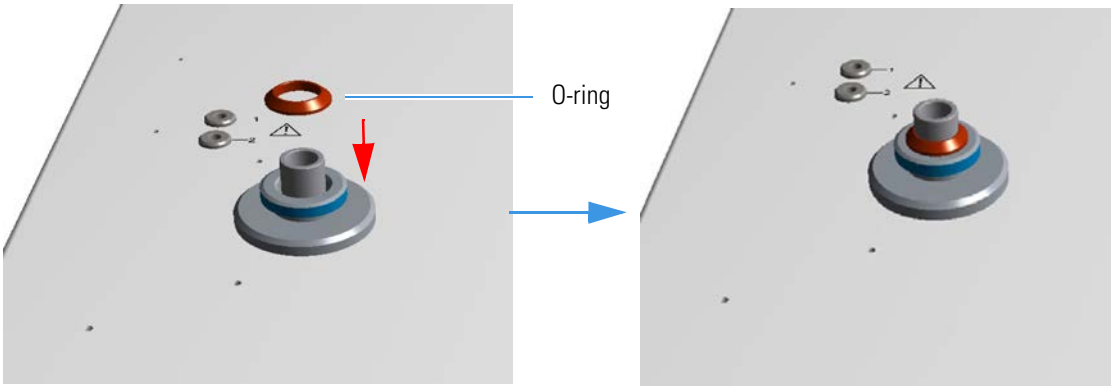



**Figure 9-9.** O-ring

6. By using the tool for alloy steel (HPAR) reactors, introduce the crucible into the combustion reactor, which is in the left furnace. See Figure 9-10.

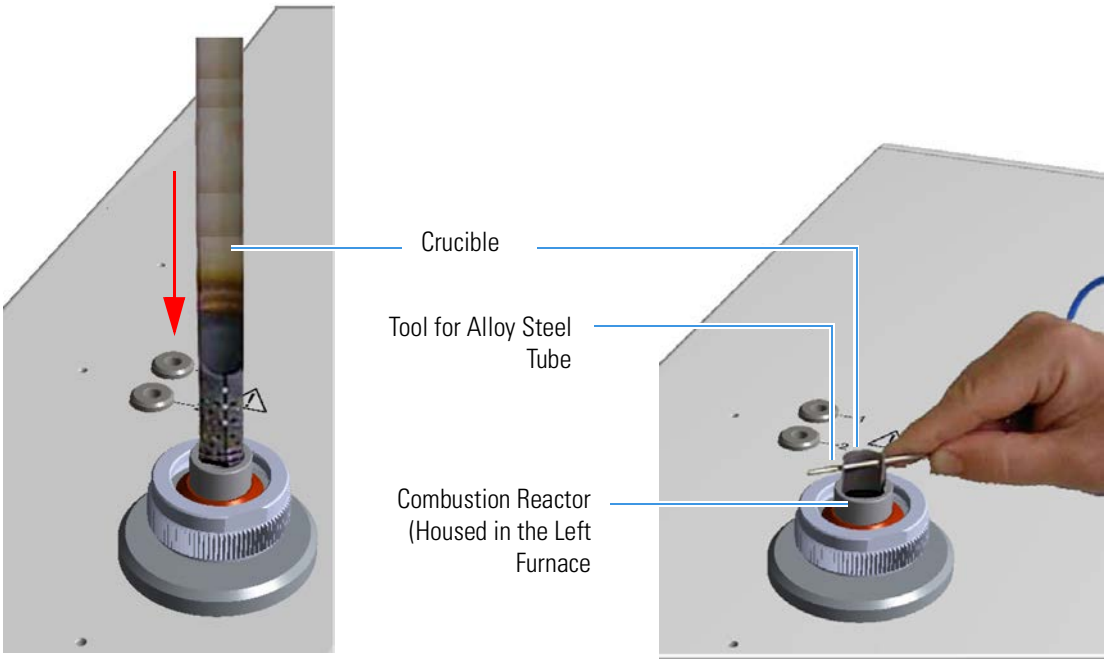



**Figure 9-10.** Introduction of the Crucible into the Combustion Reactor

7. Manually screw the nut of the MAS Plus autosampler. See Figure 9-11.

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

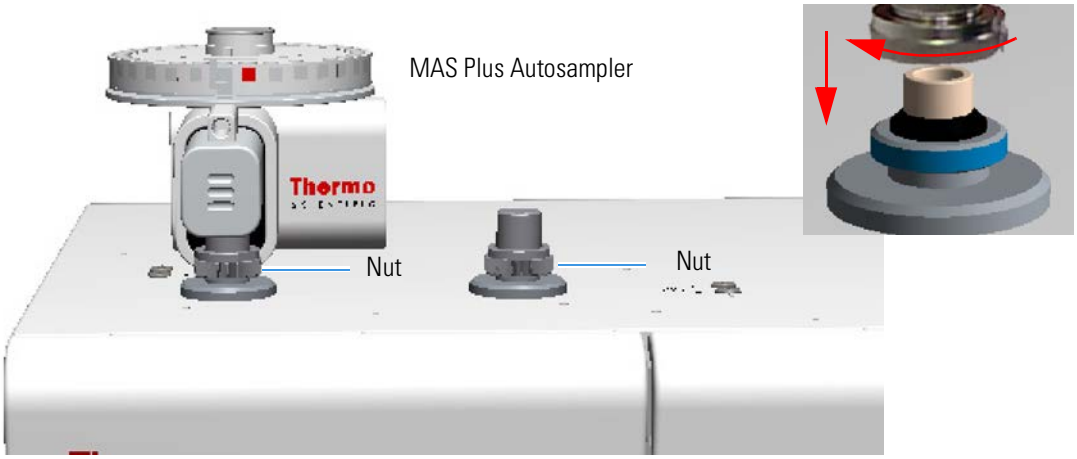



**Figure 9-11.** Mounting the MAS Plus Autosampler on the Left Furnace

8. Install the reduction reactor into the right furnace:
   - In case of an alloy steel (HPAR) reactor, follow the instructions in "To install the alloy steel (HPAR) reactor into the furnace" on page 9-5.
   - In case of quartz tubes, follow the instructions in "To install the quartz reactor into the furnace" on page 9-3.

9. At the end of the operation, manually screw the fixing nut or the autosampler nut if installed. See Figure 9-12.

**NOTICE**

The MAS Plus autosampler installed on the right channel is used for double channel configurations, as for example CHN/CHN, CHN/O, CHNS/CHNS, CHNS/O, etc.

**Figure 9-12.** 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

Table 9-2 summarizes the type of adsorption filter required and the channel to which it should be connected according to instrument configuration. See "Introduction to the Preparation of Reactors and Filters" on page 8-6.

**Table 9-2.** Adsorption Filters

| Determination | Filter |
|---|---|
| CHN | --- |
| CHN/O | Pyrex™ filter |
| CHNS | --- |
| CHNS/O | Pyrex filter |
| S (Sulfur) | Pyrex filter |
| O (Oxygen) | Pyrex filter |
| N (Nitrogen) | Two Plexiglas™ filters in series |
| N Lubricant | Two Plexiglas filters in series |
| NC | Pyrex filter |
| NCS | Pyrex filter |
| NC Soils | Pyrex filter |
| NC Sediments | Pyrex filter |
| NC Filters | Pyrex filter |
| N/Protein | Two Plexiglas filters in series |
| N Brew | Two Plexiglas filters in series |

## Preliminary Operations

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

1. Access the detector compartment by opening the right side door of the instrument. See "Oven Compartment" on page 3-15. Figure 9-13 shows the detector compartment.

**Figure 9-13.** Oven Compartment

## Connecting the Adsorption Filters

❖ **To connect the adsorption filters**

Figure 9-14 shows the result of the installation of two adsorption filters connected in series.

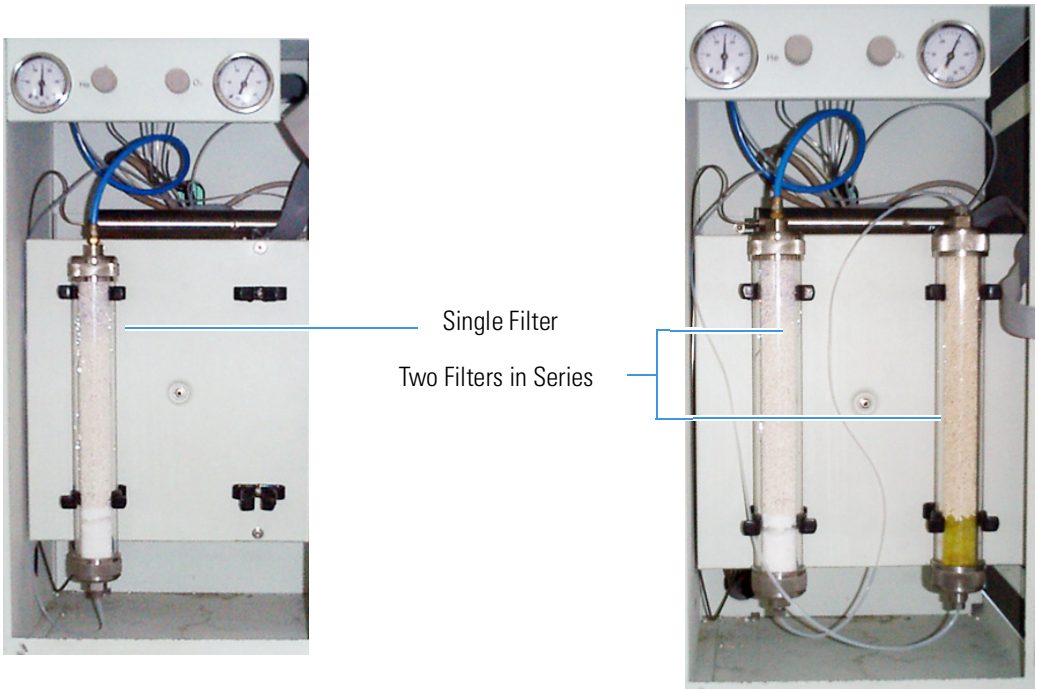



**Figure 9-14.** Adsorption Filters Installed in the Detector Compartment

According to your instrument configuration, do the installation and connection of the adsorption filters following the instructions in "Single Filter" or "Two Filters in Series."

### Single Filter

❖ **To connect a single filter**

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**

1. Connect the filters to one another and then to the circuit as per relevant diagram. See Figure 9-15. See "Introduction to the Preparation of Reactors and Filters" on page 8-6.

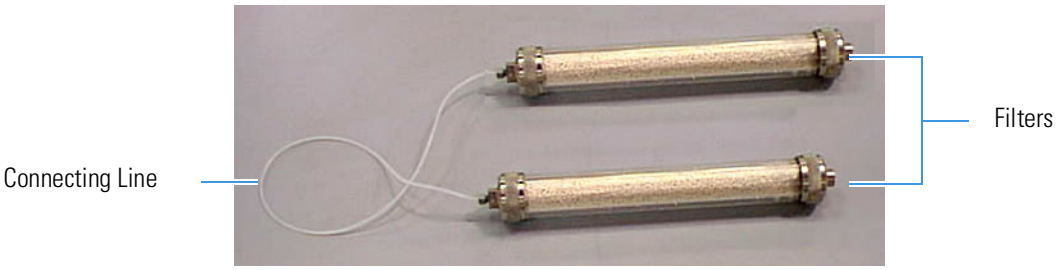



**Figure 9-15.** Connection of Two Filters in Series

2. Introduce the filter into the securing clips, as shown in Figure 9-16.

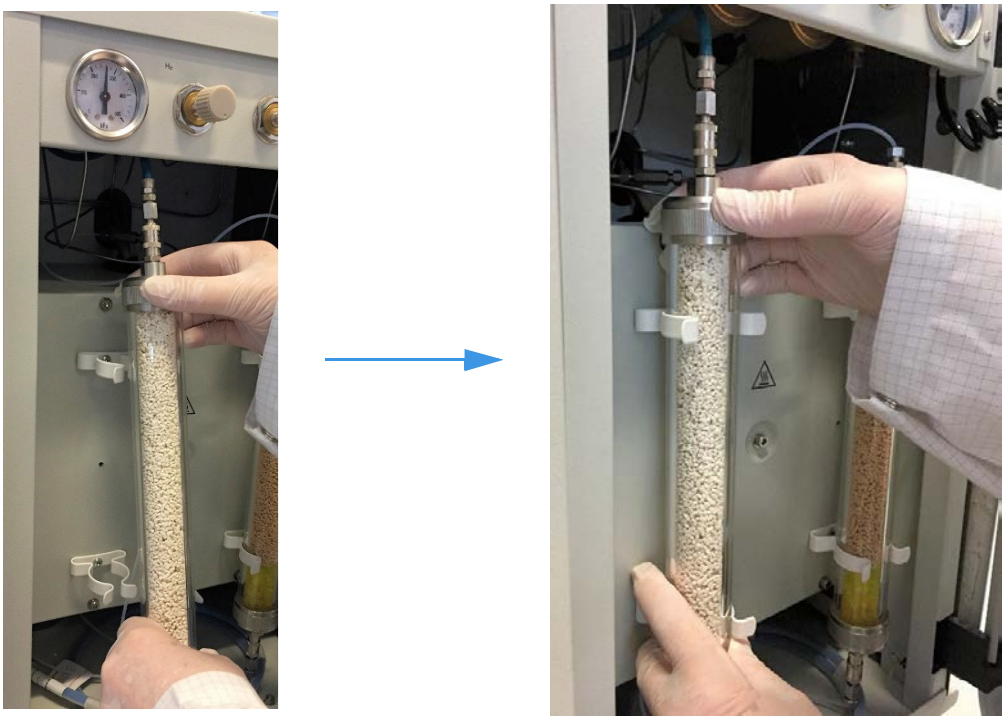


**Figure 9-16.** Installation of the Filters into the Detector Compartment

# Removing the Reactors

Before you start, this operation must be carried out:

1. Make sure that the furnaces are at room temperature.
2. Open the furnaces compartment by lifting the cover and removing the protecting plate. See "Furnace Compartment" on page 3-13.
3. Undo the nuts securing the reactors. If the MAS Plus autosampler is installed, manually unscrew the fixing nut to remove it as shown in Figure 9-1 on page 9-2.

## Removing the Quartz Reactor from the Furnace

The following operating procedure provides the instructions to remove the quartz reactors from the left and right furnaces. The figures in this operating sequence show the installation of a reactor into the left furnace.

**NOTICE** The reactors must be removed with the furnaces at room temperature.

❖ **To remove the quartz reactor from the furnace**

1. Remove the O-ring from the top of the reactor See **A** of Figure 9-17.
2. Using both hands, one on the top and the other an the bottom of the quartz reactor, turn it counter-clockwise and simultaneously pull it upward. See **B** and **C** of Figure 9-17.

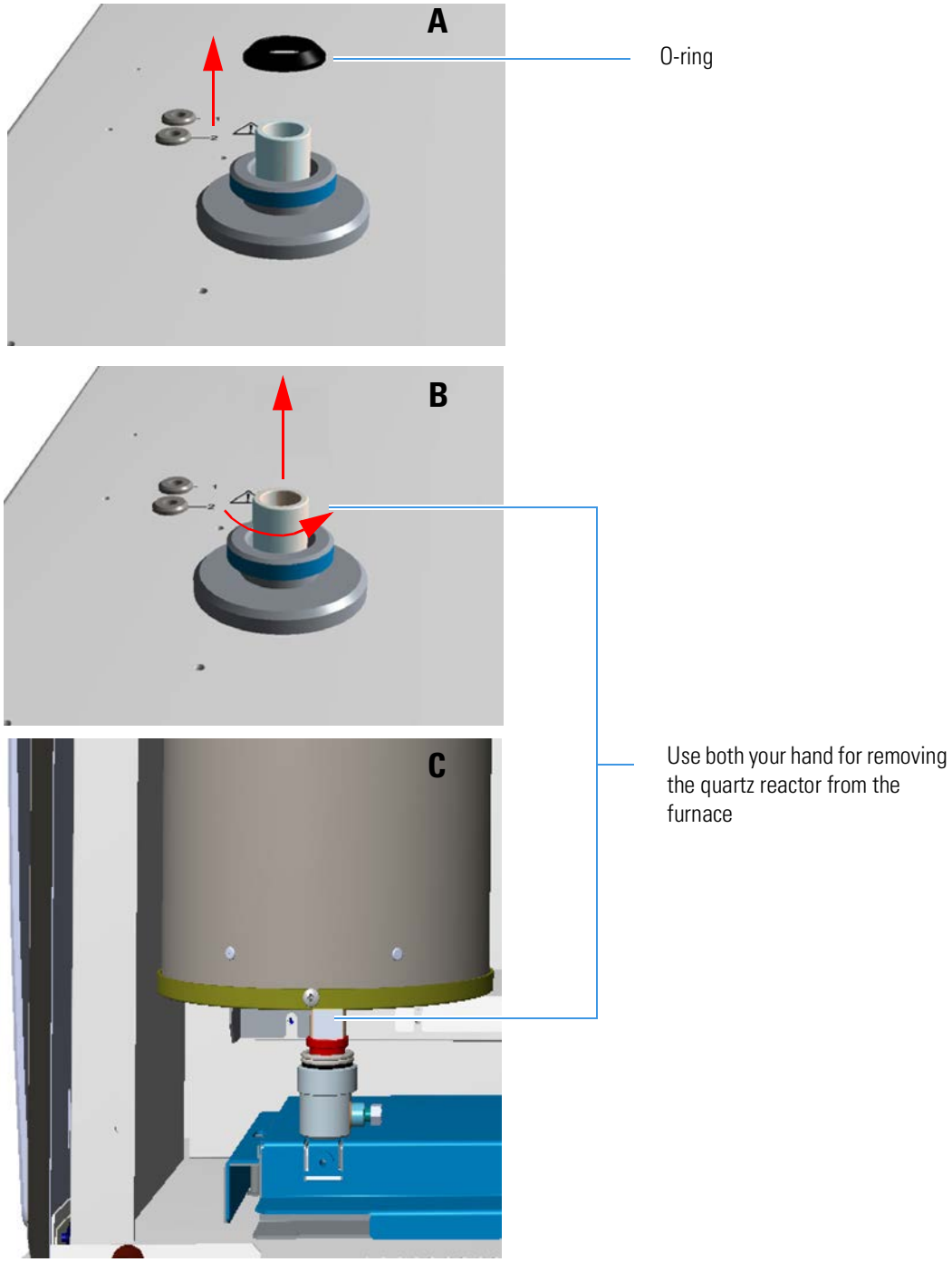



**Figure 9-17.** O-ring and removal of the quartz reactor from the furnace

## Removing the Alloy Steel (HPAR) Reactors from the Furnaces

The following operating procedure provides the instructions for removing the alloy steel (HPAR) reactors from the left and right furnaces.

**NOTICE** The reactors must be removed with the furnaces at room temperature.

❖ **To remove the alloy steel (HPAR) reactors from the furnaces**

| **Material required** |
| --- |
| Tool for alloy steel reactor |
| Tool for crucible |

1. Remove the crucible from the combustion reactor (left furnace) with the appropriate tool. See Figure 9-18.

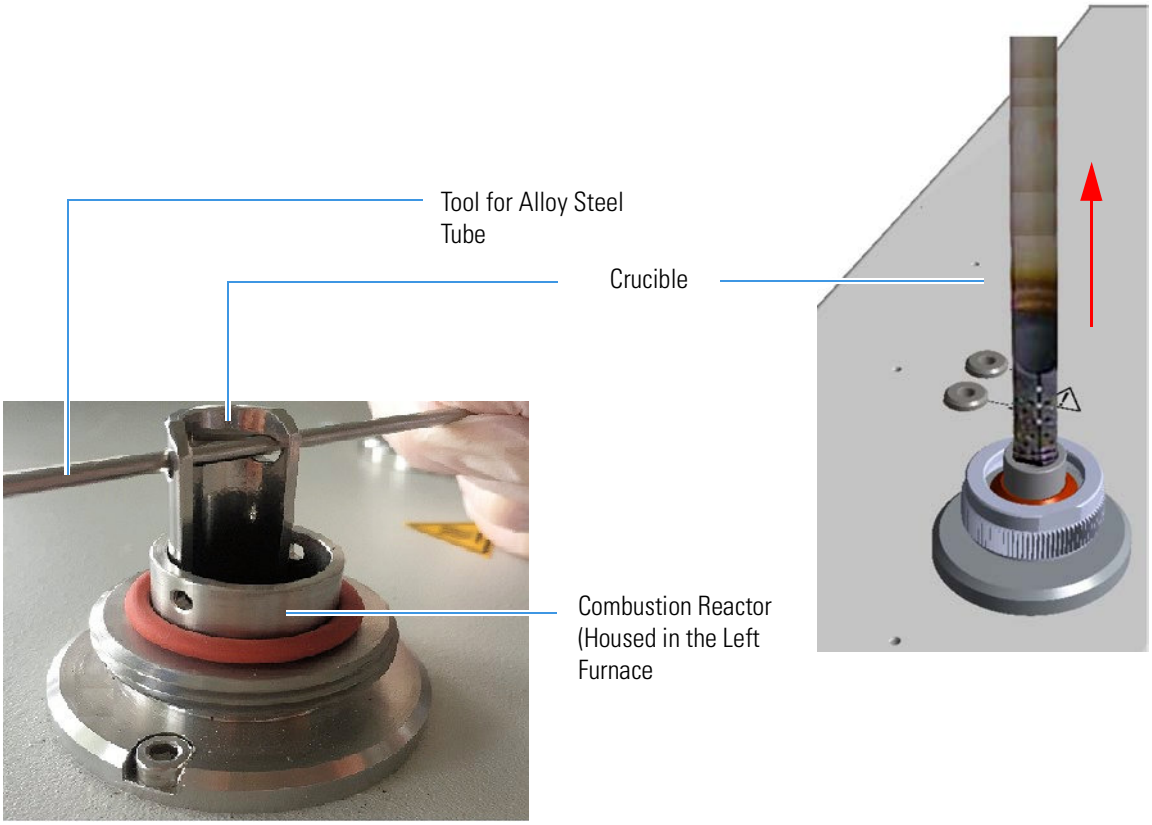



**Figure 9-18.** Removal of the Crucible

2. Remove the O-ring from the top of the alloy steel reactor (HPAR). See Figure 9-19.

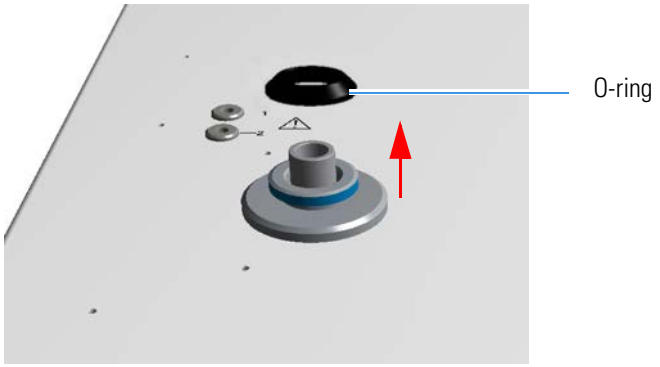



**Figure 9-19.** Removal of the Alloy Steel Reactor O-ring

3. By using the proper tool, remove the alloy steel (HPAR) reactor turning it counter-clockwise and simultaneously pulling it upwards. See Figure 9-20.

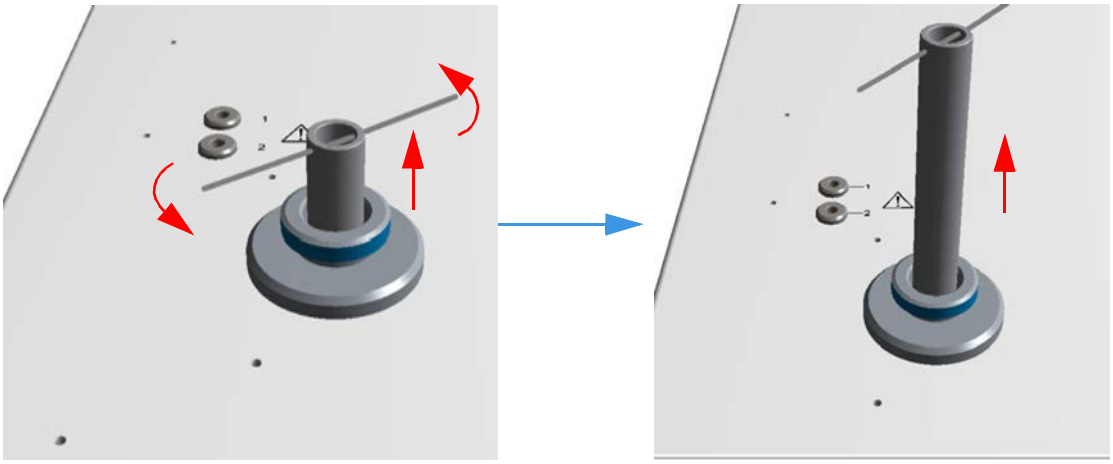


**Figure 9-20.** Removal of the Reactor

4. Remove the tool from the alloy steel (HPAR) reactor.

# Removing the Adsorption Filters

❖ **To remove the adsorption filter from the system**

1. Before you start this operation, open the right side door of the instrument to have access to the detector compartment. See "Oven Compartment" on page 3-15.
2. Remove the filter from the securing clips.
3. 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 instruction to homogenize and weigh the sample.

⚠ Be very careful when you prepare the samples because the substances to be analyzed might be dangerous. Read the Safety Data Sheets referring to the various chemicals. Handle them in the appropriate environment, for example under a fumes hood, strictly obeying the local safety regulations.

**Contents**

# Homogenizing the Sample

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

Table 10-1 gives you indication to weigh the sample depending on your instrument configuration.

**Table 10-1.** Information on Sample Weighing

| | Sample | Information |
|---|---|---|
| **Sulfur** | Soils | In soils, sulfur is often present as the sulfate ion. Therefore, it is necessary to add about 10 mg of vanadium pentoxide ($V_2O_5$) to 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 (for example, bauxite) to definitely higher values (for example, pyrite). If the mineral to be analyzed is unknown, a pre-analysis is recommended. When the sulfur content is defined, the required analyses with proper sample amounts can be performed:<br><br>**Example:** For minerals rich in sulfur, samples of 2–5 mg are prepared.<br>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, make sure that the peak of sulfur dioxide is correctly integrated. |
| **Nitrogen** | Soils<br>Sediments | The nitrogen content in such samples is generally very low (0.1%). 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 µm) is reached. The resulting sample is dried in an oven.
- **Carbons** — The technique for homogenizing 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, 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 do not 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.

- **Vegetables** — Two types of mills are normally used for preparing samples of vegetable products:

  - **Blade mills** for homogenizing cereals, leaves, forage and wood. In these mills, devices with 1 mm mesh sieves are used for N/Protein determination.
  - **Ball mills** for homogenizing samples of fruit and vegetables after lyophilizing. 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 with the optional manual injection device, or automatically with the AI/AS 1310 autosampler for liquids.

# Sample Weighing Techniques

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

For weighing samples, you need a precise micro-balance and some tools for sealing the container containing the sample.

Samples of different nature require specific weighing techniques.

**Tip** 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 depending on the sample nature.

## Solid Samples

Solid samples are introduced directly into the tin container with a spatula. Depending on the sample quantity to be analyzed, see the following operating procedures:

- "To weigh large quantities of solid samples" on page 10-5.
- "To weigh small quantities of solid samples" on page 10-10.

## Liquid Samples

The weighing procedure depends on 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 with a syringe of 10 µL or 100 µL capacity depending on 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 layer previously introduced into the container.

  See the following operating procedures:

  - "To weigh liquid samples" on page 10-11.
  - "To weigh liquid samples deposited on adsorbent material" on page 10-12.

- **Samples Characterized by High Viscosity** — They must be properly mixed before being drawn. To introduce the sample into the container, take some of it with a spatula and let it slide along the container walls. See "Weighing Technique for Viscous Samples" on page 10-13.

  **Tip** Only for N determination, depending to the sample viscosity, it may be necessary to adsorb the sample on a Chromosorb WAW (only for N, N/Brew, and N/Protein configurations) layer previously introduced into the tin container.

- **Samples Available in Liquid Phase** — They can be **manually** injected directly into the reactor, through the manual injection device with a micro syringe, or **automatically** injected with the AI/AS autosampler for liquids.

## Weighing Technique for Large Quantities of Solid Samples

❖ **To weigh large quantities of solid samples**

| Materials required |
|---|
| Balance |
| Tin disks |
| Spring tweezers |
| Sealing device and cylindrical tool |
| Spatula |
| Brush |

1. By using tweezers, take a tin disk and rest it on the cavity of the sealing device. See **A** in Figure 10-2.
2. By using the cylindrical tool, press the tin disk and make it enter the cavity of the sealing device. See **B** in Figure 10-2.
3. Press the top of the sealing device downwards to have the container come out of the cavity.
4. Take out the container with the spring tweezers. See Figure 10-3.
5. Put the prepared container on a clean surface.

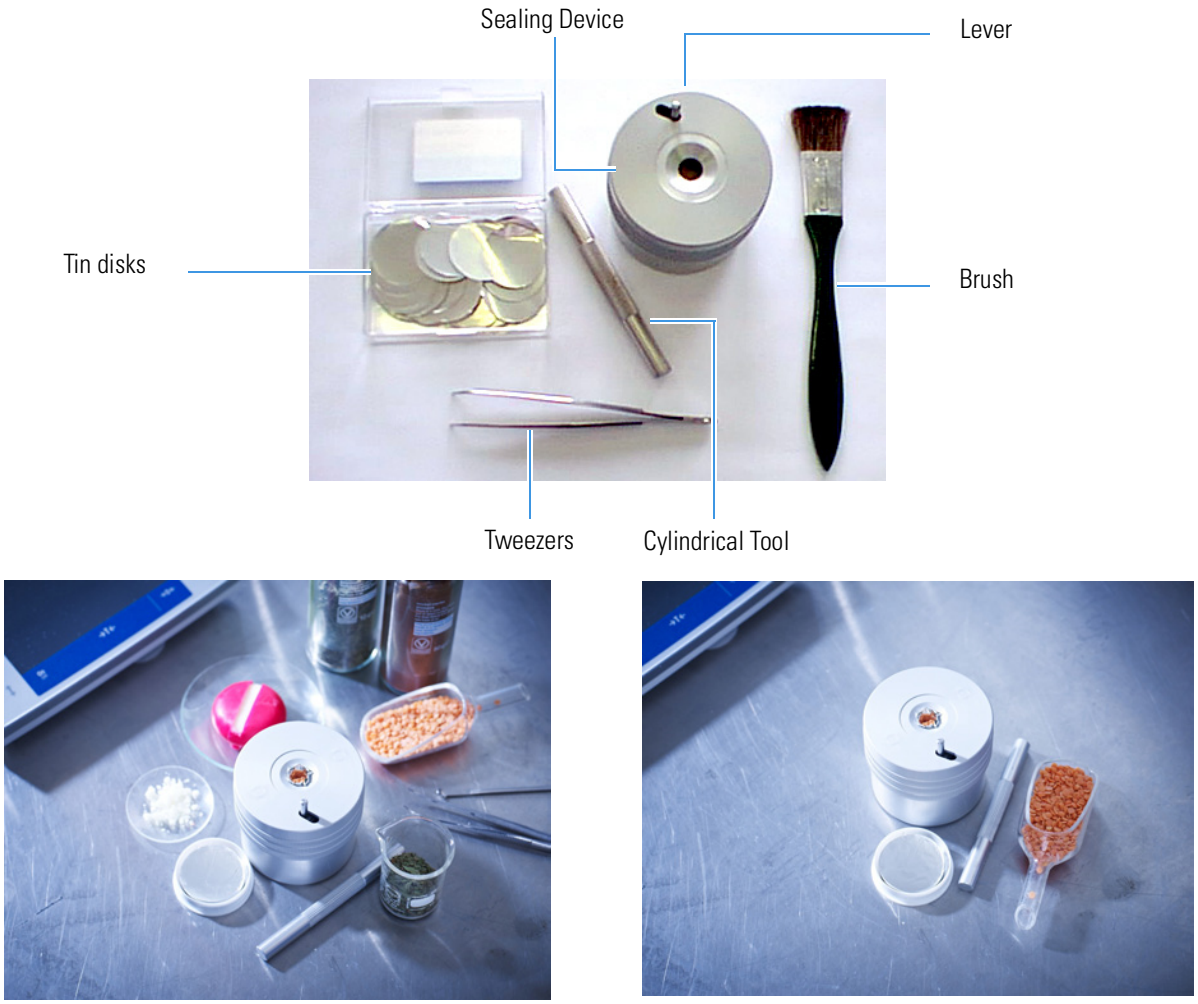



**Figure 10-1.** Accessories Required for Weighing Solid Samples

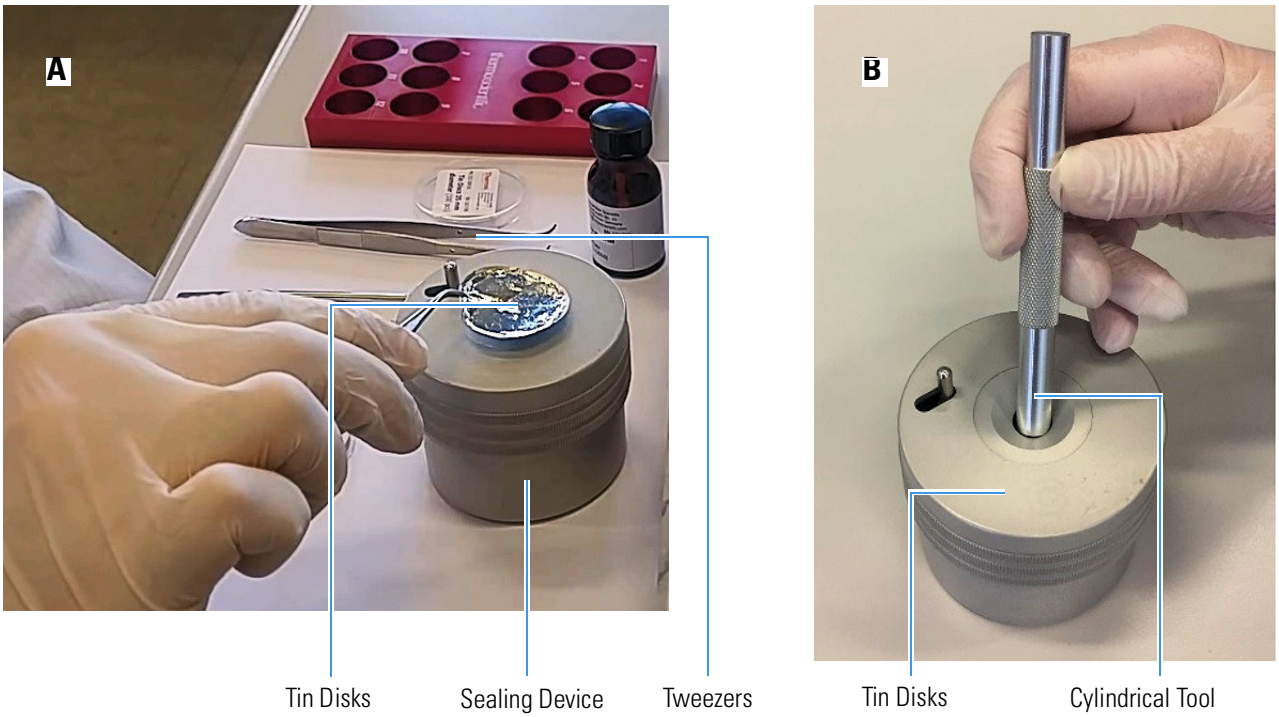



**Figure 10-2.** Preparation of the Tin Container (1)

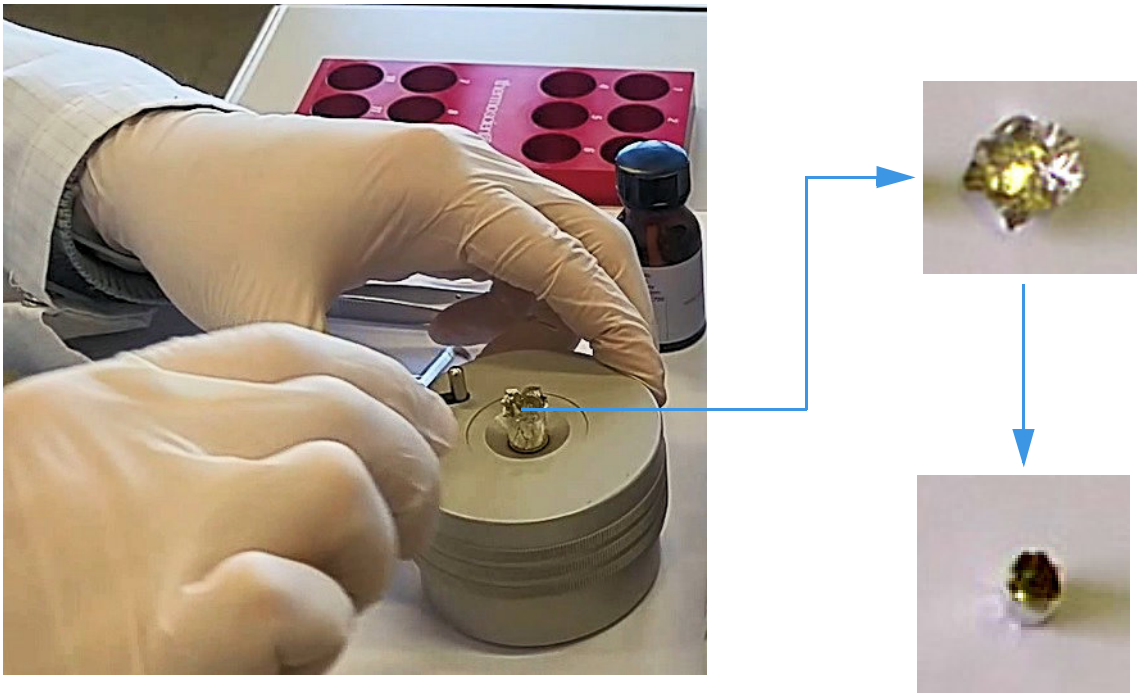


**Figure 10-3.** Preparation of the Tin Container (2)

6. Using a spatula, introduce some sample into the tin container until sufficiently filled. Then press the sample carefully with the cylindrical tool as shown in Figure 10-4.

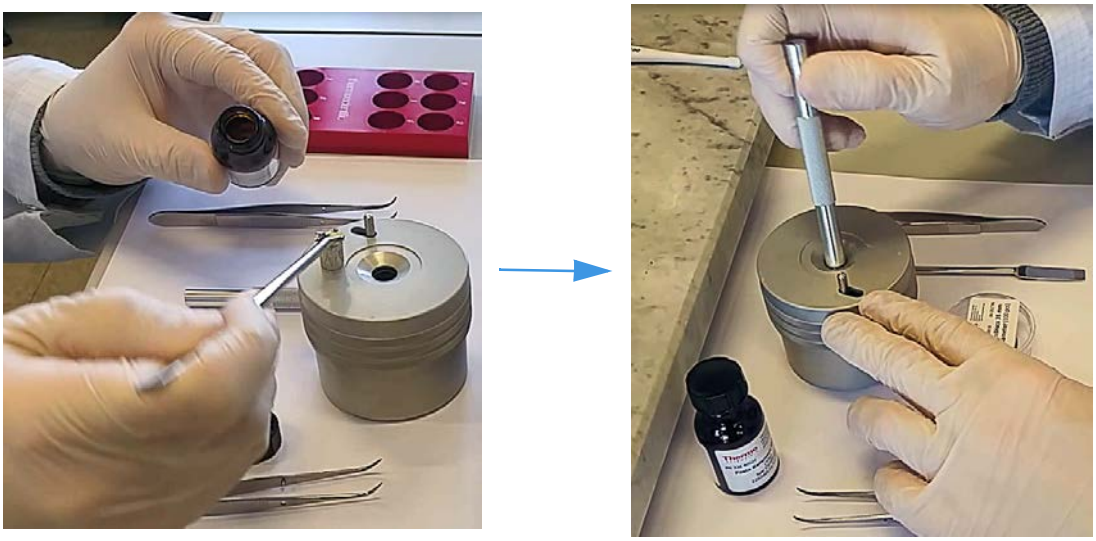


**Figure 10-4.** Introduction and Compression of the Sample

7. Close the container with 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 with the tweezers. See Figure 10-5. Then clean the contact surface with the brush.

**Figure 10-5.** Removal of the Closed Container

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 10-2 on page 10-7.

    b. Using the cylindrical tool, press the tin disk and make it enter the cavity of the sealing device, as shown in Figure 10-2 on page 10-7.

    c. Press the top of the sealing device downwards to have the container come out of the cavity.

    d. Take out the container with the spring tweezers, as shown in Figure 10-3 on page 10-7.

    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 gently press the sample with the cylindrical tool, as shown in Figure 10-4 on page 10-8.

    k. Close the container with 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 with the spring tweezers, as shown in Figure 10-5, and rest it on a clean surface.

    n. Clean the contact surfaces with 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, 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 for preventing sample losses due to defective sealing.

❖ **To weigh small quantities of solid samples**

| Materials required |
|---|
| Electronic microbalance |
| Tin containers for small weighing |
| Two spring tweezers |
| Spatula |

1. Take two containers for small weighing, 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 the sample quantity required for the analysis into the container.
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 with two spring tweezers, as shown in Figure 10-6, to obtain a pellet.

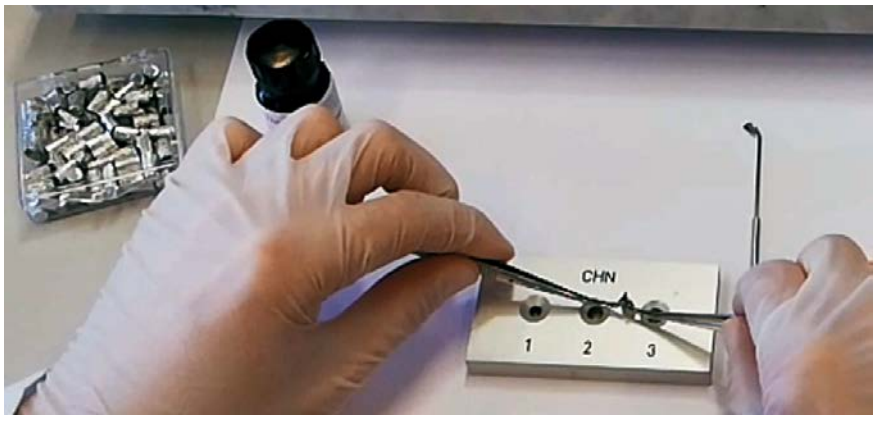



**Figure 10-6.** Preparation of the Container for Small Sample Quantities

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

❖ **To weigh liquid samples**

| Materials required |
|---|
| Electronic micro balance |
| Tin container for liquid samples |
| Spring tweezers |
| Sealing device (optional) |
| Spatula |
| 10 or 100 µl syringe depending on the instrument configuration |

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 with a syringe. See Figure 10-8.

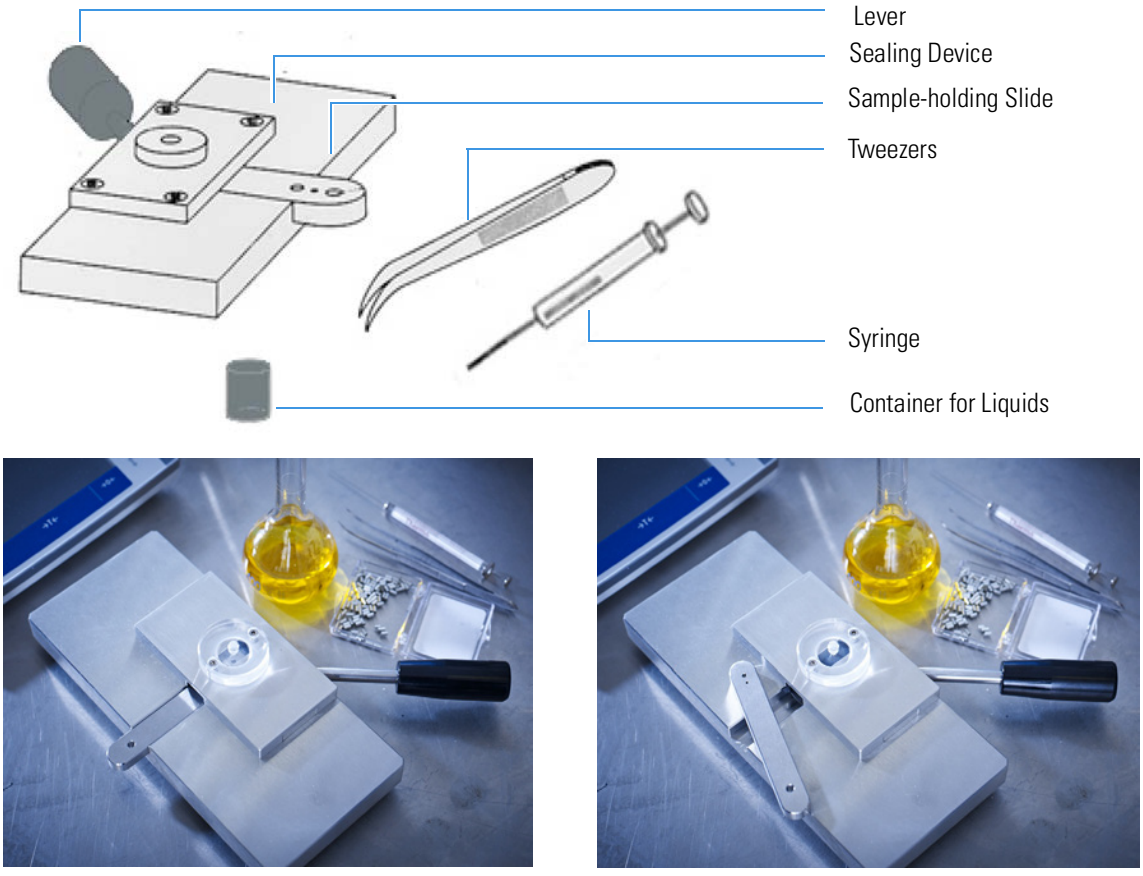



**Figure 10-7.** Accessories for Weighing Liquid Samples

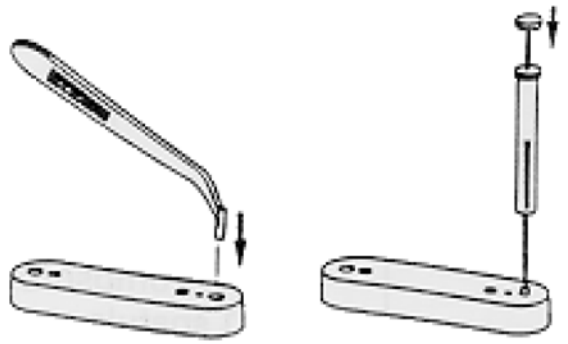


**Figure 10-8.** Housing for the Container and Sample Injection

3. Put the slide with the container into the sealing device and tighten the container with the appropriate lever. See Figure 10-9.
4. Remove the slide from the sealing device and then the container with the sample from the slide. See Figure 10-9.

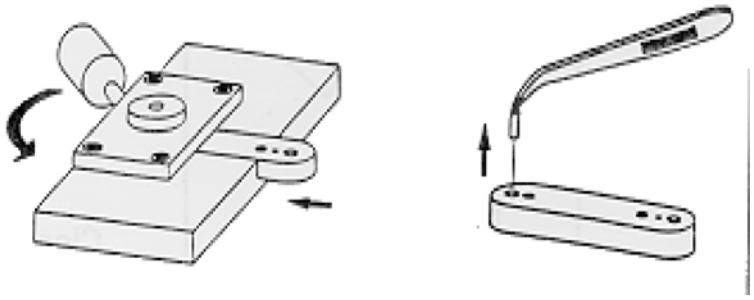


**Figure 10-9.** Closing and Removing the Container

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

❖ **To weigh liquid samples deposited on adsorbent material**

| Materials required |
|---|
| Balance |
| Tin containers for liquid samples |
| Two spring tweezers |
| Spatula |
| 100 µL syringe |
| Chromosorb™ WAW (Only for N, N/Protein and N-Brew Determinations) |

**Tip** Chromosorb WAW 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 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. See Figure 10-10.

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

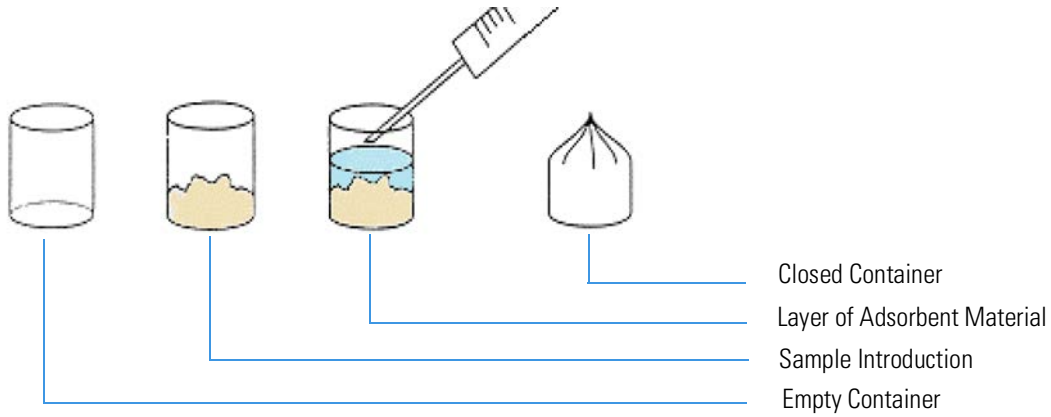



**Figure 10-10.** Weighing of a Liquid Sample Deposited on Adsorbent Material

5. Close the container with 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

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

- "To weigh liquid samples" on page 10-11.
- "To weigh liquid samples deposited on adsorbent material" on page 10-12.

**Tip** When a liquid sample is too viscous to be drawn by means of a syringe, use the spatula provided in the instrument standard outfit. See Figure 10-11.

**Figure 10-11.** Introduction of a Viscous Sample into the Container

# Use of Additives for Elemental Analysis

This section gives you some information on the most common additives and their application in elemental analysis. Some additives help the complete combustion of the sample, other protect the analyzer from harmful components present in the sample and few sample wrapping materials can also provide benefits for the proper analysis.

Table 10-2 gives you indication to use the additive depending on your instrument configuration and sample matrix, including the amount to use and specific comments.

**Table 10-2.** Information of additives for elemental analysis

| Additive | Config. | Samples | Ratio additive/sample | Comments |
|---|---|---|---|---|
| Aluminum foil (Al) | NC, CHN | Analysis of explosives | Sample wrapping | No exothermic reaction. However, aluminum in high content can produce obstruction in the crucible. |
| Calcium oxide (CaO) | CHN, CHNS<br>NCS | Fluorides, fluorocarbons | 1:1 | In some cases, with the addition of small quantities of magnesium oxide (MgO) or manganese oxide ($MnO_2$).<br>Resulting calcium fluoride is very stable, and works well for CHNS determination. |
| CF (Teflon™ powder) | O | Minerals | 1:1 | Silicon tetrafluoride ($SiF_4$) is formed very stable and releases O from silicon dioxide ($SiO_2$) to form CO. |
| Copper oxide powder (CuO) | N, NCS, CHNS | Organic waste | 1:1 | The organic matter reduces copper oxide (CuO) to Cu and uses oxygen to oxidize and release $CO_2$ more quantitatively. |
| FluoAdso | CHN | Fluorine containing samples | Filled in the reactor | Traps the fluorine immediately after the combustion, can also trap the other halogens and sulfur. |
| Fluorene/<br>Polystyrene/<br>Benzopyrene | O | | Few mg | A few capsules of these materials are dropped into the reactor for conditioning at the beginning of a sequence. The amount of carbon in close contact with the sample promotes the formation of CO. |
| Gold powder (Au) plus manganese oxide ($MnO_2$) | NC, CHN, CHNS | Mercury containing samples | 1:1 | Mercury (Hg) precipitates in the reactor and retains the sulfur as mercury sulfide (HgS). By adding gold, the released mercury amalgamates with the gold and both are retained in the copper zone of the reactor. |
| Graphite | O | Minerals | 1:1 | The amount of carbon in close contact with the sample promotes the formation of CO. |
| Iron chips (Fe) | CHN | For sulfur containing samples | 1:1 | Sulfur will bind to the iron. |

**Table 10-2.** Information of additives for elemental analysis, continued

| Additive | Config. | Samples | Ratio additive/sample | Comments |
|---|---|---|---|---|
| Lead oxide ($Pb_3O_4$), selenium dioxide ($SeO_2$), cerium oxide ($CeO_2$) | NC | Carbides, refractory materials | 1:1 | Sometimes it is suggested to mix the additive with tin powder. |
| Magnesium oxide (MgO) | CHN | Fluorine containing samples | 1:1 | Trap the fluorine. |
| Nickel carbon (NiC) powder | O (for IRMS) | Minerals | 1:1 | With nickel carbon (NiC), the amount of carbon in close contact with the sample promotes the formation of CO. Can be also used as 1 cm layer in the pyrolysis reactor. |
| Saccharose, sucrose, fructose | N, NS | Fertilizers, urea, amino acids, nitrates samples | 1:1 | It is suggested to optimize the oxygen injection time and the sample weight according to the *OEA CookBook* information. However, sometimes the nitrogen value comes out low and the addition of sugar helps to obtain the total nitrogen content. |
| Silver powder (Ag) plus manganese oxide ($MnO_2$) | NC, NCH | Fluorocarbons | 1:1 | Silver fluoride (AgF) is formed and is very stable. |
| Silver tungstate ($Ag_2WO_4$), Vanadium pentoxide ($V_2O_5$) | NC, NCS, CHNS | Mineral sulfides (pyrites), fluorides | 1:1 | Silver reacts with fluorine, $V_2O_5$ increases the availability of oxygen in close contact with the sample |
| Silver wool | OH | Sulfur and halogens containing samples | Filled in the reactor | Silver wool reacts with sulfur containing compounds and traps the sulfur. |
| Silver wool or foil (Ag) | NC, CHN | High salt samples | 1:1 or sample wrapping | Salts damage combustion reactors, silver wool or foil reduces the risk of damage. |
| Tin foil (Sn) | | Increase combustion temperature | Sample wrapping | Exothermic combustion causes local temperature to reach 1800 °C. |
| Tin powder (Sn) | NC, NCS | Charcoal, mineral carbons, PAH, adamantanes | 1:1 | Tin oxidizes resulting in temperature increase due to the exothermic reaction. Sometimes it is suggested to mix with vanadium pentoxide ($V_2O_5$). $V_2O_5$ is reduced by releasing oxygen which improves combustion efficiency. |
| Tungsten oxide ($WO_3$) | CHNS, NCS, S | For complete conversion of sulfur.<br>For adsorption of alkaline ions. | 1:1 or 10:1 | Like vanadium pentoxide ($V_2O_5$) but less hazardous.<br>In general not necessary for pure organic compounds |

**Table 10-2.** Information of additives for elemental analysis, continued

| Additive | Config. | Samples | Ratio additive/sample | Comments |
|---|---|---|---|---|
| Tungsten oxide ($WO_3$) plus magnesium oxide (MgO) | NC, CHN, CHNS | Phosphines | 1:1 | Magnesium oxide (MgO) acts as an alkaline-oxidant fusion forming phosphorus tungstates. Phosphorous-bound carbon is released and a better carbon data is obtained. |
| Tungsten oxide ($WO_3$) plus selenium oxide (SeO) | NC, NCH, NCHS | Organometallics | 1:1 | In some cases, is it used a mix with vanadium pentoxide ($V_2O_5$). Some metals of the complexes to be analyzed form sulfides or nitrides, which seem to be avoided with these additives. |
| Vanadium pentoxide ($V_2O_5$) | NC, NCH, NCHS, NCS | For complete conversion of sulfur in vegetables, flour, meat, feed, sediments, soil, inorganic samples, etc<br>Carbides, Refractory materials | 1:1 | $V_2O_5$ is reduced by releasing oxygen, improving combustion efficiency and higher $O_2$ accessibility. |

**Notes:**

- Check the additives for contaminants and store them in a desiccator over NaOH tablets or any other water adsorbing material.
- If the elements content is low or in trace amounts, the blank with additives must be evaluated and a similar weight must be used for the blank and for the sample analysis.
- It is strongly recommended to eliminate carbonation of metal oxides (CaO, MgO, CuO) by putting them in an oven for half an hour at 800 °C.

11

# Instrument Start-up

This chapter provides information and instructions for preparing the instrument for running analyses.

**Contents**

# Introduction

To analyze any type of sample, the instrument must be in the correct operating condition. Proceed according to the following operating sequences:

1. Powering on
2. Installation of the Eager*Smart* Data Handling Software into the PC
3. Analytical configuration
4. Leak checking
5. Adjustment of the detector signal level

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

# Powering On the System

❖ **To power on the instrument**


1. Power on the instrument by lifting the circuit breaker located at the back of the instrument (position **I**). At the powering on, the indicating LED **Power On** on the status panel lights up.
2. Power on the computer and any complementary units with their main switches.

# Installing the Eager*Smart* Data Handling Software

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

| **Material required** |
| --- |
| *EagerSmart* Data Handling Software package |

The Eager*Smart* Data Handling Software fully controls all operations of the Flash*Smart*, Flash 2000, and EA 1110 Elemental Analyzers. It is compatible with commercially available computers and requires the use of Microsoft Windows™ 7 / 8 / 10 operating systems.

The free space on the PC hard disk must be at least 1 GB. The Eager*Smart* Data Handling Software is installed by using the yellow pen driver (USB stick) provided in the standard outfit and operating as follows:

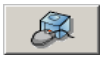
**NOTICE** Before you install the Eager*Smart* Data Handling Software, make sure that any previous version of Eager software is removed from the disk.

❖ **To install the Eager*Smart* software**

1. Remove a previous version of the Eager software installed on your computer.
   a. Select **Control Panel | Add/Remove Programs**.
   b. In the dialog window shown, select the previous **Eager** version to remove.
   c. Click **Add/Remove**.
2. Install the new version of the Eager*Smart* Data Handling Software. When the blue USB stick is introduced into a free USB port of the computer, the installation menu shown in Figure 11-2 appears.

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


**Figure 11-1.** Eager*Smart* Installation Menu

3. Start the installation by clicking the push-button  **Install EagerSmart for Flash/FlashSmart**.

4. Follow the instructions prompted step by step.

5. At the end of installation, in the page **Start-Program EagerSmart**, double-click the **EagerSmart for Flash** icon. The window of Figure 11-2 is shown.

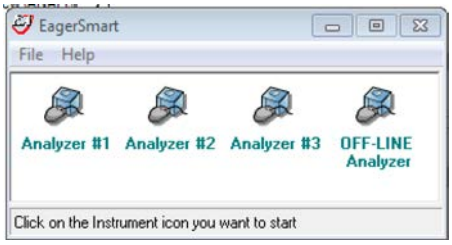



**Figure 11-2.** Selection of the Instrument

6. Click the icon of the instrument selected.The program is designed to work with four instruments. Each icon corresponds to one instrument. The instrument name shown below the icon can be changed. To do this, click the existing name and overwrite the new one.

7. Eager*Smart* proceeds with the registration and the activation of some drivers needed for the correct functioning of the software.

   a. Click **Ok** to the answers prompted step by step.

   b. At the end of the operation, reboot the computer. Start Eager*Smart* again selecting **Start | Programs | EagerSmart**.

   **Tip** Run the Eager*Smart* software with Administrator privileges.

8. Follow the prompted indications. At the end of the installation, the Eager*Smart* Data Handling Software Main Menu appears.

# Eager*Smart* Data Handling Software Main Menu

The Main Menu of the Eager*Smart* Data Handling Software, shown in Figure 11-3, is the starting point to enter all menus and relevant functions. The menus and the icons of the Main Menu are described in Table 11-1 and Table 11-2 respectively.

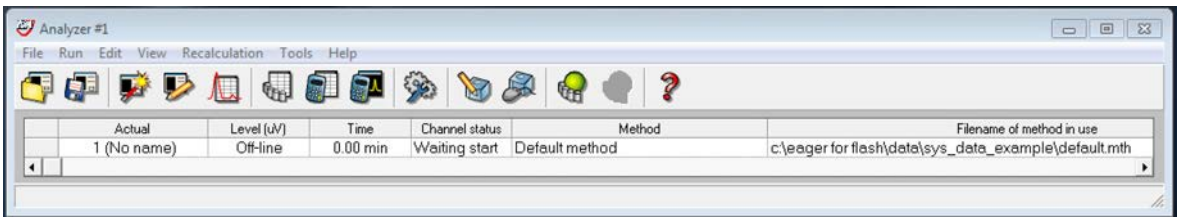



**Figure 11-3.** Eager*Smart* Main Menu

**Table 11-1.** Main Menu: Description of Menus

| Menu | Description | Sub-menus and Options |
|---|---|---|
| File menu | Provides functions concerning the instrument operation.<br><br>Used during the analyzer installation procedure. | • Set language<br>• Color setup<br>• Instrument name and configuration<br>• System administration<br>• Installation qualification<br>• Load method<br>• Load system defined method<br>• Save method<br>• Copy method from...<br>• Printer setup<br>• Print method<br>• Exit Flash*Smart* |
| Run Menu | Chooses the type of start command to be sent to the analyzer, to stop the analytical cycle, or abort the current analysis. | • Start sequence of samples<br>• Stop sequence in progress<br>• Start single sample data acquisition<br>• Stop data acquisition<br>• Abort data acquisition<br>• Run macro |
| Edit Menu | Provides functions related to the instrument setup and analytical parameters. | • Edith Method<br>• Component table<br>• Sample table<br>• Wizard method development<br>• Edit Elemental Analyzer parameters<br>• Edit Sampler Parameters[1]<br>• Evaluate Injection Delay[2]<br>[1, 2] These sub-menus are shown only if the autosampler for liquids has been configured in Instrument Name and Configuration. |

**Table 11-1.** Main Menu: Description of Menus, continued

| Menu | Description | Sub-menus and Options |
|---|---|---|
| View Menu | Monitors the analysis in real time. Reads the result of the last sample run; checks the calibration curve; compares and overlays chromatograms; checks the instrument status and maintenance. | • View sample being acquired<br>• Last sample calculated results<br>• View Calibration curve<br>• View Chromatograms<br>• Overlay Chromatograms<br>• Operate on Chromatograms<br>• Compare Chromatograms<br>• View Elemental Analyzer Status<br>• View Maintenance |
| Recalculation Menu | Cancels the calibration curve and the results of previous analyses. You can recalculate previous results individually or sequentially. Provides also the summary of results. | • Reset calibration factor<br>• Recalculation<br>• Summarize results |
| Tool Menu | Used when ashes removal, reactor replacement, or both, are required as maintenance. The command **MVC management** is enabled when the **MultiValve Control (MVC)** module is connected to your Flash*Smart* or Flash 2000 EA. | • Ashes removal<br>• Reactor replacement<br>• Cleaning the MAS Piston<br>• MVC management (enabled when selected. See "Configuring the Analyzer" on page 11-8). |
| Help Menu | Enters the Flash*Smart* help program. Subdivided into different modules, each one designed to cover specific issues of the module currently in use. | • Help<br>• About Eager*Smart* |

**Table 11-2.** Main Menu: Description of the Menus

| Icon | Function | Description |
|---|---|---|
|  | Load method | Loads a previously saved analytical method. |
|  | Save method | Stores new operating methods. |
|  | Wizard method development | Develops new operating methods. |
|  | Edit Method | Accesses to the integration and calculation parameters, and to the parameters for printing analytical reports. |
|  | Components Table | Contains the stored retention times, which allow to identify N, C, H, S, and O. |
|  | Sample Table | Contains all functions related to sample records, and the function allowing communication with the balance. |

**Table 11-2.** Main Menu: Description of the Menus, continued

| Icon | Function | Description |
|---|---|---|
|  | Summarize Results | Contains analytical results, print options and chromatograms. |
|  | Recalculation | Recalculates previous results. |
|  | View Maintenance | Programs current maintenance by recording the number of analyses run by each reactor of the analytical circuit. |
|  | Edit Elemental Analyzer Parameters | Opens the pages containing the commands for the setting of temperatures, flows, times, detector, and the analyzer control functions. |
|  | View Elemental Analyzer Status | Comprises four pages displaying the analyzer conditions. Provides special functions to check the system pneumatic tightness (Leak Test), to check the baseline level, and to program automatically the "Autoready" function. |
|  | Edit Sampler Parameters | Shown in the Main Menu when an autosampler for liquids has been configured. See "Configuring the Analyzer" on page 11-8. |
|  | Start Sequence | Starts a series of analyses having different current and timed requirements. At the end of the analytical cycle, the instrument can either be put in Stand-by Mode, or the furnace and detectors be switched off, or the gas flows turned off. |
|  | Stop Sequence | Stops in any moment the sequence of analyses only completing the current run. |
|  | Access the Help System | Explains in detail the Eager*Smart* functions. |

# Configuring the Analyzer

The analytical conditions are set in our laboratories during the final test of the analyzer. To put the analyzer in operating conditions, follow the instructions in "To configure the analyzer."

❖ **To configure the analyzer**

1. In the Main Menu of Figure 11-3 on page 11-5, choose **File | Instrument Name and Configuration.** The window shown in Figure 11-4 appears.

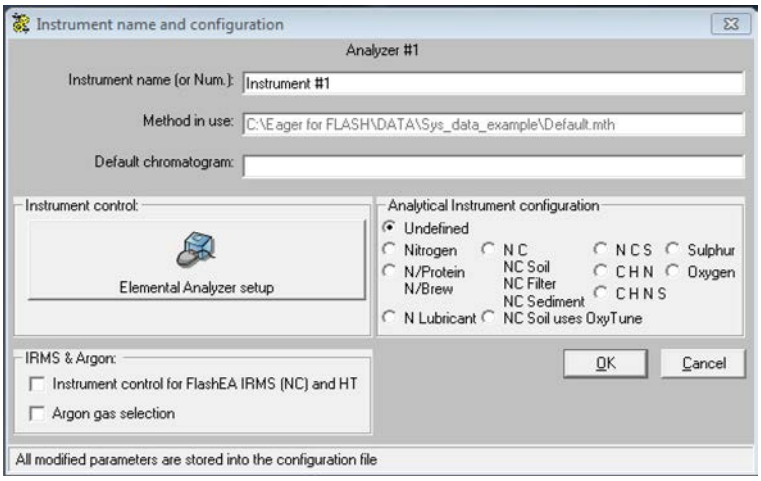



**Figure 11-4.** Instrument Name and Configuration

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

b. In the field **Analytical instrument configuration**, select the configuration of your instrument.

**NOTICE**

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

**Tip** The option **Undefined** can be used if the intended operating conditions are different from those defined for the instrument configuration.

c. If the instrument is equipped with an EFC-t module for argon carrier gas, in the field **IRMS & Argon** select **Argon gas selection**. The gas control page is shown for setting the **Argon Gas Option** parameters.

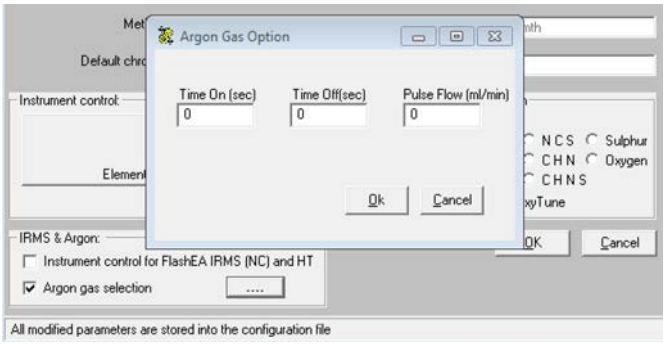



**Figure 11-5.** Argon Gas Option

**Argon Gas Option** parameters allow to inject a supplementary flow of argon in the first part of the analysis to obtain a smoothly baseline allowing an excellent integration of the nitrogen peak.

- In the field **Time On**, set the time at which the injection must begin.
- In the field **Time Off**, set the time at which the injection must end.
- In the field **Purge Flow**, set the flow.

d. Click **Elemental analyzer setup** to display the dialog window shown in Figure 11-6, where the configuration parameters must be set.

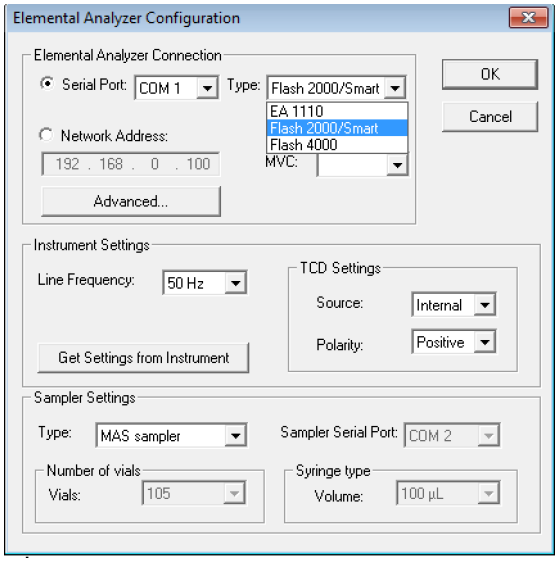



**Figure 11-6.** Configuration Dialog Window

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

f. In the field **Type**, make sure that the instrument in use is **FlashSmart**.

g. In the field **Instrument Settings**, choose the following settings:

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

**Tip** For the oxygen determination in CHN/O and CHNS/O configurations, select the negative polarity. If for the same configurations two MAS Plus autosamplers for solid samples are used, see the relevant Analytical Method described in Chapter 4, "Analytical Principles."

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

- In the case of an autosampler for liquid samples, also specify the serial port of the computer to which the autosampler is connected, and the number of vials.
- Click **Ok** to go back to the window of Figure 11-6, then click **Ok** to return to the Main Menu.

2. In the Main Menu, select **File | Load System Defined Method.** The file name of the loaded method is shown in the grid **Filename of method in use** of Main Menu.

3. In the Main Menu, select **Edit | Edit Elemental Analyzer Parameters** or click the  icon. The following window appears where the analyzer operating parameters are shown. See Figure 11-7.

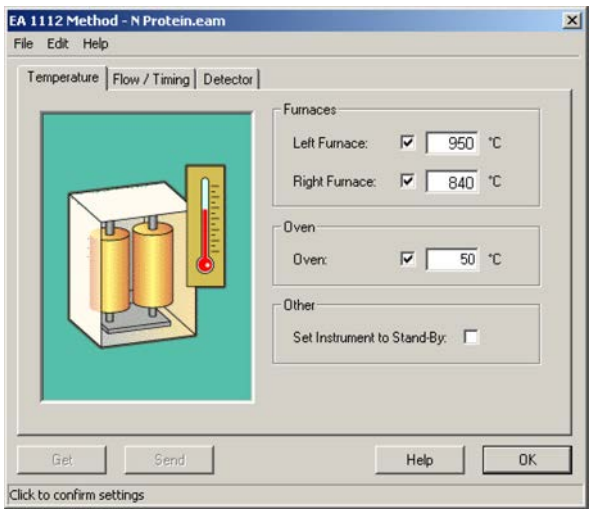



**Figure 11-7.** Example of Analyzer Parameters

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

b. The furnaces begin to heat, and the helium (argon) flows in the circuit.

After about 50 minutes, the furnaces reach the temperature settings, and the LED **Ready** on the status panel lights up. The instrument is now ready to run analyses. However, before you start an analytical cycle, perform a **leak test** to check if the **Carrier** and **Reference** pneumatic circuits are free of leaks.

The leak test must be performed also any time a component of the pneumatic circuit is replaced to make sure that reactors, filters, if any, and gas chromatographic columns have been properly installed. See "Performing a Leak Test" on page 11-12 for details.

# Performing a Leak Test

❖ **To check for leaks**

1. In the Main Menu, select **View | View Elemental Analyzer Status**, or click the  icon. The following dialog window is shown. See Figure 11-8.

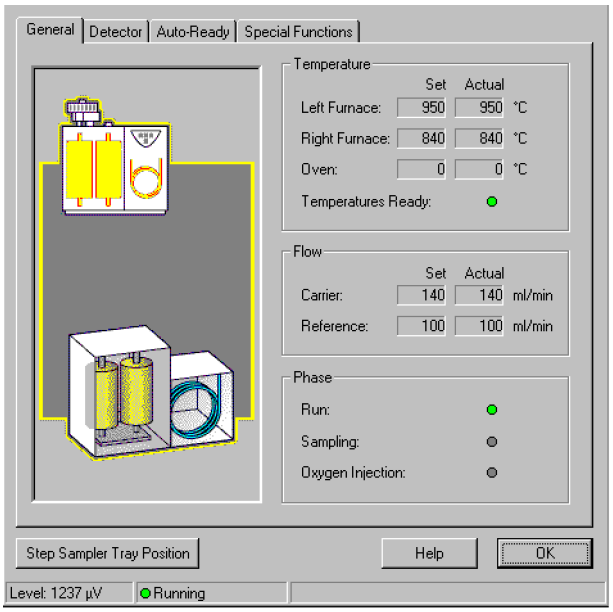



**Figure 11-8.** Analyzer Status Page

2. Select the **Special Functions** tabbed page. See Figure 11-9.

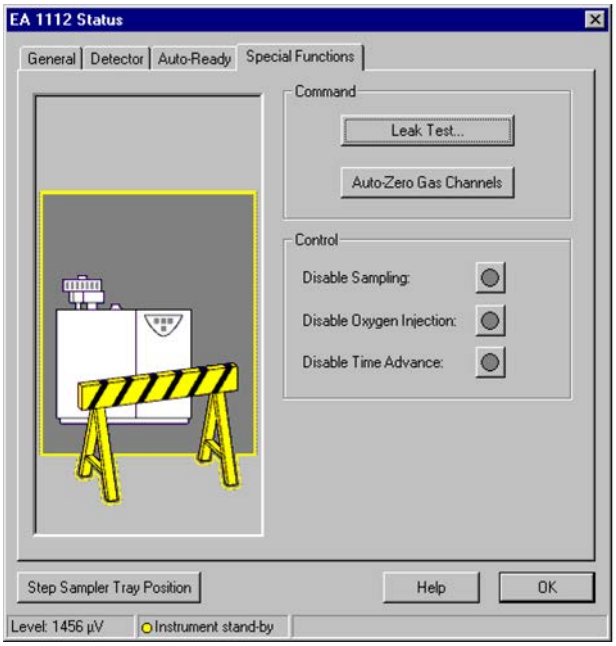



**Figure 11-9.** Special Function Tab

3. In the field **Command**, click **Leak Test**. The window shown in Figure 11-10 indicates the status of the leak test.

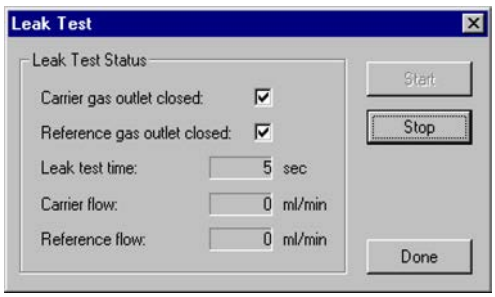



**Figure 11-10.** Leak Test Status Window

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

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

      **Tip** Due to the different thermoconductibility of argon, when the leak test is performed, the values obtained for Carrier Flow and Reference Flow can be within 0 and 15 mL/min.

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

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

# Adjusting the Detector Signal Level

❖ **To adjust the level of the TCD detector signal**

1. In the Main Menu, select **View | View Elemental Analyzer Status**, or click the  icon, then select the **Detector** tabbed page. See Figure 11-11.

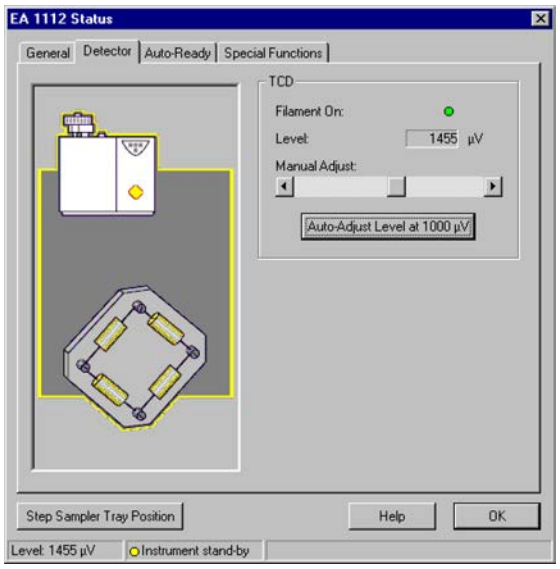



**Figure 11-11.** Detector Status Tab

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

**Tip** Due to the different thermoconductibility of argon, the actual TCD signal may be within 1000±50 µV.

12

# Applications

This chapter provides guidelines referring to the applications of the Flash*Smart* Elemental Analyzer.

**Contents**

# 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.

Thanks to the Eager*Smart* Data Handling Software, the Flash*Smart* Elemental Analyzer 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, follow the instructions in "To change the oxygen quantity" on page 12-3.
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 quantity of oxygen required for the combustion, do what is described in "To establish the required oxygen quantity" on page 12-4.

For **N/Protein** and **N Brew** analyzers, we recommend to always use the **OxyTune™** function (*Automatic Oxygen Dosing System*). See "Automatic Oxygen Dosage" on page 12-6.

### ❖ To change the oxygen quantity

1. In the Main Menu, select **Edit | Edit Elemental Analyzer Parameters**, or click the  icon. The following window is shown with the analyzer operating parameters. See Figure 12-1.

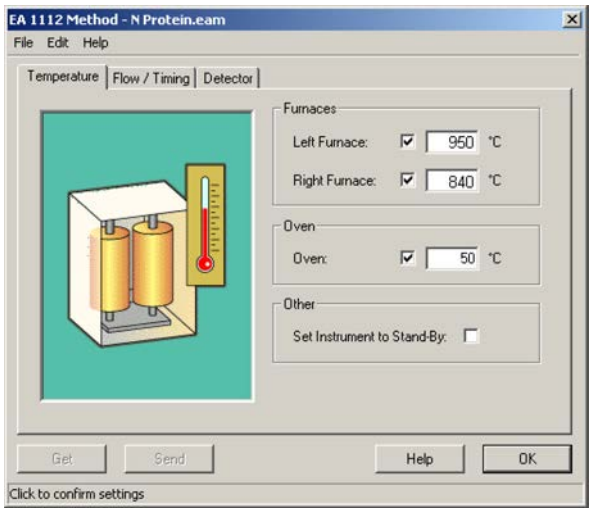



**Figure 12-1.** Example of Analyzer Parameters

2. Select the **Flow/Timing** tab. See Figure 12-2.

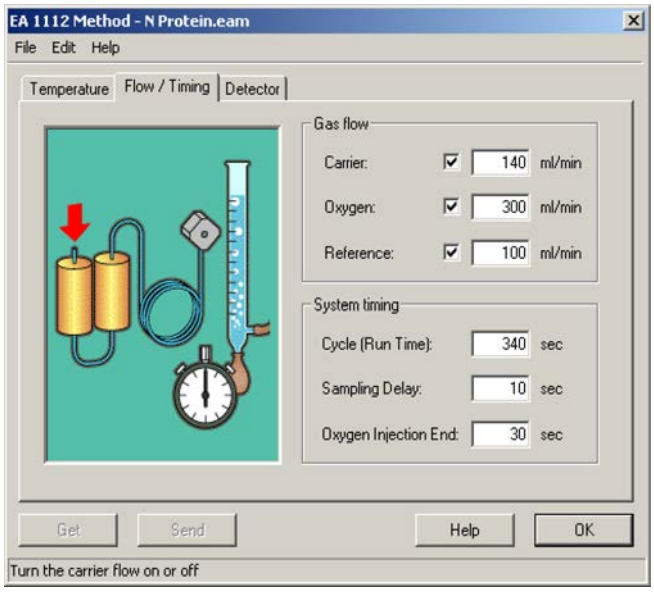



**Figure 12-2.** Flow Timing Tag

   a. To change the time of oxygen introduction, enter the value in the appropriate box **Oxygen Injection End** in the field **System timing**.

   b. To change the oxygen flow rate, enter the flow value in the appropriate box **Oxygen** in the field **Gas flow**.

❖ **To establish the required oxygen quantity**

1. Analyze the sample setting the oxygen flow to its maximum value (300 mL/min).
2. At the end of the analysis, run a blank. If the area value found is equal or very close to the traditional blank value (±50%), it means that the sample is completely burnt without leaving any *memory effects*.
3. 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. You establish the oxygen quantity required for the combustion of that kind of sample with that particular weight.

**Tip** The same result can be obtained by presetting a value of oxygen flow and varying the time of oxygen injection **Oxy Inj End**.

**NOTICE**

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, and 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, and rocks).
- **Nitrogen-Carbon Analysis** — The weighing range is generally between 1 and 5 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, and so on)** — We recommend a weighing range between 100 mg and 1g depending on the sample nature. See "Method for Oxygen Dosage (OxyTune)" on page 12-6.
- **Nitrogen analyses in food and agricultural samples** — We recommend a weighing range between 100 and 500 mg depending on the sample nature. See "Method for Oxygen Dosage (OxyTune)" on page 12-6.

# 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 quantity of oxygen versus other samples that require greater amounts of oxygen. For avoiding that you are obliged every time to modify the weighing range, or adjust the quantity of oxygen, the software provides a table comprising various categories where samples of different nature are memorized or can be memorized. See "Sample Table" on page 13-9.

Selecting, after the weight entry, the category to which the sample belongs, the system will deliver the correct quantity of oxygen required for a complete combustion. This condition is obtained by a feature provided thanks to the method for oxygen dosage. See "Method for Oxygen Dosage (OxyTune)" on page 12-6.

## Method for Oxygen Dosage (OxyTune)

❖ **To dose the oxygen**

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 with different nature were analyzed as follows:
    a. 200 mg of sample weighed and oxygen was injected for 60 seconds in a resulting theoretical quantity of 300 mL.
    b. At the end of the analysis, after taking note of the value, the blank was run again.
    c. The same procedure (sample analysis and subsequent blank analysis) was followed with reducing the injection time at every sequence run. By checking the increased blank value, the combustion critical point was established.
    d. 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.
    e. The sample analysis was repeated starting by reducing the weight to 100 mg, and then by progressively increasing the sample weight until reaching the maximum quantity accepted

by the container. Being ideal factor multiplying value memorized, the amount of oxygen for each single analysis was automatically calculated simply by selecting the sample category.

Based on these results, the table of sample category was created. See "Table of Sample Category" on page 12-7.

## Table of Sample Category

The table shown in Figure 12-3 appears after following the instructions in "To fill the sample table" on page 13-9.

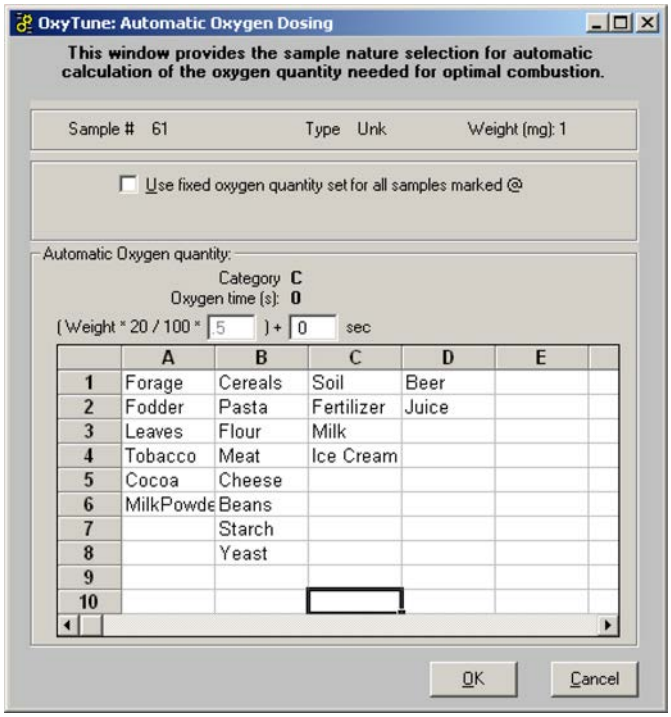



**Figure 12-3.** Table of sample category

The table contains:

- **Four categories of samples A, B, C, and D** — 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, and H** — Sample names not considered (extraneous) in categories A, B, C, and D can be saved. When a free category is selected, the ideal factor value, calculated by using the **Oxygen Dosage method OxyTune™**, must be entered in the appropriate box.
  Before you save the sample name in the new category and the ideal factor value, repeat the procedure "To dose the oxygen" on page 12-6.

**Tip** The correct selection of the group to which the sample belongs helps you keep the analyzer perfectly efficient.

**NOTICE** Non-compliance with allocation criteria may adversely affect analytical results.

The sample category **Blank**, **Bypass**, and **Standard** is the same; it is marked by the symbol **@**.
When **@** is selected, a set time is entered and consequently an equal quantity of oxygen 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.

**Tip** In analyses of nitrogen traces, for example 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.

13

# Running Analyses

This chapter provides information, instructions for running sample analyses, and practical advises for daily operation. The comparison methods for a correct evaluation of the results is also provided.

**Contents**

# Introduction

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

1. Create a directory of analyses.
2. Program the current maintenance (recommended).
3. Choose the calibration.
4. Set up a sample table.
5. Determine the blank value.
6. Run the sequence of analyses.
7. Directory for analyses.

**NOTICE** Before you start the operating sequences, make sure that the instrument start-up operations have been performed as described in Chapter 11, "Instrument Start-up."

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

- Sample table
- Integration parameters
- Calculation parameters
- All the necessary data for running the analysis.

# Programming Current Maintenance

Each reactor, each filter and relevant fillings, need to be replaced according to the analytical configuration used. An average life of its components has been established for each configuration. The **View Maintenance** option indicates when the different components must be replaced.

❖ **To start-up the current maintenance program**

1. In the Main Menu, select **View | View Maintenance**. Depending on your analytical configuration, a window like the following one is shown. See Figure 13-1.

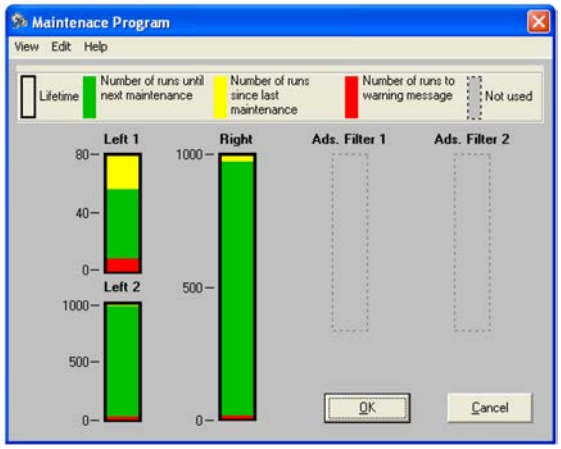



**Figure 13-1.** Maintenance Program Schedule

The example of Figure 13-1 shows the analytical circuit components for which the maintenance routine is required.
Table 13-1 details the analytical circuit components shown in the maintenance program schedule.

**Table 13-1.** Analytical Circuit Components

| Component | Description |
|---|---|
| Left | Represents the oxidation reactor. In Figure 13-1 **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. |

The diagram indicates the active components with colored areas and shows the numerical scale of their lifetime. The meaning of each colored area is indicated in the upper section of the diagram.

A dashed line indicates the components not present in the concerned instrument configuration.

2. 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 is shown. The values shown cannot be changed. See Figure 13-2.

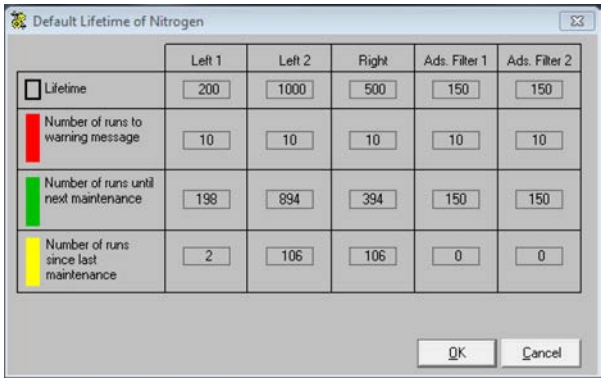



**Figure 13-2.** Maintenance: Example of Default Lifetime Condition

Table 13-2details the default lifetime conditions.

**Table 13-2.** Default Conditions

| 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 shows the message **Check Maintenance**. If the message is ignored and analyses are continued, when the preset number of runs is reached, the message **Alarm** is shown. 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 |

3. If you want to use a different maintenance program from the default one, in the menu **Edit** select **Set Maintenance | Manual**. A window like the following is shown. See Figure 13-3.

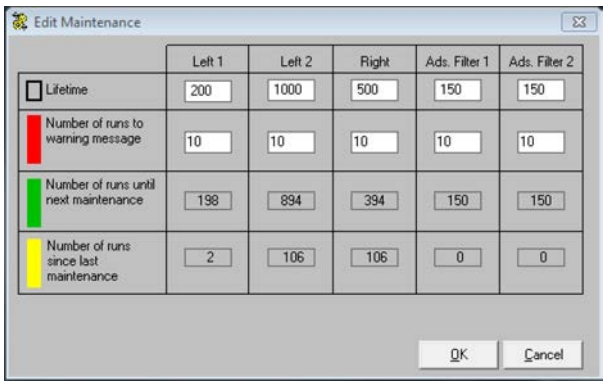



**Figure 13-3.** Maintenance: Manual Program

In the window of Figure 13-3, you can change any value by clicking on the various boxes and entering the new value.

# Instrument Calibration

The Eager*Smart* Data Hnadling Software offers three calibration methods:

- K-Factor
- Linear
- Non Linear

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

$$K = \frac{AreaStd - AreaBlank}{(\%\ Tstd \times Wstd)/100}$$

where:

| | |
|---|---|
| *AreaStd* | = Peak area or integral of standard |
| *AreaBlank* | = Peak area or integral of blank |
| *% Tstd* | = Theoretical percentage of standard |
| *Wstd* | = Weight of standard |

For the calculation of an **Unknown** sample, the Eager*Smart* Data Handling Software uses the reverse formula:

$$Calculated\ \% = \frac{(AreaUnk - AreaBlk)/K}{Wunk} \times 100$$

where:

| | |
|---|---|
| *K* | = Average K-Factor |
| *AreaUnk* | = Peak area or integral of the unknown |
| *AreaBlk* | = Peak area or integral of the blank |
| *Wunk* | = Weight of the unknown |

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.

For selecting the calibration method or view the calibration curves of a memorized method, follow the instructions in "Calibrating Method and Curves" on page 13-7.

## Calibrating Method and Curves

❖ **To calibrate method and curves**

1. Select the calibration method.

   a. In the Main Menu, choose **Edit | Method** or click the  icon. The following window is shown. See Figure 13-4.

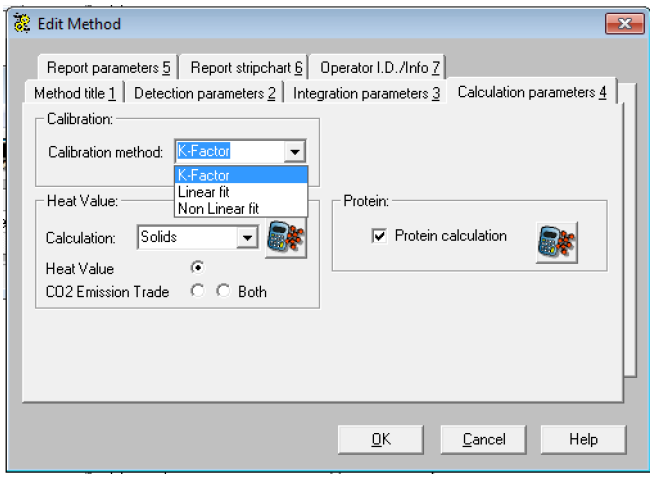



**Figure 13-4.** Method Editor

   b. Select the option **Calculation parameter**.

      i. In the **Calibration Method** combo box in the **Calibration** area, select the calibration method required among the options **K-Factor**, **Linear fit**, or **Not linear fit**.

      ii. If desired, in the **Calculation** combo box in the field **Heat value**, select the type of sample among the options **None**, **Liquids**, or **Solids**. Click the adjacent icon for displaying the calculation scheme related to the selected option.

      iii. In the case of **N/Protein** and **N/Brew** configurations, in the field **Protein**, select **Protein Calculation**. Click the adjacent icon for displaying the relevant calculation scheme.

2. View the calibration curves.

   At the end of analyses of the standard samples, you can display the calibration curve operating as follows:

   a. In the Main Menu, choose **View | View Calibration curve**. A window similar to Figure 13-5 is shown.

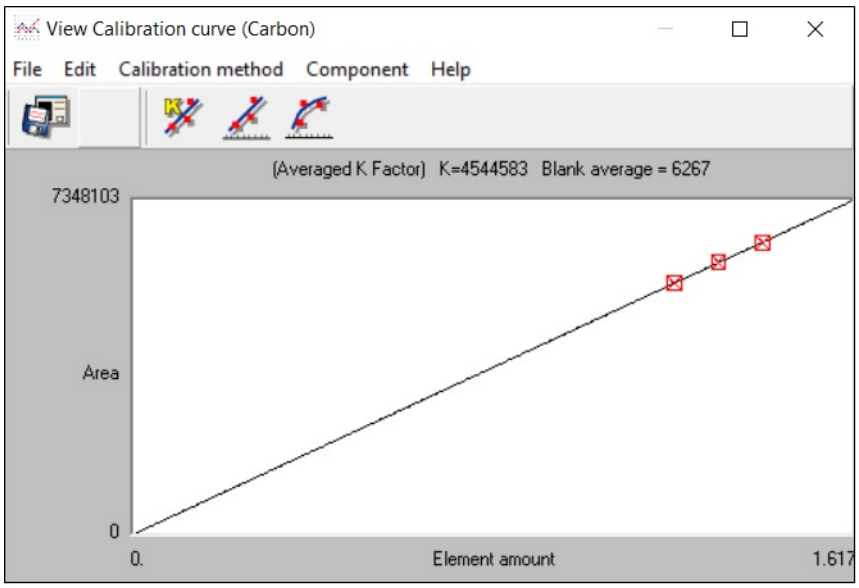
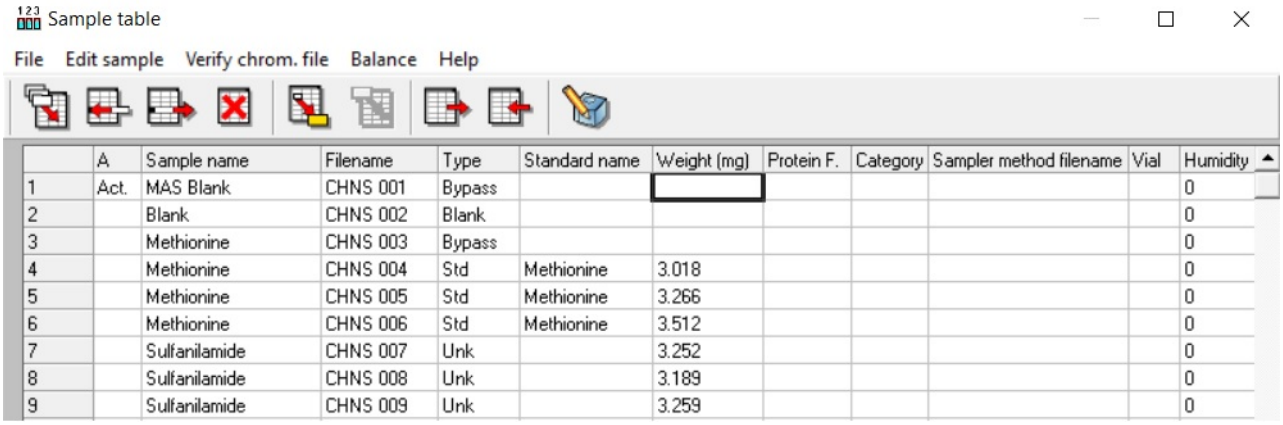



**Figure 13-5.** Example of Calibration Curves

b. Select **Calibration method**. The calibration points with peak area and concentration are shown according to the calculation method.

# Sample Table

The sample table of the analytical method provides information concerning the series of samples to be acquired and processed. See Figure 13-6.




| | A | Sample name | Filename | Type | Standard name | Weight (mg) | Protein F. | Category | Sampler method filename | Vial | Humidity |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Act. | MAS Blank | CHNS 001 | Bypass | | | | | | | 0 |
| 2 | | Blank | CHNS 002 | Blank | | | | | | | 0 |
| 3 | | Methionine | CHNS 003 | Bypass | | | | | | | 0 |
| 4 | | Methionine | CHNS 004 | Std | Methionine | 3.018 | | | | | 0 |
| 5 | | Methionine | CHNS 005 | Std | Methionine | 3.266 | | | | | 0 |
| 6 | | Methionine | CHNS 006 | Std | Methionine | 3.512 | | | | | 0 |
| 7 | | Sulfanilamide | CHNS 007 | Unk | | 3.252 | | | | | 0 |
| 8 | | Sulfanilamide | CHNS 008 | Unk | | 3.189 | | | | | 0 |
| 9 | | Sulfanilamide | CHNS 009 | Unk | | 3.259 | | | | | 0 |

**Figure 13-6.** Example of Sample Table

❖ **To fill the sample table**

1. Select **Edit sample | Fill Sample table** or click the icon. The window shown in Figure 13-7 offers all the functions necessary to fill, change, or cancel the **Sample table**.

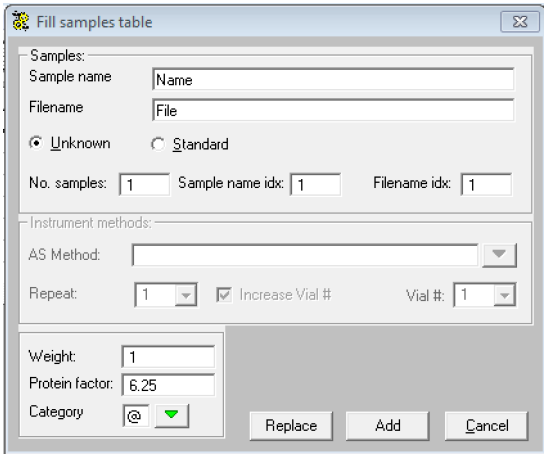



**Figure 13-7.** Window for Filling Sample Table

a. In the **Samples** area, 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 **Filename** text box, type the filename to be used to save the sample.

iii. In the box **No. samples**, enter the number of up to 200 samples to be analyzed.

iv. Make sure that the **Unknown** option is selected.

2. Leave both **Sample name idx** and **Filename idx** set to **1**.

a. In the field **Instrument methods**, enter the required parameters if the instrument is equipped with the autosampler for liquids.

b. In the left bottom field, enter the following:

i. Leave **Weight** set to **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 select the sample category, press the adjacent green down-arrow. The window of Figure 13-8 is shown.

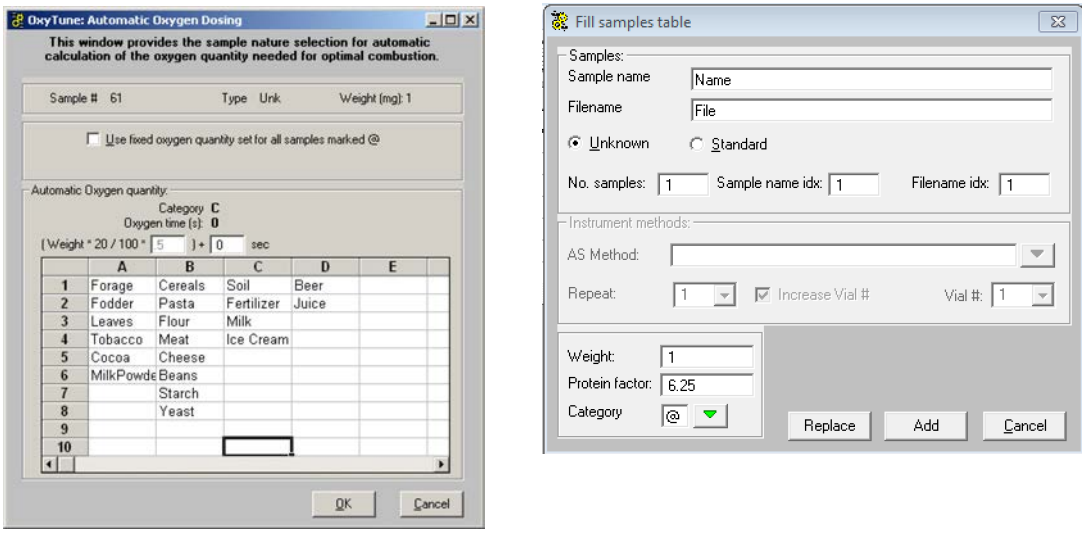



**Figure 13-8.** Window for Selecting the Sample Category

3. At the end of editing, click **OK**, then **Replace**. The sample table reappears. In the sample table grid, you find all the informations entered. See Figure 13-9.

**Tip** In the case of analyses of liquid samples, the sample table grid shows the column **Density** in which you type the density of the liquid sample. The density will automatically be turned into weight.

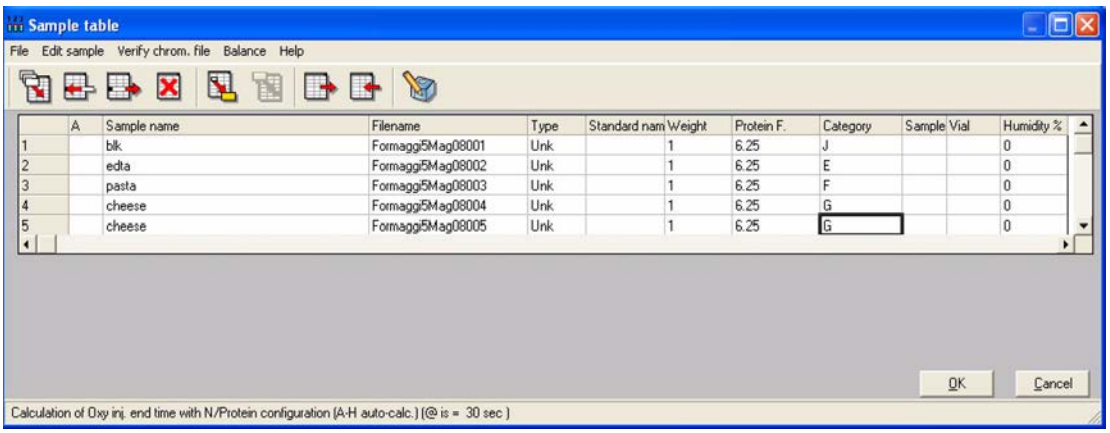



**Figure 13-9.** N/Protein: Window of Sample Table Editing

# Determining the Blank Value

❖ **To determine the blank value**

1. Put in the autosampler an air-tightly closed container. When **N/Protein** is used, it is suggested introducing about **50-80 mg** of **sugar** into the container to avoid that a high quantity of **oxygen** freely flows in the reduction reactor.

2. In the Main Menu, select **Edit | Sample table**, or click the  icon. The sample table is shown. See Figure 13-6 on page 13-9.

   a. In the sample table grid, click the column **Type**. Click the arrow, and the following window is shown for the selection of the type of sample. See Figure 13-10.

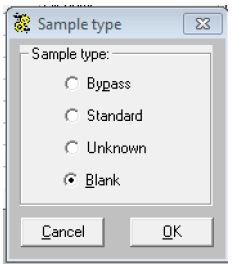



**Figure 13-10.** Window for selecting Sample Type

   b. In the window of Figure 13-10 select **Blank**, click **OK** to confirm, and go back to the Sample table.

   c. Click **OK** to confirm and go back to the Main Menu.

3. In the Main Menu, select **Run | Start Single Sample Data Acquisition**.

4. When the analysis run time has elapsed, compare the chromatogram obtained with that of the final test provided with the instrument.

Two procedures are available, depending on whether a printer is available or not, for comparing the chromatogram obtained with that of the final test, and to check the blank value.

- In the first case, see "To check the blank value with a printer available" on page 13-13.
- In the second case, see "To check the blank value with no printer available" on page 13-15.

## Checking the Blank Value with a Printer Available

❖ **To check the blank value with a printer available**

1. In the Main Menu, select **Edit | Method**, or click the  icon. The Method editor window is shown. See page 13-13.

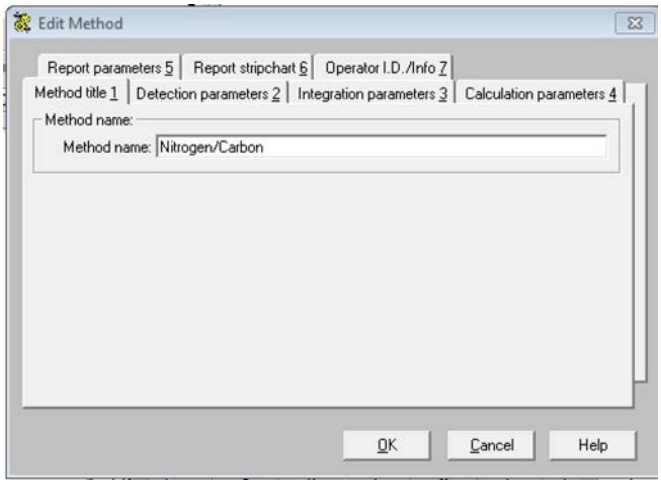



**Figure 13-11.** Example of Method Editor Windows

2. Select the **Report Parameters** tab. The window of Figure 13-12 is shown.

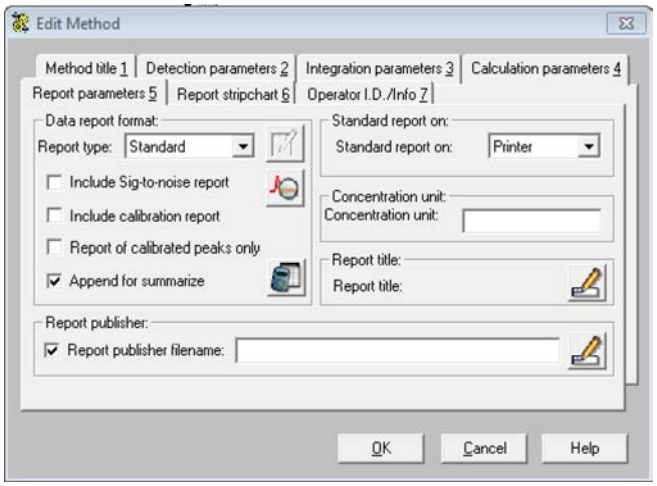



**Figure 13-12.** Report Parameter Window

   a. In the **Data report format** area, select **Standard** in the drop-down combo **Report type.** Then select **Append to summarize**.

   b. In the **Standard report on** area, select in the box **Report destination** the option **Printer**.

3. In the window shown in Figure 13-11 on page 13-13, select the **Report Stripchart** tab. The following dialog window is shown. See Figure 13-13.

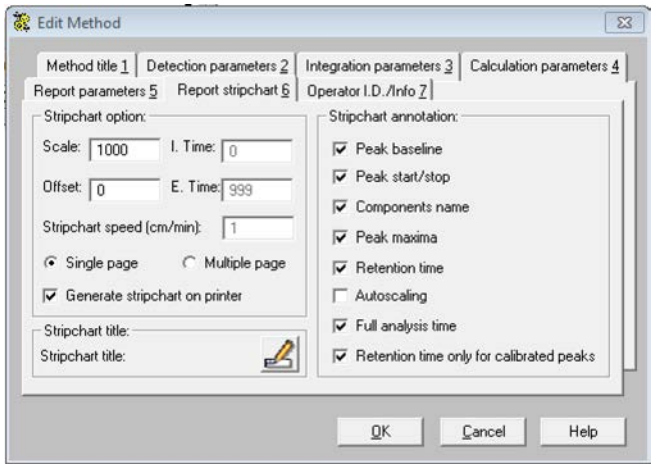



**Figure 13-13.** Report Stripchart Window

   a. In the **Stripchart annotation** area, clear the **Autoscaling** check box.

   b. If required, modify the values in the field **Stripchart option** area.

4. Click **OK** to confirm the settings.

5. In the Main Menu, select **Recalculation | Recalculation**. The following dialog window is shown. See Figure 13-14.

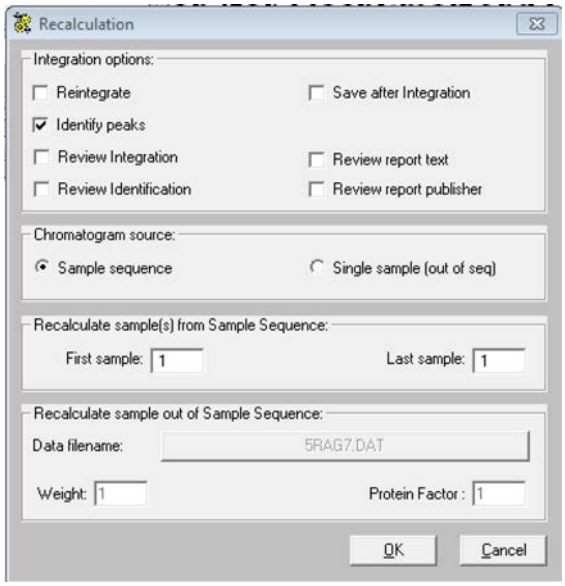



**Figure 13-14.** Recalculation Window (1)

   a. In the **Integration options** area, select **Identify peaks**.

b. In the field **Chromatogram source,** select **Sample sequence**.

c. In the field **Recalculate sample(s) from Sample Sequence,** set **First Sample 1** and **Last Sample 1**.

6. Click **OK**. The report of values will be printed.

## Checking the Blank Value with No Printer Available

❖ **To check the blank value with no printer available**

1. In the Main Menu, select **Recalculation | Recalculation**. The dialog window of Figure 13-15 is shown.

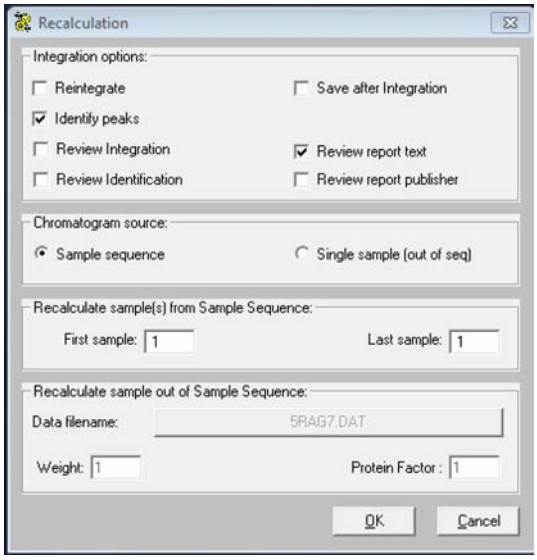



**Figure 13-15.** Recalculation Window (2)

a. In the field **Integration options**, select **Review report text**.

b. In the field **Integration options,** select **Identify peaks**.

c. In the field **Chromatogram source,** select **Sample sequence**.

d. In the field **Recalculate sample(s) from Sample Sequence,** set **First Sample 1** and **Last Sample 1**.

2. Click **OK**. The report of values is shown.

## Evaluating the Blank Value

The blank value is a function of the type of containers used and of oxygen purity. Verify that the values found are within acceptable limits versus those reported in the final test certificate. If the values found are higher, see the possible causes and the relevant remedies listed in Table 13-3.

**Table 13-3.** Blank Value Diagnostic Guide

| Blank | Cause and remedy |
|---|---|
| Nitrogen | If the value of nitrogen is definitely higher than that indicated, repeat the blank according to the above described procedures.<br>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.<br>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. |
| 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 the circuit or the oxygen cylinder. The value tends to decrease in time. |
| Sulfur | Generally absent or negligible. |
| Oxygen | Generally negligible. High values are due to the container contamination. |

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

# Sequence of Analyses

**Tip** Before the shipment, every instrument is submitted to an analytical final test procedure depending on 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 perform the analytical sequence**

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

- "Mode 1: Manual entry of the weights" on page 13-17
- "Mode 2: Automatic entry of the weights from the balance to the computer" on page 13-19

### Mode 1: Manual entry of the weights

1. Weigh the samples and put them sequentially into the autosampler tray.
2. In the Main Menu, select **Edit | Sample table** or click the  icon. The sample table is shown.
   a. Enter **Sample name**, **Type** and **Weight**. In case of **N/Protein** and **N/Brew** configurations, also specify the sample **Category**.
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 the column **Type**. Click the arrow to display the window for the sample type selection.
   b. Depending on the sample type, select **Bypass**, **Standard**, **Unknown**, or **Blank** as shown in Figure 13-16.

**Tip** When you select **Standard**, the window of Figure 13-17 appears. Set the desired standard.

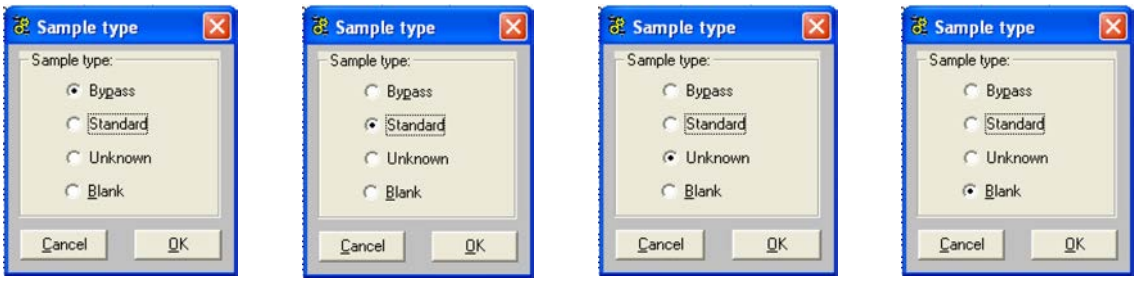



**Figure 13-16.** Selection of Sample Type

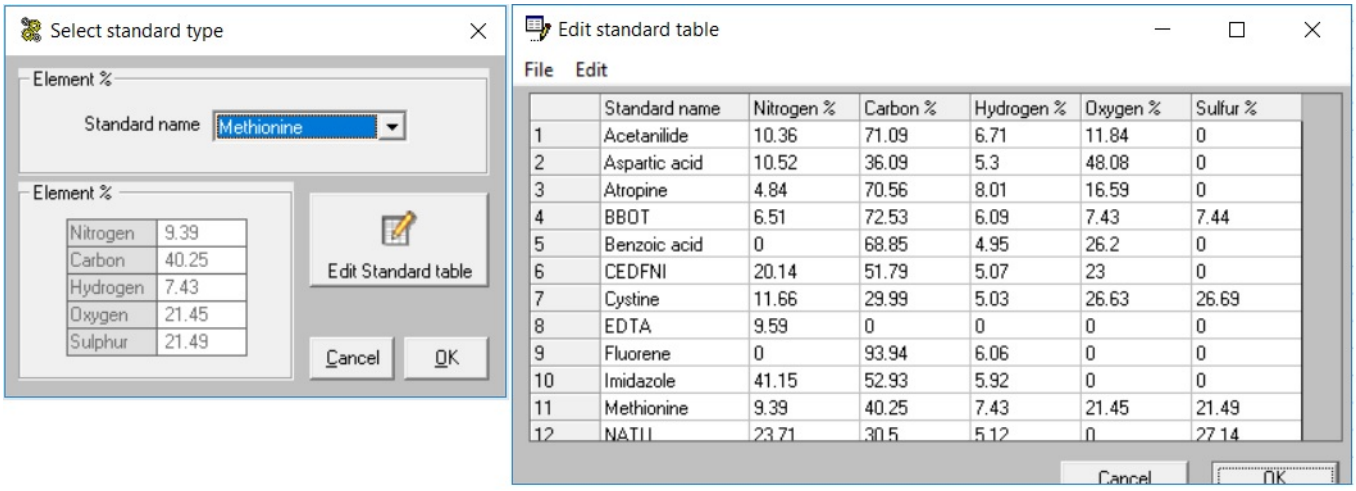



**Figure 13-17.** Selection of Standard

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

**Tip** New standards may be saved by clicking **Edit Standard table**. After the number 5, add the standards of interest and the percentages of the relevant elements. See Figure 13-18.

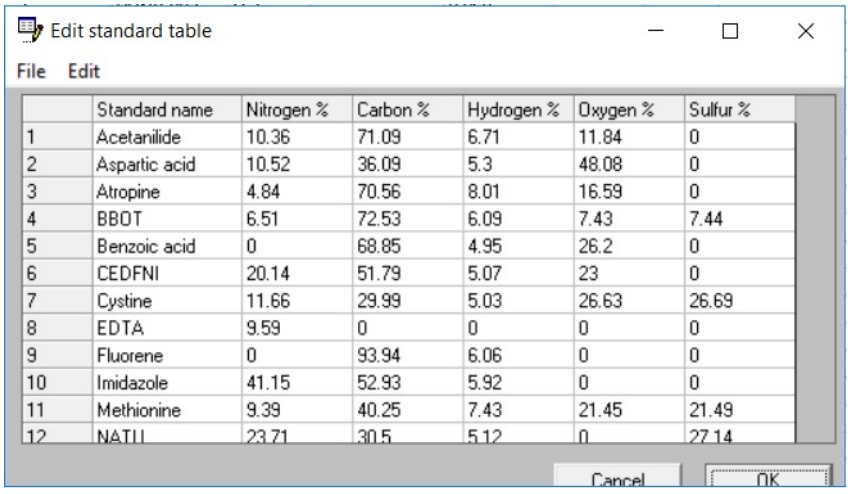



**Figure 13-18.** Edit Standard Table

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 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. See "Comparing Analytical Results and Final Test Results" on page 13-21.

### Mode 2: Automatic entry of the weights from the balance to the computer

1. Connect the RS 232 connecting cable between the balance and the computer.

2. Select **Sample table | Balance | Balance setup**. The following window is shown. See Figure 13-19.

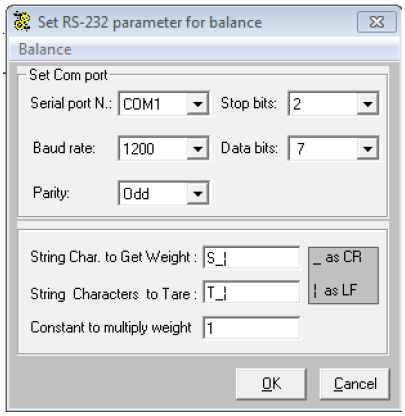



**Figure 13-19.** Balance Parameters Setting

3. Select the serial port of the computer (for example, COM1) to which the balance is connected. Make sure that the serial port is different from the serial port selected for the analyzer.

4. Select the **Balance** menu.

   a. Choose the type of balance in use.

   b. Press **OK**. The dialog window of Figure 13-19 is shown again.

   c. Press **OK** to return to the sample table.

5. In the sample table, select **Balance | Receive weight from balance**. After you have selected the sample number (for example, sample

number 3), click **Weight**. When you click the down arrow, the following window is shown. See Figure 13-20.

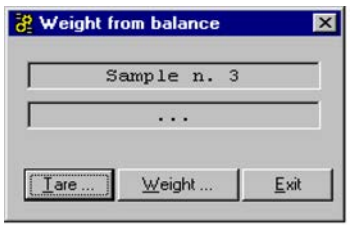



**Figure 13-20.** Weight From Balance

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

   **Tip** If the balance Mettler Toledo ME104 is used, perform the tare with 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 click the  icon.

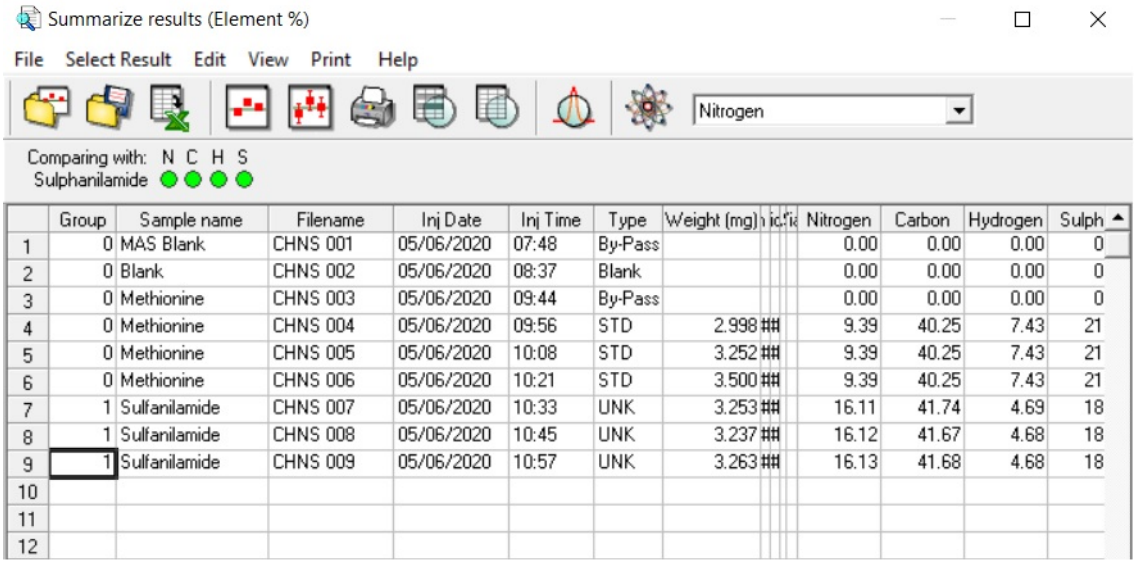
8. In the Main Menu, select **Run | Start sequence of sample**.

At the end of the analytical sequence, the results obtained must be compared with those of the final test. See "Comparing Analytical Results and Final Test Results" on page 13-21.

# Comparing Analytical Results and Final Test Results

❖ **To compare analytical results and final test results**

1. In the Main Menu, select **Recalculation | Summarize results** or click the  icon. The following table is shown. See Figure 13-21.




| | Group | Sample name | Filename | Inj Date | Inj Time | Type | Weight (mg) | Nitrogen | Carbon | Hydrogen | Sulph |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 | MAS Blank | CHNS 001 | 05/06/2020 | 07:48 | By-Pass | | 0.00 | 0.00 | 0.00 | 0 |
| 2 | 0 | Blank | CHNS 002 | 05/06/2020 | 08:37 | Blank | | 0.00 | 0.00 | 0.00 | 0 |
| 3 | 0 | Methionine | CHNS 003 | 05/06/2020 | 09:44 | By-Pass | | 0.00 | 0.00 | 0.00 | 0 |
| 4 | 0 | Methionine | CHNS 004 | 05/06/2020 | 09:56 | STD | 2.998 | 9.39 | 40.25 | 7.43 | 21 |
| 5 | 0 | Methionine | CHNS 005 | 05/06/2020 | 10:08 | STD | 3.252 | 9.39 | 40.25 | 7.43 | 21 |
| 6 | 0 | Methionine | CHNS 006 | 05/06/2020 | 10:21 | STD | 3.500 | 9.39 | 40.25 | 7.43 | 21 |
| 7 | 1 | Sulfanilamide | CHNS 007 | 05/06/2020 | 10:33 | UNK | 3.253 | 16.11 | 41.74 | 4.69 | 18 |
| 8 | 1 | Sulfanilamide | CHNS 008 | 05/06/2020 | 10:45 | UNK | 3.237 | 16.12 | 41.67 | 4.68 | 18 |
| 9 | 1 | Sulfanilamide | CHNS 009 | 05/06/2020 | 10:57 | UNK | 3.263 | 16.13 | 41.68 | 4.68 | 18 |
| 10 | | | | | | | | | | | |
| 11 | | | | | | | | | | | |
| 12 | | | | | | | | | | | |

**Figure 13-21.** Example of Summarize Results

2. In the **Group** text box, 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 the Results

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

**Tip** It is suggested to see Chapter 10, "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.

**Tip** 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. See "Mode 2: Automatic entry of the weights from the balance to the computer" on page 13-19.

4. If the weight of an unknown sample is wrong, then the error immediately becomes clear from its percentage result. If the error is due to the weight of one or more standards, then the three results of the unknown sample will all be wrong.

5. Another cause of error is the incorrect integration of the peak. If it happens, then the correction of the baseline is required proceeding as follows:

   a. Open the chromatogram selecting the sample to adjust. In the Main Menu, choose **View | View Chromatogram | Peak**.

   b. To modify the baseline, choose **Move peak start** or **Move peak end** accordingly.

   c. **Save** the new chromatogram.

   d. Recalculate the chromatogram following the instructions in **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, the maximum and minimum acceptable values must be defined. These values can be stored and used as comparative parameters for next analyses. A similar condition occurs if you have many samples to run, for which you 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, see "Method to Compare Results" and "Graphic Display of the Result" on page 13-25.

## Method to Compare Results

After having analyzed the standard samples, and checked the instrument precision, proceed as follows:

❖ **To compare results**

1. In the Main Menu, select **Recalculation | Summarize** results, or click the  icon. The Summarize result table is shown. See Figure 13-21 on page 13-21.

2. Select the **Edit** menu and then **Select reference compounds.** A table like the following is shown. See Figure 13-22.

Compound of reference selection

Selected: Aspartic Acid | None

| | Name | Nitrogen | ±Ran. | Carbon | ±Ran. | Hydrogen | ±Ran. | Sulphur | ±Ran. | Oxygen | ±Ran. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Acetanilide | 10.36 | 0.1 | 71.09 | 0.3 | 6.71 | 0.1 | 0 | 0 | 11.84 | 0.12 |
| 2 | Aspartic Acid | 10.52 | 0.1 | 36.09 | 0.28 | 5.3 | 0.08 | 0 | 0 | 48.08 | 0.3 |
| 3 | Atropine | 4.84 | 0.07 | 70.56 | 0.3 | 8.01 | 0.1 | 0 | 0 | 16.59 | 0.18 |
| 4 | BBOT | 6.51 | 0.1 | 72.53 | 0.3 | 6.09 | 0.1 | 7.44 | 0.1 | 7.43 | 0.1 |
| 5 | Benzoic Acid | 0 | 0 | 68.85 | 0.3 | 4.95 | 0.08 | 0 | 0 | 26.2 | 0.25 |
| 6 | CEDFNI | 20.14 | 0.2 | 51.79 | 0.3 | 5.07 | 0.08 | 0 | 0 | 23 | 0.22 |
| 7 | dl-Methionine | 9.39 | 0.1 | 40.25 | 0.3 | 7.43 | 0.1 | 21.49 | 0.2 | 21.45 | 0.2 |
| 8 | Imidazole | 41.15 | 0.3 | 52.93 | 0.3 | 5.92 | 0.09 | 0 | 0 | 0 | 0 |
| 9 | L-Cystine | 11.66 | 0.12 | 29.99 | 0.28 | 5.03 | 0.08 | 26.69 | 0.25 | 26.63 | 0.25 |
| 10 | Nicotinamide | 22.94 | 0.22 | 59.01 | 0.3 | 4.95 | 0.08 | 0 | 0 | 13.1 | 0.14 |
| 11 | Sulphanilamide | 16.27 | 0.16 | 41.84 | 0.3 | 4.68 | 0.07 | 18.62 | 0.2 | 18.58 | 0.2 |
| 12 | Urea | 46.65 | 0.3 | 20 | 0.2 | 6.71 | 0.1 | 0 | 0 | 26.64 | 0.25 |
| 13 | Thocophenol Nicotinate | 2.61 | 0.3 | 78.46 | 0.3 | 9.97 | 0.1 | 0 | 0 | 8.96 | 0.1 |
| 14 | Polyethilene | 0 | 0 | 85.71 | 0.3 | 14.47 | 0.15 | 0 | 0 | 0 | 0 |
| 15 | Polystyrene | 0 | 0 | 91.95 | 0.3 | 7.84 | 0.1 | 0 | 0 | 0 | 0 |

OK | Cancel

**Figure 13-22.** Example of the Reference Compounds Selection Table

a. In the text-box **Name**, enter the sample name.

b. In the text boxes **Nitrogen**, **Carbon**, **Hydrogen**, **Sulfur**, and **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 **±%**) 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.

**Tip** The calculation of the deviation (±Range) is valid only for pure organic standards.

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

4. At the end of the analytical cycle, compare the values found with the theoretical ones.

a. Go to the table of Figure 13-22 doing what is described in steps 1 and 2. Select the sample to be compared, then click **OK**. The Summarize Results table is shown.

b. In Summarize Results, select the sample to be compared as shown in the example of Figure 13-23.

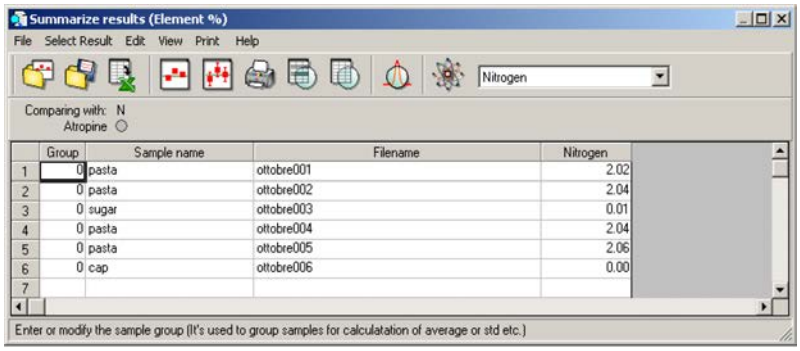



**Figure 13-23.** Summarize Results Table: Comparison of Samples (1)

c. Select **View | Compare to reference compound**. On the left side over the **Summarize Results** table, you will read the name of the analyzed sample followed by one or more **green/red light** indicators depending on to the number of analyzed elements. See Figure 13-24.

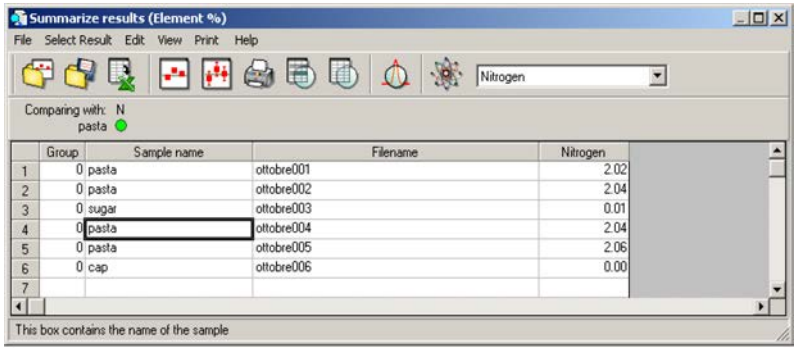



**Figure 13-24.** Summarize Results Table: Comparison of Samples (2)

- **Green Light** indicator — The values found are close to the theoretical values and within the preset error limits.
- **Red Light** indicator — 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 visualized. 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**

1. In the **Summarize results** table, select the group of samples to be visualized.
2. Click the **Show summary graph of selected group** icon  . A window like the following is shown. See Figure 13-25.

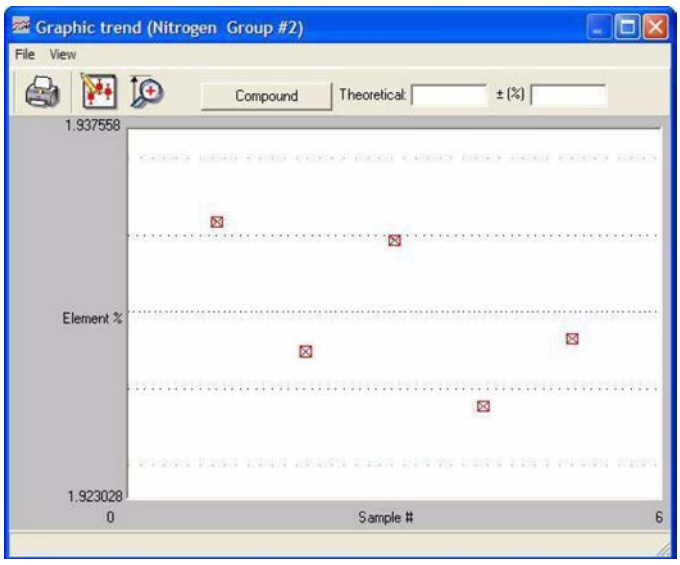
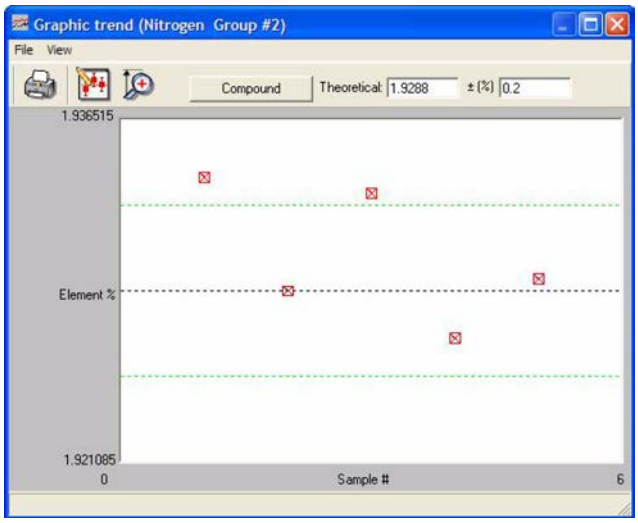



**Figure 13-25.** Summarize Graphic (1)

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 is shown where a graph is plotted consisting of two parallel green lines, a white middle line, and red small squares. See Figure 13-26.




Middle line = Theoretical value
Green lines = Minimum and maximum acceptable values
Red square = Sample analyzed

**Figure 13-26.** Summarize Graphic (2)

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. When clicked, the red square becomes white, and the percent value is shown. See Figure 13-27.

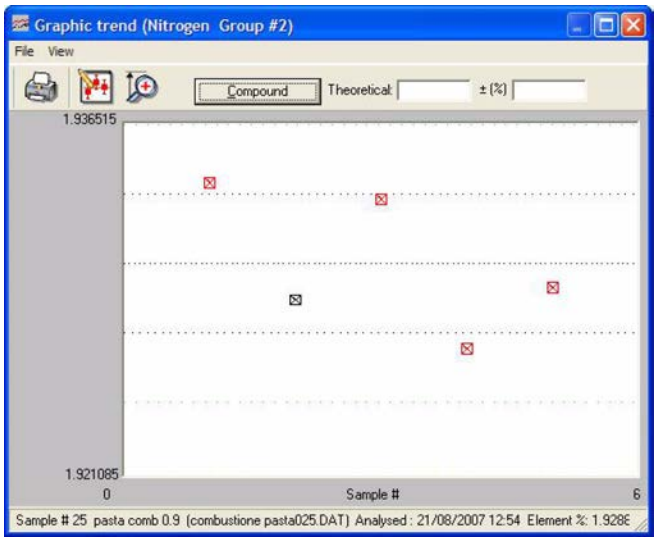



**Figure 13-27.** Summarize Graphic (3)

# Post-Analysis Operations

For obtaining precise, accurate, and constant in time results, and at the same time to reduce operating costs, we recommend that you follow some practical suggestions.


- "Putting the Instrument in Standby Mode" on page 13-27
- "Shutting Off Furnaces, Detector, and Cutting Off Gas Flows" on page 13-28
- "Setting the Wake-up Function" on page 13-30
- "Setting the Auto-Start Function" on page 13-30


## 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 carrier and reference flows are brought to 10 mL/min each.

**Tip** Using argon carrier gas, both carrier and reference flows are brought to 2-5 mL/min each.

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

❖ **To manually set the standby function**

1. In the Main Menu, select **View | View Elemental Analyzer Status** or click the  icon. The page of Figure 13-28 is shown.

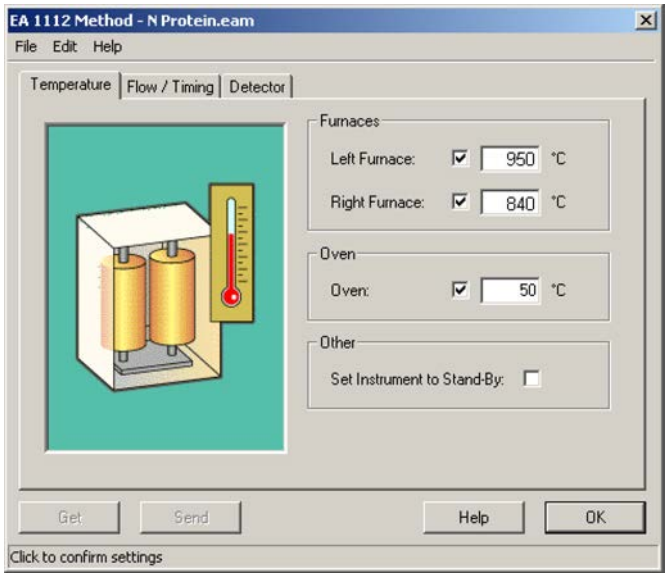



**Figure 13-28.** Analyzer Parameters

2. In the **Other** area, select **Set instrument to Stand-By**.
3. Click **Send** to send the command to the instrument and click **OK**.

❖ **To automatically set the standby function**

1. In the Main Menu, click the icon. The window of Figure 13-29 is shown.

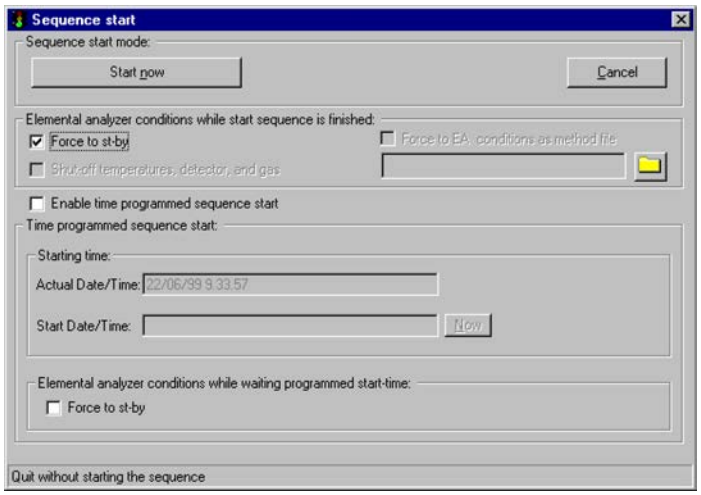



**Figure 13-29.** Sequence Start Window (1)

2. In the **Elemental analyzer conditions while start sequence is finished** area, select the **Force to Stand-by function** 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 shutoff of furnaces and detector and the cutoff of gas flows can be programmed as described in the following operating sequence.

❖ **To shut off temperature, detector and gas**

1. In the Main Menu, click the icon. The window of Figure 13-30 is shown.

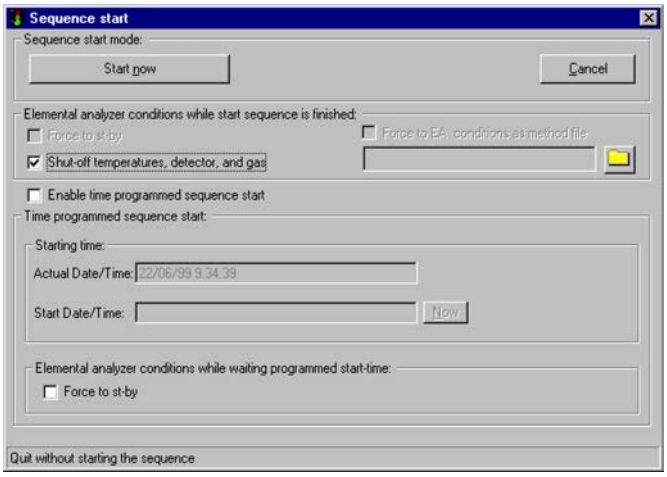



**Figure 13-30.** Sequence Start Window (2)

2. In the **Elemental analyzer conditions while start sequence is finished** area, select **Shut-Off temperature, detector and gas**.

3. When the oxidation reactor requires cleaning, the furnace should be switched off and then the helium flow reduced with the **Stand-by** function.

    a. In the Main Menu, select **Edit | Edit Elemental Analyzer parameters,** or click the  icon. The window of Figure 13-31 is shown.

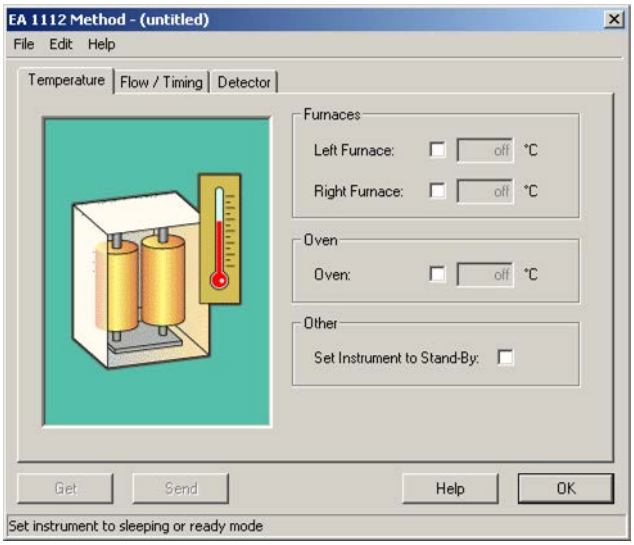



**Figure 13-31.** Edit Elemental Analyzer Parameters

    b. In the **Furnaces** field, select **Left Furnace** to enable the **Off** condition.

    c. In the **Other** field, select **Set instrument to Stand-By**.

d. Click **Send** to send the command to the instrument. Then click **OK**.

## Setting the Wake-up Function

These are timed functions, which can be programmed to minimize dead times.

❖ **To set the wake-up function**

To pass from **Stand-by** to **Ready** status, operate as follows:

1. In the Main Menu, select **View | View Elemental Analyzer status** or click the  icon.
2. Select the **Auto-ready** menu. The window of Figure 13-32 is shown.
   a. In the field **Control**, set the date and time for the activation of the **Wake-up** function.
   b. Click **Activate** and then **OK** to confirm.

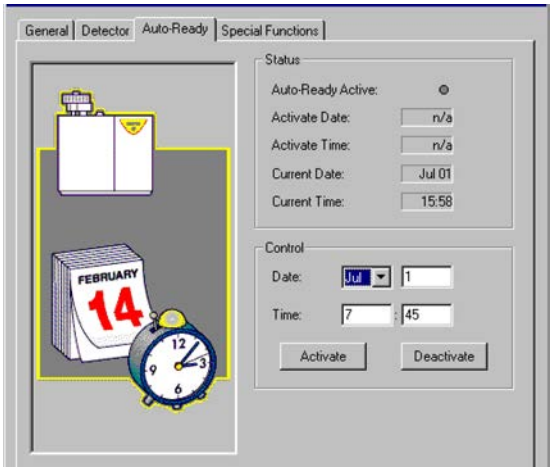



**Figure 13-32.** Analyzer Status

## Setting the Auto-Start Function

If you wish the Auto-Ready to be followed by Auto-Start when programming an analytical sequence, operate as follows:

❖ **To set the Auto-Start function**

1. In the Main Menu, click the  icon. The following window is shown.

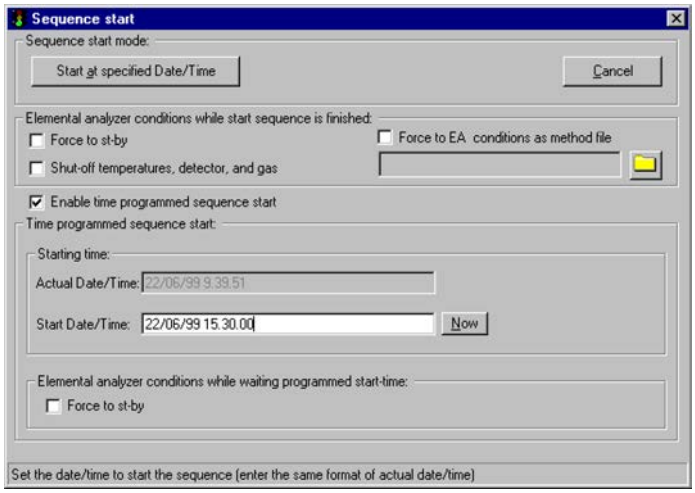



**Figure 13-33.** Start sequence

2. Select **Enable time programmed sequence start**.

    a. In the **Starting time** field, click **Now**.

    b. In the **Start Date/Time** text box, enter the date and the time of the function activation.

**Tip** The function activation should be programmed with a delay of at least 60 minutes versus the time programmed for the Auto-Ready Function. This allows 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, the Flash*Smart* Elemental Analyzer will provide correct results.

The lack of the above conditions will be indicated by anomalies in the chromatograms and the relevant analytical reports.

Table 13-4 reports the most common anomalies with the relevant diagnosis and remedy.

**Table 13-4.** Analytical Troubleshooting Guide (Sheet 1 of 2)

| Problem | Diagnosis | Remedy |
|---|---|---|
| High nitrogen blank. | Presence of leak. | Check that helium and oxygen lines are sealed and in case eliminate possible leak. |
| | Oxygen line or cylinder contaminated. | Purge for any minutes.<br>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. |
| 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. |

**Table 13-4.** Analytical Troubleshooting Guide (Sheet 2 of 2)

| Problem | Diagnosis | Remedy |
|---|---|---|
| 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 |

14

# Maintenance

This chapter provides information on the current and periodic maintenance of the instrument, and the instructions for maintaining the MAS Plus autosampler.

**Contents**



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

**⚠CAUTION** When the instrument is switched off, consider that its does not cool down immediately, but heat tends to concentration in the upper part of the furnaces area.
The openings provided for the chamber aeration will cause a slow cooling of the same, which however, in the vicinity of the areas marked with the symbol “hot surfaces”, might even reach temperatures higher than ambient temperature. Therefore in the minutes immediately following the instrument switching off, the operator must consider this risk and pay adequate attention during any maintenance operations following the use of the instrument.

# Cleaning the Instrument

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

❖ **To clean the instrument**

1. Externally clean the instrument with a soap and water solution, or with a household non-abrasive product. Avoid that any liquid seeps into the instrument.
2. If you suspect that a substance used for cleaning or a product submitted to analysis has infiltrated 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.

***NOTICE*** It is your responsibility to avoid that dangerous liquids and/or materials seep inside the Flash*Smart* Elemental Analyzer during operation and maintenance.

# Maintaining the Instrument

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*Smart* Elemental Analyzer instrument is evaluated in years.
- Replacement of the seals of the reactors coupling unions placed on the furnace compartment base.

For some maintenance operations, the furnaces and the oven need to be at room temperature. Follow the instructions given in "Shutting Off Furnaces, Detector, and Cutting Off Gas Flows" on page 13-28.

## Replacing the Reactors and the Adsorption Filters

The replacement of the reactors and the adsorption filters is performed after a preset number of analyses according to the setting entered in "Programming Current Maintenance" on page 13-3.

Replace and install the reactors and the filters according to the instructions in Chapter 8, "Preparing the Reactors and the Adsorption Filters."

- "Removing the Quartz Reactor from the Furnace" on page 9-13
- "Removing the Alloy Steel (HPAR) Reactors from the Furnaces" on page 9-14
- "Removing the Adsorption Filters" on page 9-16
- "Installing the Quartz Reactor into the Furnace" on page 9-3
- "Installing the Alloy Steel (HPAR) Reactor into the Furnace" on page 9-5

# Replacing the Filling Materials

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

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

Perform these operations according to these instructions:

- "To replace the filling material in alloy steel reactors" on page 14-4
- "To replace the filling material in adsorption filters" on page 14-5

### ❖ To replace the filling material in alloy steel reactors

| Material required |
|---|
| Tool for cleaning special steel reactors P/N 27606025 (included in the Standard Outfit of N/Protein, N/Brew, and NC Soils configurations. |
| Filling materials |

**⚠CAUTION** Before you start the operation, make sure that the furnaces are at room temperature.

1. Remove the special steel reactor from the furnace following the instructions given in "Removing the Alloy Steel (HPAR) Reactors from the Furnaces" on page 9-14
2. Introduce the cleaning tool into the reactor as shown in Figure 14-1.

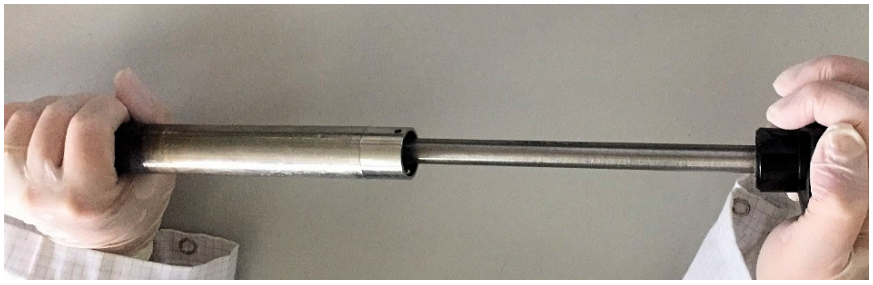


**Figure 14-1.** Removal of the Filling Material from an Alloy Steel Reactor

3. Rotate the tool exerting a slight pressure to scrape off the filling material and collect the removed filling material.
4. Repeat steps 2 and 3 until the complete elimination of the exhausted filling materials.

5. At the end of the operation, restore the layers of filling materials introducing into the reactor the new ones. To do this, see "Preparing the Reactors" on page 8-35 and "To prepare the crucible" on page 8-40 depending on the analyzer configuration.

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

| **Material required** |
| --- |
| Filling materials |

1. Remove the adsorption filter from the detector compartment according to the instructions given in "Removing the Adsorption Filters" on page 9-16.
2. Unscrew the filter nut and remove the filling material.

**Tip** 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.

3. Restore the sequence of the layers of filling materials introducing the new ones into the filter.
   See "Introduction to the Preparation of Reactors and Filters" on page 8-6 and "Preparing the Adsorption Filters" on page 8-42, depending on the analyzer configuration.

## Removing the Ashes and Cleaning the Crucible

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

**⚠CAUTION** Before you start the operation, make sure that the furnaces are at room temperature.

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

1. Remove the crucible from the reactor.
2. Depending on the crucible material, do one of the following:
   - If the crucible is made in quartz, then remove the ashes with a spatula.
   - If the crucible is made of steel, then do the following:

a. Introduce the cleaning tool into the crucible as shown in Figure 14-2.

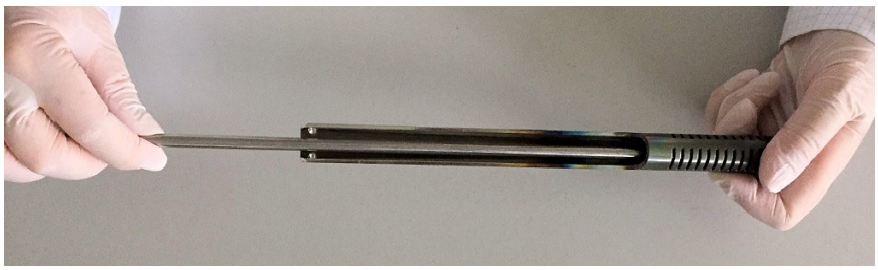


**Figure 14-2.** Removal of Ashes and Quartz Wool from the Crucible

b. Rotate the cleaning tool exerting a slight pressure in a way to scrape off the ashes and the quartz wool material, then collect the material removed.

c. At the end of the operation, introduce new quartz wool into the crucible.

## Replacing the Gas Chromatographic Column

The instrument rarely requires the gas chromatographic column replacement.

❖ **To replace the gas chromatographic column**

| Material required |
| --- |
| Open end wrenches for the column fittings |

The gas chromatographic column can be installed outside or inside of the oven compartment.

1. Open the instrument right door and remove the adsorption filters from their securing clips. See Figure 14-3.

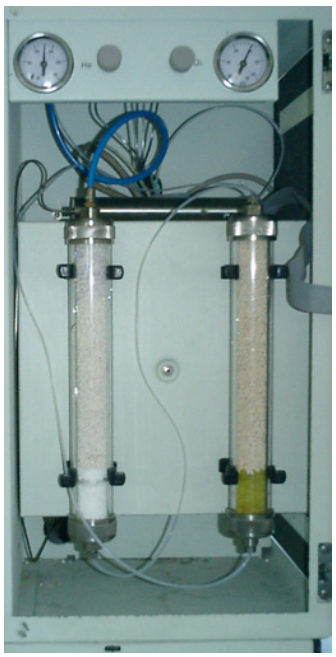


**Figure 14-3.** View of the Oven Compartment

**Tip** Depending on the instrument configuration, one or two adsorption filters can be in the detector compartment.

2. Loosen the 4 fixing screws securing the protection panel. See Figure 14-4.

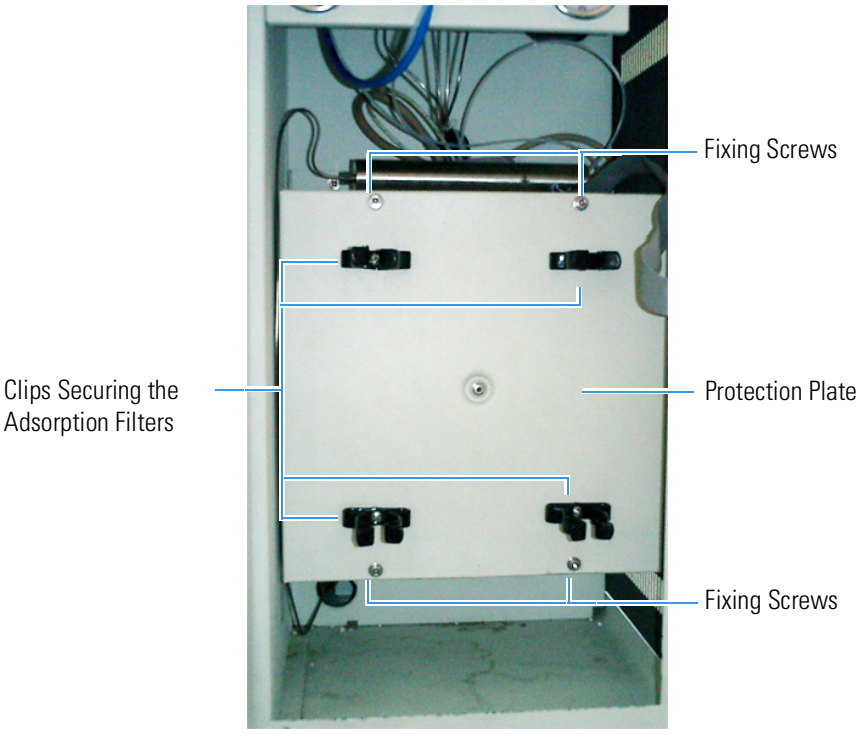



**Figure 14-4.** Access to the Detector

Figure 14-5 shows the detector compartment, the heating block where the detector is housed, and the gas chromatographic column.

**Tip** Depending on the instrument configuration, one or two GC columns can be in the detector compartment.

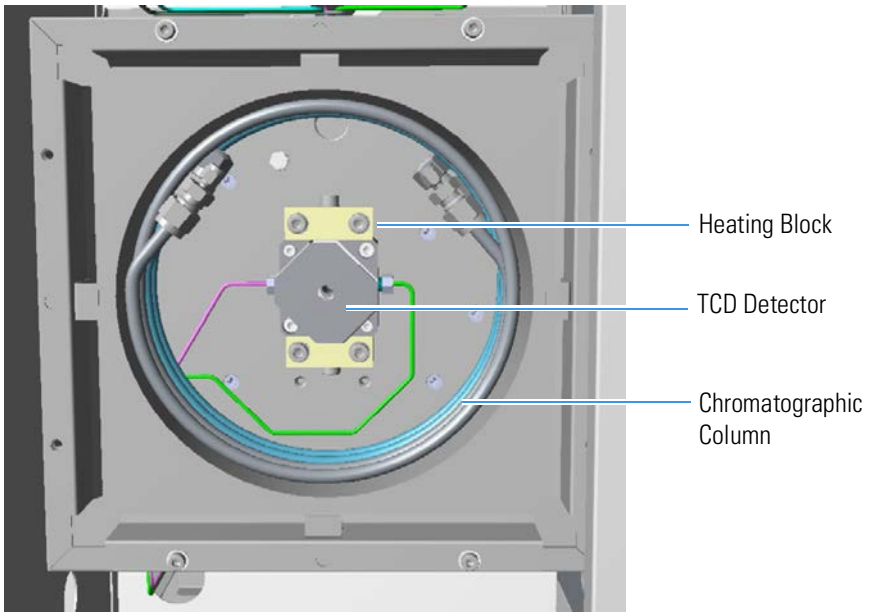



**Figure 14-5.** Compartment Housing the Chromatographic Column

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

## Replacing the O-Rings of the Reactors Coupling Unions

**⚠CAUTION** Before you start the operation, make sure that the furnaces are at room temperature.

❖ **To replace the o-rings of the reactors coupling unions**

| Materials Required |
|---|
| Allen wrench |
| Screwdriver |
| Spare seals |

1. Open the furnaces compartment by lifting the cover and removing the protection wall. Also see "Furnace Compartment" on page 3-13.

2. Remove the reactors from the furnaces according to the instructions given in "Removing the Quartz Reactor from the Furnace" on page 9-13 or "Removing the Alloy Steel (HPAR) Reactors from the Furnaces" on page 9-14.

3. Loosen the hex screws securing the reactors coupling unions to the base as shown in Figure 14-6.

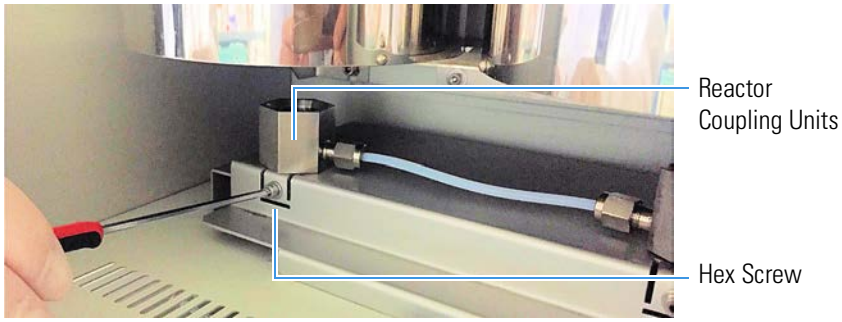



**Figure 14-6.** Removal of the Reactors Coupling Unions

4. Remove the reactors coupling unions from the base and rest them on a clean surface, as shown in Figure 14-7.

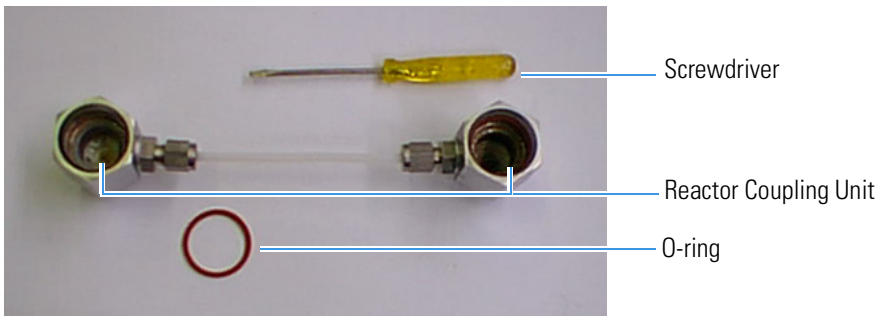



**Figure 14-7.** View of the Reactors Coupling Unions

5. Using the small screwdriver, remove the O-ring from each union, as shown in Figure 14-8.

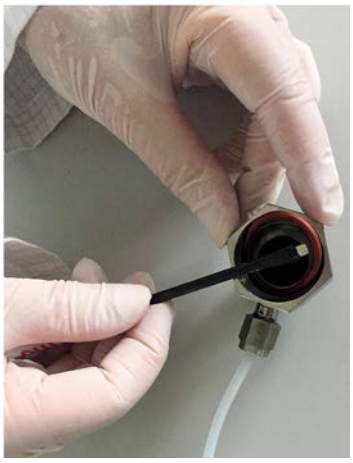


**Figure 14-8.** Removal of the O-ring from the Coupling Union

6. Put a new O-ring into each union. Making sure by using an appropriate tool that it fits correctly in its seat, as shown in Figure 14-9.

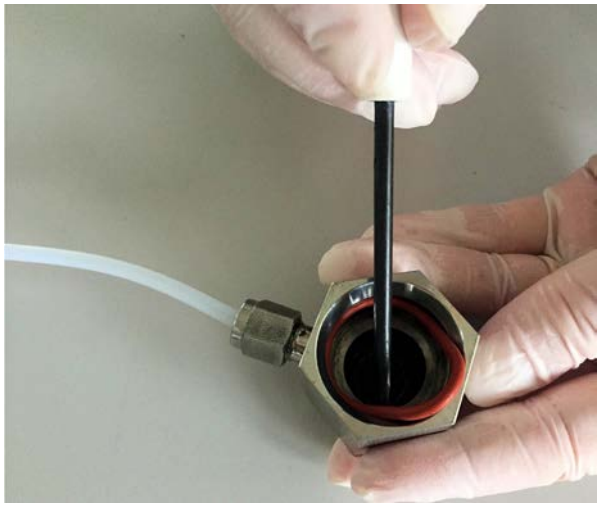


**Figure 14-9.** Introduction of the O-ring from the Coupling Union

# Maintaining the MAS Plus Autosampler

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

❖ **To clean the shaft of the MAS Plus autosampler**

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

1. In the Main Menu, select the menu **Edit | Edit Elemental Analyzer parameters**, or click the  icon. The **Analyzer Parameter** window is shown.
2. Select the **Flow/Timing** tab. In the **Gas Flow** box, set **Carrier** Off.
3. Click **Send** and wait 2-3 minutes to discharge the gas out the circuit.
4. Remove the frontal cover of the MAS Plus autosampler as shown in Figure 14-10. First move it up, then move it towards you.

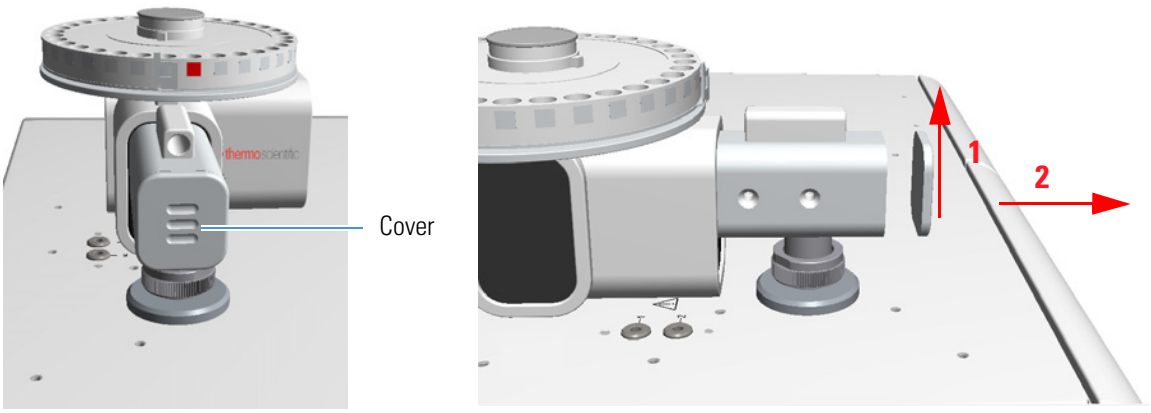



**Figure 14-10.** Removal of the MAS Plus Autosampler Cover

5. From the Main Menu, select **View | View Elemental Analyzer status**, or click the  icon.
6. In the **Status** window, select the **Special function** tag, then click **Step sampler tray position**. The autosampler mechanism pushes the shaft forward and then ejects it.
7. Take out the shaft from the autosampler, as shown in Figure 14-11.

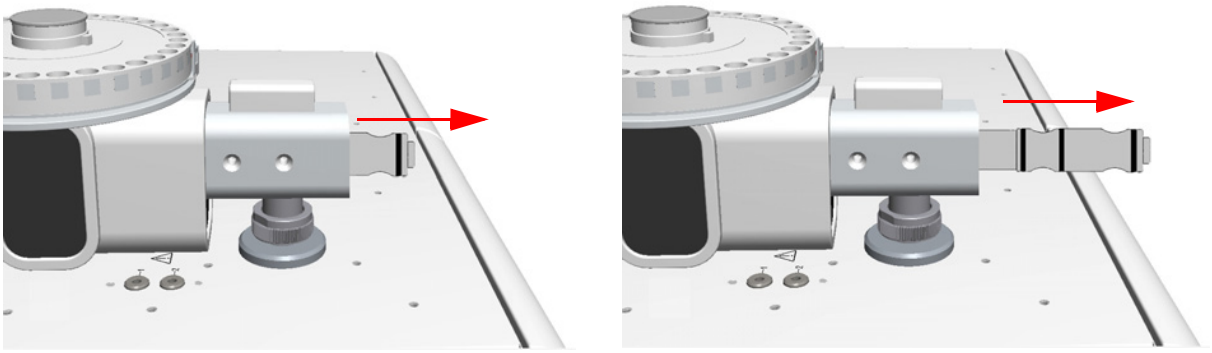


**Figure 14-11.** Removal of the Shaft

8. Remove possible traces of dirt from the shaft seals with a dry clean cloth.

9. Smear a slight layer of silicon grease on the o-ring. Do not use solvents. Figure 14-12 shows the shaft of the MAS Plus autosampler.

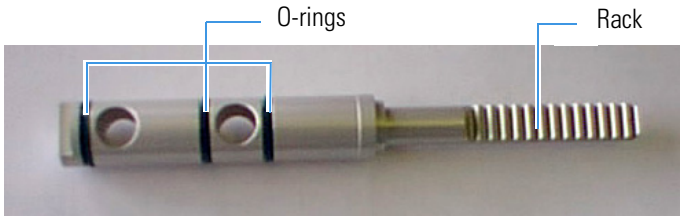



**Figure 14-12.** Shaft of the Autosampler

10. Re-introduce the shaft into the autosampler until it is in place keeping its rack turned downward, as shown in Figure 14-13.

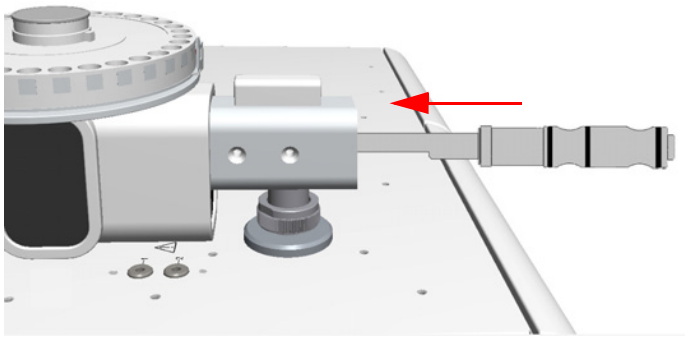


**Figure 14-13.** Reinstalling the Shaft (1)

11. While slightly pushing the shaft with one finger of your hand, as shown in Figure 14-14, 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).

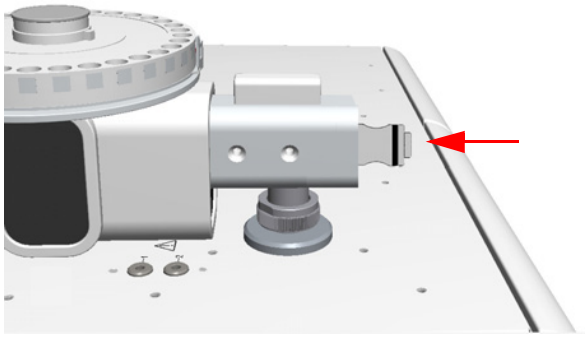


**Figure 14-14.** Reinstalling the Shaft (2)

12. Re-install the autosampler front cover.
13. Return to the **Analyzer Parameter** window. To restore the operating conditions, set the carrier gas flow to the initial value.

❖ **To replace the O-rings of the MAS shaft**

| **Materials required** |
|---|
| Tweezers or spatula |
| Green O-rings package (set of 3) for MAS Plus shaft (P/N 29030343) |
| MAS O-ring Removal Tool (P/N 20502613) |

The shaft of the MAS plus has three conical O-rings (lip seal) that must be replaced when worn-out. The MAS O-ring removal tool allows an easier replacement of the internal and middle O-rings into their respective seats.

1. Remove the three O-rings from the shaft body with tweezers or a spatula. Do not scratch the shaft.
2. Take the new green O-rings package and the removal tool.
3. Insert the first O-ring into the internal seat of the shaft.
    a. Place one of the three O-ring, with the lip turned inwards, on the conical section of the tool.
    b. Move the O-ring along the tool up to reach the border.
    c. Move the tool on the shaft body up to reach the internal seat.
    d. Move and inset the O-ring into the internal seat, then remove the tool.

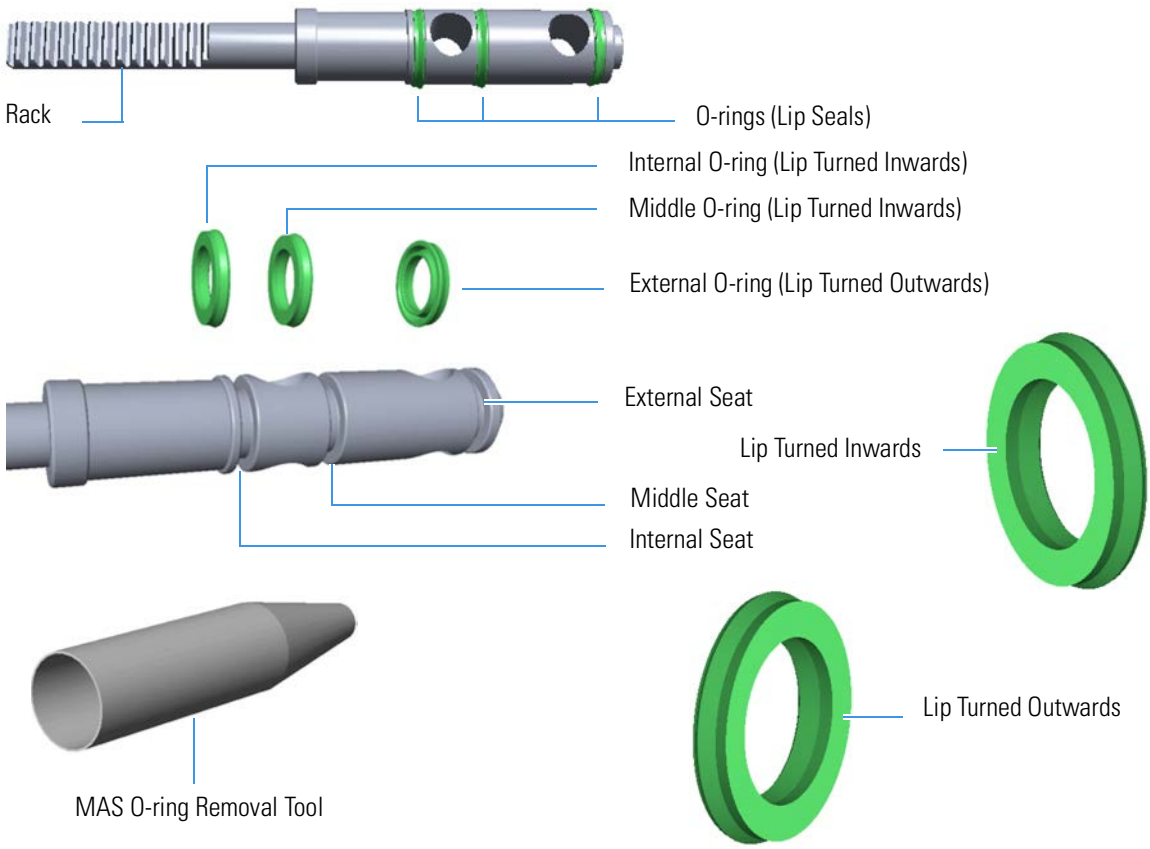



**Figure 14-15.** MAS O-ring removal tool

**Figure 14-16.** Inserting the first O-ring

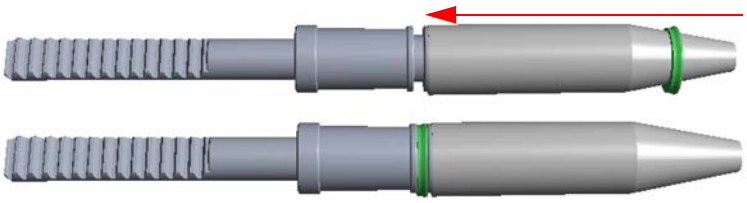


**Figure 14-17.** Moving the O-ring into the internal seat

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

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

**Figure 14-18.** Inserting the second O-ring

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

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

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

**Figure 14-19.** Moving the O-ring into the second seat

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

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

**Figure 14-20.** Inserting the third O-ring

15

# Troubleshooting

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

**Contents**

# Safety Cutoff Device

An instrument malfunctioning, due to a component failure or to abnormal operating conditions, is identified by the red lighting of the Safety Cutoff LED indicator. See Figure 15-1

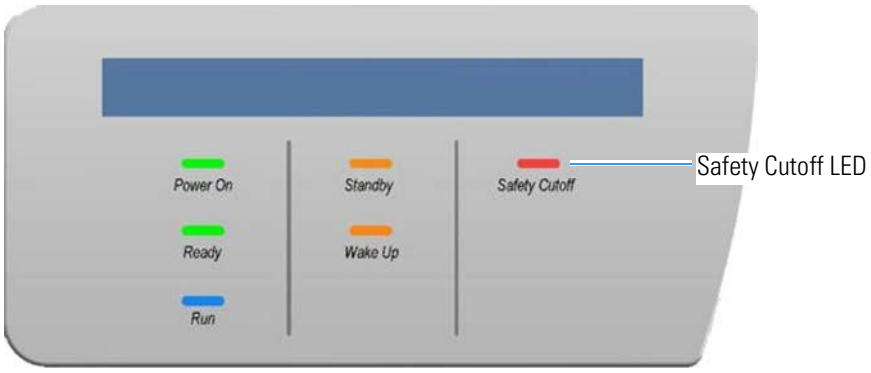



**Figure 15-1.** Safety Cutoff LED

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

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

❖ **To display the error message**

1. In the Main Menu, select **View | View Elemental Analyzer Status**, or click the  icon.

2. In the shown dialog window, select the **Special Functions** tabbed page. The dialog window shown in Figure 15-2 appears.

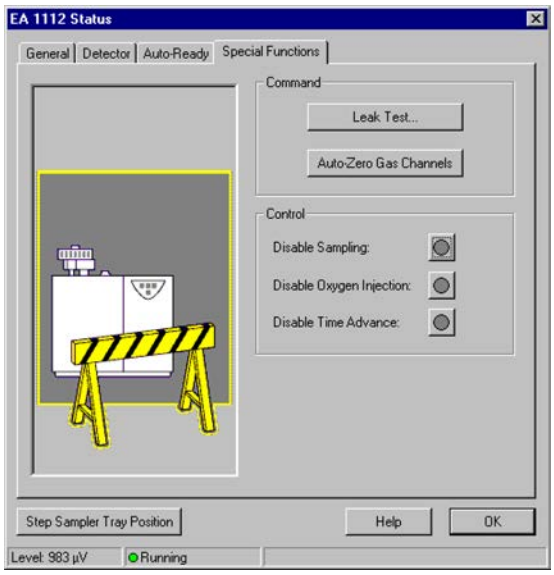



**Figure 15-2.** Special function window

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. See Table 15-1 to find out the error status and have mode information.

The following Table 15-1 reports the error messages and the explanation of the relevant correlated problem.

**Table 15-1.** Error Messages

| Message | Description | What to do |
|---|---|---|
| **Under voltage protection** (Safety Cut Off message) | Voltages supplied to the electrical circuits are too low or out of tolerance. | Check all the voltages and main power. |
| **Oven over limit** (Safety Cut Off message) | The oven temperature exceed 190 °C or does not reach the setting temperature. | Check the functionality of the PT 100 probe. Replace the HWD 1112 board. |
| **Left furnace over limit** (Safety Cut Off message) | The left thermocouple or the resistance of the furnace is damaged or interrupted. | Verify the continuity of the thermocouple and replace it. |
| **Right furnace over limit** (Safety Cut Off message) | The right thermocouple or the resistance of the furnace is damaged or interrupted. | Verify the continuity of the thermocouple and replace it. |
| **Thermal pre-protection** (Safety Cut Off message) | Anomalous temperature inside the transformers compartment. | Check the functionality of the fans. Replace the AS 1112 board. |
| **Oxygen pressure too low** (NO Safety Cut Off message) | If the oxygen pressure is too low (<150 kPa) the flow of oxygen does not reach the set point. | Verify that the pressure at the manometer is at second stage higher or equal to 350 kPa. |

**NOTICE**

The error message is generally due to the specific cause indicated. Sometimes, it may be generated by different electric factors or caused by failures not depending on the system. In this case, contact your local Thermo Fisher Scientific Customer Support.

# EFC-t Module

The failures that may be generated on the EFC-t Module are connected to the breakage, or to the malfunctioning of solenoid valves and flow sensors. See Table 15-2 to find the component responsible of the EFC-t Module malfunctioning and to solve the relevant problem.

**Table 15-2.** EFC-t module troubleshooting

| Failure | Defective Component | Remedy |
|---|---|---|
| Oxygen does not flow to the point 2 of the autosampler. | EV1 | Check voltage supply.<br>Replace the EFC-t Module. |
| | EV2 | Check voltage supply.<br>Replace the EFC-t Module. |
| The helium (argon) flow measured on point 1 or 2 cannot be adjusted. | Flow sensor 1 or 2 | Replace the EFC-t Module. |
| | EVP1 or EVP2 | Check voltage supply.<br>Replace the EFC-t Module. |
| The pneumatic circuit is perfectly close but the flow value do not decrease up to zero performing the leak test. | EV3 and/or EV4 | Check voltage supply.<br>Replace the EFC-t Module. |

# A

# Legal Documents

**Contents**

# FCC Compliance Statement

This equipment has been tested and found to comply with the limits for a Class B digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference in a residential installation. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instructions, may cause harmful interference to radio communications. However, there is no guarantee that interference will not occur in a particular installation. If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient or relocate the receiving antenna.
- Increase the separation between the equipment and receiver.
- Connect the receiver into an outlet on a circuit different from that to which the equipment is connected.
- Consult the dealer or an experienced radio/TV technician for help.

# EU REACH Statement

The European Commission promulgated legislation that covers the registration, evaluation, authorization and restriction of chemicals within the European Union community under (EC) No 1907/2006. This regulation is commonly known as REACH. Thermo Fisher Scientific is committed to meeting all compliance obligations under REACH. As per Article 33 of the Regulation, this product may include items which contain more than 0.1% by weight of some SVHC Candidate Substance. Some electronic parts and copper alloys can contain lead.

# WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2012/19/EU. It is marked with the following symbol:

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

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

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

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

# Declaration of Conformity

-Original-

## EG-Konformitätserklärung
## *EC Declaration of Conformity*

CE

ThermoFisher SCIENTIFIC

Thermo Fisher Scientific (Bremen) GmbH
Hanna-Kunath-Str. 11
28199 Bremen, Germany

**Wir erklären hiermit, dass das folgende Produkt**
*We hereby declare that the following product*

| | |
|---|---|
| **Bezeichnung:**<br>*Designation:* | **Organische Elementaranalyse**<br>*Organic Elemental Analyzer* |
| **Modell:**<br>*Model:* | **Thermo Scientific Flash Smart**<br>*Thermo Scientific Flash Smart* |

**alle einschlägigen Anforderungen der folgenden Richtlinien erfüllt:**
*fulfills all the relevant requirements of the following directives:*

| | |
|---|---|
| **Niederspannungsrichtlinie**<br>*Low Voltage Directive* | **Richtlinie über elektromagnetische Verträglichkeit**<br>*Electromagnetic Compatibility Directive* |
| **2014/35/EU** | **2014/30/EU** |

**Die folgenden einschlägigen harmonisierten Normen wurden zugrunde gelegt:**
*The following relevant harmonized standards were used:*

| | |
|---|---|
| **EN 61010-1:2010** | **EN 61326-1:2013** |
| **EN 61010-2-081:2015** | |

**Für die Zusammenstellung der technischen Unterlagen ist bevollmächtigt:**
*Person authorized to compile the technical file:*

Jörg Behrens (Director Operations)
Thermo Fisher Scientific (Bremen) GmbH

| | |
|---|---|
| **Unterschrift**<br>*Signature* | Bremen, 2017-01-24<br>**Datum**<br>*Date* |

# Declaration of Manufacturer

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.

### Regulatory Compliance

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations.

When the system is delivered to you, it meets all pertinent electromagnetic compatibility (EMC) and safety standards.

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

# 15-Years Warranty for Furnace and Thermal Conductivity Detector

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

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

### What is excluded from the Furnaces Warranty?

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

### Thermal Conductivity Detector Warranty

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

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

### What is excluded from the Thermal Conductivity Detector Warranty?

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

### Whom to contact in case of problems?

If you have problems with a Furnace or a Thermal Conductivity Detector, contact the local service or service engineer.

# Health and Safety Form

**This Decontamination Declaration Form must be completed for <u>all</u> materials returned to Thermo Fisher Scientific. It should be sent to the destination by e-mail, with approval from an authorized person. A signed hardcopy should be attached to the outside of the package with shipping paperwork and a further copy should be placed inside the packaging. The receiving Thermo Fisher Scientific office can help with this form and supply a return number, shipping address and e-mail address. This form can be used to request warranty. Use the text "not used" to indicate a field not being used. Where a Thermo Scientific part number is not known, add the supplier name (as for the examples below).**

## 1. General information

Thermo Fisher Scientific contact name for delivery _____

Thermo Fisher Scientific receiving site _____

Customer _____ Instrument type _____

Address _____ Instrument SN _____

_____ Order number _____

Phone _____ Return number _____

E-Mail _____ Medical Device ☐ Research Use Only ☐

SAP Service Notification _____

| Part Number | Quantity | Material Description | Error Description / Reason for Return | Return Part Serial No |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

## 2. Condition of the material or instrument

Has the material or instrumentation been removed from the shipping packaging or in contact with

- pump fluids
- service fluids
- samples
- standard solutions
- other chemicals
- hazardous materials

Tick the applicable check box.

☐ Yes → go to section 3

☐ No → go to section 5

## 3. Contamination

Use the check boxes to state any contaminants the material/instrumentation been exposed to. Contaminated materials must not be shipped to Thermo Fisher Scientific. If any exposure boxes are ticked, select 'Yes', if none, select 'No'.

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
|  | ☐ toxic |  | ☐ flammable |  | ☐ serious health hazard |  | ☐ corrosive |  | ☐ oxidizing |
|  | ☐ hazardous to environment |  | ☐ explosive |  | ☐ gas under pressure |  | ☐ other harmful substances | | |
|  | ☐ biological contaminated |  | ☐ radioactive contaminated | | | | | | |

☐ Yes → go to section 4

☐ No → go to section 5

ThermoFisher SCIENTIFIC

# Health and Safety Form

## 4. Description of process substances and/or compounds

Which substances have been in contact with the material or instrumentation? (trade name and/or chemical term of service fluids and substances; properties of substances or compounds according to the Material Safety Data Sheet; e.g. toxic, flammable, corrosive, radioactive)

| | Part Number | Trade Name | Chemical / Substance Name / Properties |
|---|---|---|---|
| a) | | | |
| b) | | | |
| c) | | | |
| d) | | | |
| e) | | | |
| f) | | | |

## 5. Legally binding declaration

Has the material/instrument undergone a decontamination process? ☐ Yes → go to section 6 ☐ No

Is the material/instrument safe to handle for Thermo Fisher Scientific and third-party personnel? ☐ Yes ☐ No

Components, materials and/or instruments that have been contaminated to a harmful level by whatever substances and/or compounds as stated in sections 3. and 4. above will not be accepted without written evidence of proper decontamination.
I hereby declare that the instrument has undergone successfully all required decontamination procedures and is safe to handle for Thermo Fisher Scientific and/or third-party service personnel or suppliers such as Pfeiffer Vacuum, Leybold Vacuum, Edwards Vacuum products, or others.
I confirm that all information, which is supplied on this form, is accurate, complete and sufficient to judge any contamination level. I acknowledge and agree that I will be liable for any personal injury or any other damage, which might result from a false, inaccurate or incomplete statement and that I will indemnify and defend Thermo Fisher Scientific and/or any other concerned third party for and against any liabilities, claims, losses, and/or damages of all kinds arising out of and/or caused by such false, inaccurate or incomplete statements.
Thermo Fisher Scientific reserves the right not to process refunds or returns where the declared or observed use or previous contamination of the product/material has by Thermo Fisher Scientific judgement impacted its integrity.

## 6. Detailed description of the decontamination process used

| Part Number | Serial Number | Describe the decontamination process |
|---|---|---|
| | | |
| | | |

| Return Number | Name of authorized person (block letters) | Date | Signature | Company stamp |
|---|---|---|---|---|
| | | | | |

# Glossary

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

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

## A

**A** ampere

**ac** alternating current

**ADC** analog-to-digital converter

**AP** acquisition processor

**ASCII** American Standard Code for Information Interchange

## B

**b** bit

**B** byte (8 b)

**baud rate** data transmission speed in events per second

**BBOT** 2,5-Bis 5-Tert-Butyl-Benzoxazol-2-Yl Thiophene

**BEST** Brightly Enhanced Sample Transfer

**BF** backflush

## C

**°C** degrees Celsius

**CE** European conformity. Mandatory European marking for certain product groups to indicate conformity with essential health and safety requirements set out in European Directives.

**cfm** cubic feet per minute

**CI** chemical ionization

**CID** collision-induced dissociation

**cm** centimeter

**cm$^3$** cubic centimeter

**Continuous-Flow (CF)** Automated preparation device and mass spectrometer in which sample analysis is conducted in a continuous stream of helium carrier gas.

**CPLD** Complex Programmable Logic Device

**CPU** central processing unit (of a computer)

**CSE** Customer Service Engineer

**CSIA** Compound Specific Isotope Analysis

**<Ctrl>** control key on the terminal keyboard

## D

**d** depth

**Da** dalton

**DAC** digital-to-analog converter

**dc** direct current

**driver** A device-specific control program that enables a computer to work with a particular device.

**DS** data system

**DSP** digital signal processor

**DSQ™** Dual Stage Quadrupole

**Dual Inlet (DI)** Inlet method in which a pure gas sample is admitted into an isotope ratio mass spectrometer (IRMS) by a variable volume bellows. A reference gas is admitted into the IRMS via a second variable volume bellows. The bellows are balanced to provide sample and reference signal responses of equal intensity.

## E

**EA** Elemental Analyzer

**EA-IRMS** Elemental Analyzer Isotope Ratio Mass Spectrometry

**EI** electron ionization

**Elemental Analyzer (EA)** Automated sample preparation instrument in which samples are automatically converted into pure gases for isotope ratio analysis. An elemental analyzer contains the following elements: (i) furnace for combustion, reduction or pyrolysis of sample material; (ii) chemical traps for analyte gas purification; (iii) gas chromatography for time separation of these analyte gases.

**EMC** Electromagnetic Compatibility

**<Enter>** Enter key on the terminal keyboard

**ESD** electrostatic discharge

**ESI** electrospray ionization

**eV** electron volt

## F

**f** femto ($10^{-15)}$

**°F** degrees Fahrenheit

**FID** Flame Ionization Detector

**FM** flow meter

**forepump** The pump that evacuates the foreline. A rotary-vane pump is a type of forepump.

**FSE** Field Service Engineer

**ft** foot

**FTP** file transfer protocol

**FWHM** Full Width at Half Maximum

## G

**g** gram

**G** Gauss; giga ($10^9$)

**GC** gas chromatograph; gas chromatography

**GC/MS** gas chromatograph/mass spectrometer

**GFCI** Ground Fault Circuit Interrupter: this term is mainly used in North America. As a synonym, the term Residual Current Device (RCD) is used in Europe.

**GISP** Greenland Ice Sheet Precipitation. International reference standard for hydrogen and oxygen isotopes.

See also SLAP and VSMOW.

**GLT** Glass Lined Tubing

**GUI** graphical user interface

## H

**h** hour

***h*** height

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

**HF** high flow

**HFCS** High Fructose Corn Syrup

**HOT OC** High Oven Temperature Cold On-Column

**HPAR** High Performance Alloy Reactor

**HPLC** High Performance Liquid Chromatography. Standalone liquid chromatography system (or inlet for mass spectrometry detector).

**HTC** High Temperature Conversion

**HV** high voltage

**Hz** hertz (cycles per second)

## I

**IAEA** International Atomic Energy Agency

**ICIS™** Interactive Chemical Information System

**ICL™** Instrument Control Language™

**ICP** inductively coupled plasma

**ICP-OES** inductively coupled plasma optical emission spectroscopy

**ID** inside diameter

**IEC** International Electrotechnical Commission

**IEEE** Institute of Electrical and Electronics Engineers

**in.** inch

**I/O** input/output

**ion optics** Focuses and transmits ions from the ion source to the mass analyzer.

**ion source** A device that converts samples to gas-phase ions.

**irm** isotope ratio monitoring

**IRMS** Isotope Ratio Mass Spectrometer

## K

**k** kilo ($10^3$, 1000)

**K** kilo ($2^{10}$, 1024)

**kg** kilogram

## L

***l*** length

**L** liter

**LAN** local area network

**lb** pound

**LC** Liquid chromatography. A process that separates a chemical mixture carried by liquid into components as a result of differential distribution of the solutes as they flow around or over a stationary or solid phase.

**LC/MS** liquid chromatograph / mass spectrometer

**LED** light-emitting diode

**LF** low flow

**LN2** liquid nitrogen

**log file** A text file, with a .log file extension, that is used to store lists of information.

**LVSL** Large Volume Splitless Injector

**m** micro ($10^{-6}$)

## M

**m** meter

**m** milli ($10^{-3}$)

**M** mega ($10^6$)

**$M^+$** molecular ion

**MB** Megabyte (1048576 bytes)

**MCD** Microchannel Device

**$MH^+$** protonated molecular ion

**min** minute

**mL** milliliter

**mm** millimeter

**MP** measuring point

**MS** mass spectrometer; mass spectrometry

**MS** $MS^n$ power: where n = 1

**MS/MS** $MS^n$ power: where n = 2

**$MS^n$** $MS^n$ power: where n = 1 through 10

**MSDS** Material Safety Data Sheet

**MTBE** methyl tert-butyl ether

**MVFC** multifunctional valve cluster

***m/z*** Mass-to-charge ratio. An abbreviation used to denote the quantity formed by dividing the mass of an ion (in u) by the number of charges carried by the ion.
For example, for the ion $C_7H_7^{2+}$, $m/z$=45.5.

## N

**n** nano ($10^{-9}$)

**Natural Abundance** The concentration of isotopes as found in nature.

**NCBI** National Center for Biotechnology Information (USA)

**NIST** National Institute of Standards and Technology (USA)

**noise** Any random disturbance that obscures the clarity of a signal

**NPD** nitrogen/phosphorous detector

## O

**OC** On-Column

**OD** outside diameter

**OS** open split

**W** ohm

**outlier** A calibration data point that does not appear to correlate to other calibration data points within experimental error.

## P

**p** pico ($10^{-12}$)

**Pa** pascal

**PCB** printed circuit board

**PDD** Pulsed Discharge Detector

**PE** protective earth

**PEEK** polyether ether ketone

**PID** proportional/integral/differential

**P/N** part number

**P/P** peak-to-peak voltage

**PPE** personal protective equipment

**ppm** parts per million

**psig** pounds per square inch, gauge

**PU** polyurethane

**PTV** Programmable Temperature Vaporizing

## R

**RAM** random access memory

**RCD** Residual Current Device: this term is mainly used in Europe. As a synonym, the term Ground Fault Circuit Interrupter (GFCI) is used in North America.

**relative standard deviation** A measure of the dispersion of a group of measurements relative to the mean of the group. Relative standard deviation is expressed as a percentage of the average value. The percent relative standard deviation is calculated as:

$$\%\,\mathrm{RSD} = 100 \cdot \frac{\mathrm{SD}}{\bar{x}}$$

where SD is the standard deviation and $\bar{X}$ is the sample mean.

**RF** radio frequency

**RMS** root mean square

**RoHS** Restriction of Hazardous Substances. EU directive on the restriction of the use of certain hazardous substances in electrical and electronic equipment.

**ROM** read-only memory

**rotary-vane pump** A mechanical vacuum pump that creates the vacuum necessary for the proper operation of the turbomolecular pump. Also called roughing pump or forepump.

**RS-232** An accepted industry standard for serial communication connections. This Recommended Standard (RS) defines the specific lines and signal characteristics used by serial communications controllers to standardize the transmission of serial data between devices.

## S

**s** second

**serial port** An input/output location (channel) for serial data transmission.

**SIM** selected ion monitoring

**SLAP** Standard Light Antarctic Precipitation; international reference standard for hydrogen and oxygen isotopes.

See also VSMOW.

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

**SPME** Solid Phase Micro Extraction

**SRM** selected reaction monitoring

**SS** stainless steel

**SSL** Split/Splitless

**standard deviation** In statistics, the standard deviation SD is a measure of the dispersion of a group of measurements. For example, masses, times, or intensities. Standard deviation is calculated as follows:

$$\sigma = SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

See also relative standard deviation.

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

## T

**TCD** Thermal Conductivity Detector

**TCP/IP** transmission control protocol / Internet protocol

**TIC** total ion current

**Torr** A unit of pressure, equal to 1 mm of mercury and 133.32 Pa.

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

**turbomolecular pump** A vacuum pump that provides a high vacuum for the mass spectrometer and detector system.

## U

**u** atomic mass unit

**UHV** ultra high vacuum

## V

**V** volt

**V AC** volts alternating current

**V DC** volts direct current

**VFC** voltage-frequency converter

**vol** volume

**VCDT** Vienna Canyon Diablo Troilite; international reference standard for sulfur isotopes.

**VPDB** Vienna Pee Dee Belemnite; international reference standard for carbon and oxygen isotopes.

**VSMOW** Vienna Standard Mean Ocean Water; international reference standard for hydrogen and oxygen isotopes.

See also SLAP.

## W

**w** width

**W** watt

**WEEE** European Union Waste Electrical and Electronic Equipment Directive. Provides guidelines for disposal of electronic waste.

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

# Index

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