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TriPlus 500

User Guide

Headspace Sampler

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ThermoFisher SCIENTIFIC

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General Lab Equipment. Not for Clinical, Patient, or Diagnostic Use.

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Reader's Survey

TriPlus 500 Headspace Sampler User Guide, PN 31716106, 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

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- changes and repairs

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- Machinery Directive: 2006/42/EC
- EMC Directive: 2014/30/EU
- RoHS Directive: 2011/65/EU and (EU) 2015/863

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- Machinery Directive: 2006/42/EC
- EMC Directive: 2014/30/EU
- RoHS Directive: 2011/65/EU and (EU) 2015/863

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EMC

– EN 61326-1:2013, IEC 61326-1:2012
– FCC rules: CFR no. 47 Part 15 Subpart B Section 15.107 and 15.109

Safety

– EN 61010-1:2010, IEC 61010-1:2010
– EN 61010-2-010: 2014, IEC 61010-2-010: 2014 (TriPlus 500 HS only)
– EN 61010-2-081: 2015, IEC 61010-2-081: 2015
– UL 61010-1:2012/R:2016-04
– UL 61010-2-010:2015 (TriPlus 500 HS only)
– UL 61010-2-081:2015
– CAN/CSA C22.2 No. 61010-1:2012/U2:2016-04
– CAN/CSA C22.2 No. 61010-2-010:2015 (TriPlus 500 HS only)
– CAN/CSA C22.2 No. 61010-2-081:2015

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We hereby declare that the following products

Designation:	**Autosampler**
Model:	**Thermo Scientific TriPlus 500 HS**

fulfill all the relevant requirements of the following directives:

Low Voltage Directive	**2014/35/EU**
Electromagnetic Compatibility Directive	**2014/30/EU**
RoHS Directive	**2011/65/EU and (EU) 2015/863**

The following relevant harmonized standards were used:

EN 61010-1:2020-03	**EN 61326-1:2013-07**

Person authorized to compile the technical file:

Giacinto Zilioli
(Director, Strategic Projects)
Thermo Fisher Scientific S.p.A.

Signature

Milan, March 24, 2023

Date

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-Original-

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UK CA

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Italy

Declares, under sole responsibility, that products

Designation:	**Autosampler**
Model:	**Thermo Scientific TriPlus 500 HS**

as originally delivered complies with the essential requirements of the following applicable UK Regulations:

Electrical Equipment (Safety) Regulations	**2016**
Electromagnetic Compatibility Regulations	**2016**
The Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment (ROHS) Regulations	**2012**

and complies with the following harmonized standards and other technical specifications:

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(Director, Strategic Projects)
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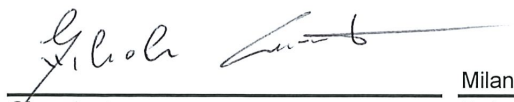

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- WEEE product class
- Name of the manufacturer or distributor (where you purchased the product)
- Number of product pieces, and the estimated total weight and volume
- Pick-up address and contact person (include contact information)
- Appropriate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

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

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

WEEE Directive
2012/19/EU

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Directive DEEE
2012/19/EU

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WEEE Direktive
2012/19/EU

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Preface

This guide contains detailed information for the use of the Thermo Scientific™ TriPlus™ 500 Headspace Sampler (TriPlus 500 HS). This documentation is intended for frequent or new TriPlus 500 HS users who are experienced at using automated systems to run existing analytical methods.

Note The TriPlus 500 HS must be installed and set up properly before this document can be used.

This guide is organized as follows:

- Chapter 1, "Getting Started with Your TriPlus 500 Headspace Sampler," provides information to familiarize you with your TriPlus 500 HS.
- Chapter 2, "Operating Principles," describes the operating principles of the TriPlus 500 HS.
- Chapter 3, "Setting Up Through the TRACE 1610 User Interface," contains the instructions to configure your TriPlus 500 HS and to edit the parameters through the TRACE 1310 GC user interface (touch screen) or through the TRACE 1310 Virtual Touch Screen software.
- Chapter 4, "Setting Up Through Chromeleon CDS," contains the instructions to configure your TriPlus 500 HS and to edit the parameters through the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS).
- Chapter 5, "Setting Up Through TraceFinder CDS," contains the instructions to configure your TriPlus 500 HS and to edit the parameters through the Thermo Scientific™ TraceFinder™ Chromatography Data System (CDS).
- Chapter 6, "Method Development," provides information for developing a method with your TriPlus 500 HS.
- Chapter 7, "Using the TriPlus 500 Web Interface," provides the instructions for using the TriPlus 500 Web Interface.
- Chapter 8, "Ordering Parts," contains part numbers for all the consumables and parts available for your TriPlus 500 HS.
- "Glossary," contains definitions of terms used in this guide. It also includes abbreviations, acronyms, metric prefixes, and symbols.

Contents

- About Your System
- Power Rating
- Contacting Us
- Related Documentation
- Safety Information and Warnings
- Instrument Markings and Symbols
- Safety Information and Warnings

About Your System

Thermo Fisher Scientific systems operate safely and reliably under carefully controlled environmental conditions.

If the equipment is used in a manner not specified by the manufacturer, the protections provided by the equipment might be impaired. If you maintain a system outside the specifications listed in this guide, failures of many types, including personal injury or death, might occur.

The repair of instrument failures caused by operation in a manner not specified by the manufacturer is specifically excluded from the Standard Warranty and service contract coverage.

WARNING 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.

AVERTISSEMENT Les systèmes Thermo Fisher Scientific fonctionnent de manière sûre et fiable dans des conditions ambiantes minutieusement régulées. La protection fournie par l'équipement peut être entravée si ce dernier est utilisé d'une manière non spécifiée par le fabricant. Si vous maintenez un système en dehors des spécifications listées dans ce guide, des défaillances de types divers sont possibles, pouvant notamment entraîner des blessures, voire la mort. La réparation des défaillances d'instruments liées à une utilisation non conforme aux spécifications du fabricant est expressément exclue de la garantie standard et de la couverture prévue par un contrat de maintenance.

Power Rating

TriPlus 500 HS alone:

- 100-240 Vac; 600 W; 50/60 Hz

Vial Loader

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

  Input 100-240 Vac; 50/60 Hz; 1.3 A — Output 24 Vdc; Power 90 W; 3.75 A

WARNING You must only use the portable external power supply provided with the instrument by Thermo Fisher Scientific.

ADVERTISSEMENT Vous ne devez utiliser que l'alimentation externe portable fournie avec l'instrument par Thermo Fisher Scientific.

Detailed instrument specifications are in the Product Specifications Sheet.

Contacting Us

There are several ways to contact Thermo Fisher Scientific for the information you need.

❖ **To find out more about our products**

Go to http://www.thermofisher.com for information about our products.

❖ **To get local contact information for sales or service**

Go to http://www.unitylabservice.com/en/home.html

Related Documentation

In addition to this guide, Thermo Scientific™ provides the following documents for TriPlus 500 HS.

- *TriPlus 500 Headspace Sampler Preinstallation Requirements Guide*, P/N 31716105
- *TriPlus 500 Headspace Sampler Hardware Manual*, P/N 31716107
- *TriPlus 500 Headspace Sampler with Transfer Line Hardware Manual*, P/N MI-317000-0013
- *TriPlus 500 Headspace Sampler Spare Parts Catalog*, P/N 31716108

To suggest ways we can improve the documentation, follow this link to our Reader's Survey.

Safety Alerts and Important Information

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

Special Notices

Notices includes the following:

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

Note Emphasizes important information about a task.

Tip Helpful information that can make a task easier.

Safety Symbols and Signal Words

All safety symbols are followed by **WARNING** or **CAUTION**, which indicates the degree of risk for personal injury, instrument damage, or both. Cautions and warnings are following by a descriptor, such as **BURN HAZARD**. A **WARNING** is intended to prevent improper actions that could cause personal injury. Whereas, a **CAUTION** is intended to prevent improper actions that might cause personal injury, instrument damage, or both. You can find the following safety symbols on your instrument, or in this guide:

Symbol	Descriptor
	**BIOHAZARD:** Indicates that a biohazard *will*, *could*, or *might* occur.
	**BURN HAZARD:** Alerts you to the presence of a hot surface that *could* or *might* cause burn injuries.
	**ELECTRICAL SHOCK HAZARD:** Indicates that an electrical shock *could* or *might* occur.
	**FIRE HAZARD:** Indicates a risk of fire or flammability *could* or *might* occur.
	**EXPLOSION HAZARD.** Indicates an explosion hazard. This symbol indicates this risk *could* or *might* cause physical injury.
	**FLAMMABLE GAS HAZARD.** Alerts you to gases that are compressed, liquefied or dissolved under pressure and can ignite on contact with an ignition source. This symbol indicates this risk *could* or *might* cause physical injury.

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

Tous les symboles de sécurité sont suivis des mots **AVERTISSEMENT** ou **ATTENTION**, qui indiquent le degré de risque de blessures personnelles, de dommages à l'instrument, ou des deux. Les mentions « Attention » et les avertissements sont suivis d'un descripteur. Un **AVERTISSEMENT** vise à empêcher des actions inappropriées pouvant entraîner des blessures personnelles. Une mention **ATTENTION** vise à empêcher des actions inappropriées pouvant entraîner des blessures personnelles ou des dommages à l'instrument. Vous pouvez trouver les symboles de sécurité suivants sur votre instrument ou dans ce guide.

Symbol	Descriptor
	**RISQUE BIOLOGIQUE :** indique qu'un risque biologique va, peut ou pourrait survenir.
	**RISQUE DE BRÛLURE :** vous avertit de la présence d'une surface chaude qui peut ou pourrait entraîner des blessures par brûlure.
	**RISQUE D'ÉLECTROCUTION :** indique qu'un choc électrique peut ou pourrait survenir.
	**RISQUE D'INCENDIE :** indique qu'un risque d'incendie ou d'inflammabilité peut ou pourrait survenir.
	**RISQUE DE GAZ INFLAMMABLE :** vous avertit que des gaz sont comprimés, liquéfiés ou dissous sous pression et qu'ils peuvent s'enflammer au contact d'une source d'inflammation. Ce symbole indique que ce risque peut ou pourrait entraîner une blessure physique.
	**GANTS REQUIS :** indique que vous devez porter des gants pour effectuer une tâche, sans quoi une blessure physique peut ou pourrait survenir
	**RISQUE PHYSIQUE ET CHIMIQUE :** indique que des dommages chimiques ou une blessure physique peuvent ou pourraient survenir.
	**DOMMAGES À L'INSTRUMENT :** indique que l'instrument ou le composant pourrait subir des dommages. Ces dommages pourraient ne pas être couverts pas la garantie standard.
	**RISQUE SOULÈVEMENT :** indique qu'une blessure physique peut ou pourrait survenir si un objet n'est pas soulevé par deux personnes ou plus.
	**RISQUE MATÉRIEL ET YEUX :** indique que des dommages aux yeux peuvent ou pourraient survenir.
	**CONSULTER LE MANUEL :** vous avertit de lire attentivement la documentation de votre instrument afin de garantir votre sécurité et la capacité opérationnelle de l'instrument. Ne pas lire attentivement la documentation peut ou pourrait vous exposer à un risque de blessure physique.
	**RISQUE DE SUBSTANCES TOXIQUES :** indique que l'exposition à une substance toxique peut survenir et que l'exposition peut ou pourrait entraîner des blessures personnelles ou la mort.

	**RISQUE RADIOACTIF :** indique qu'une exposition à des matériaux radioactifs peut ou pourrait survenir.
	Pour prévenir les blessures personnelles, ce symbole général d'avertissement précède le mot **AVERTISSEMENT**et est conforme à la norme ISO 3864-2. Dans le vocabulaire des signes ANSI Z535, ce symbole indique un risque de blessures personnelles si l'instrument est utilisé de manière inappropriée ou en cas d'actions dangereuses. Ce symbole et un autre symbole de sécurité approprié vous avertissent d'un risque imminent ou potentiel pouvant entraîner des blessures personnelles.

Instrument Markings and Symbols

Table 1 explains the symbols used on Thermo Fisher Scientific instruments. Only a few of them are used on TriPlus 500 HS, which are annotated with an asterisk below.

Table 1. Instrument Marking and Symbols (Sheet 1 of 2)

	Symbol	Description
		Direct Current
*		Alternating Current
		Both direct and alternating current
		Three-phase alternating current
		Earth (ground) terminal
		Protective conductor terminal
		Frame or chassis terminal
		Equipotentiality
*		On (Supply)
*		Off (Supply)
		Equipment protected throughout by DOUBLE INSULATION or REINFORCED INSULATION (Equivalent to Class II of IEC 536)
		Fuse

Table 1. Instrument Marking and Symbols (Sheet 2 of 2)

	Symbol	Description
*		Instruction manual symbol affixed to product. Indicates that the you must refer to the manual for specific **WARNING** or **CAUTION** information to avoid personal injury or damage to the product.
		Caution, risk of electric shock
*		Caution, hot surface
*		Caution, biohazard
*		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.

Safety Information and Warnings

This safety guide raises awareness of potential safety issues and general points for consideration for Thermo Fisher Scientific representatives during installation, and repair of TriPlus 500 HS, or parts of it (following the life cycle principle), as well as for the end user of TriPlus 500 HS in the lab during the learning phase, and in routine work.

IMPORTANT Read this section first before operating with TriPlus 500 HS.

General Considerations

- Before a unit is put to use, consult the TriPlus 500 HS manuals and related documents under all circumstances.
- Changes or modifications to this unit not expressly approved by the party responsible for compliance, could void your authority to operate the equipment.
- Be aware that if the equipment is used in a manner not specified by the manufacturer, the protective and safety features of the equipment might be impaired.
- The repair of instrument failures caused by operation in a manner not specified by the manufacturer is expressly excluded from the standard warranty and service contract coverage.
- When for technical reasons it is necessary to work on instrument parts which might involve a potential hazard (heated/moving parts, components under voltage, and so on) contact the Thermo Fisher Scientific authorized representative.

Routine maintenance operations can be performed by a Thermo Fisher Scientific representative. Alternatively they can be performed by a trained operator. Routine maintenance can be performed according to the instructions reported in the *TriPlus 500 Headspace Sampler Hardware Manual*.

Electrical Hazards

Every analytical instrument has specific hazards. Be sure to read and comply with the following precautions. They ensure the safety and long-term use of your TriPlus 500 HS.

The installation over-voltage category is Level II. The Level II category pertains to equipment receiving its electrical power from the local level, such as an electrical wall outlet.

The power line and the connections between TriPlus 500 HS and other instruments, used in the configuration setup of the total analytical system, must maintain good electrical grounding. Poor grounding represents a danger for the operator, and might seriously affect the performance of the instrument.

Do not connect TriPlus 500 HS to power lines that supply devices of a heavy duty nature, such as motors, refrigerators and other devices that can generate electrical disturbances.

Use only fuses of the type and current rating specified. Do not use repaired fuses, and do not short-circuit the fuse holder. The supplied power cord must be inserted into a power outlet with a protective earth (ground) contact. When using an extension cord, make sure that the cord also has an earth contact.

If the supplied power cord does not fit the local electrical socket and a replacement or adapter has to be purchased locally, make sure that only a certified power cord is used. Any power cord used must be certified by the appropriate local authorities.

Do not to leave any cable connecting TriPlus 500 HS and the chromatographic system, or the power cord close to heated zone, such as the injector or detector heating blocks, or the GC hot air vents.

Always replace any cable showing signs of damage with another one provided by the manufacturer. Safety regulations must be respected.

Do not change the external or internal grounding connections. Tampering with or disconnecting these connections could endanger you and damage the TriPlus 500 HS.

The instrument is properly grounded in accordance with these regulations when shipped. To ensure safe operation, do not make any changes to the electrical connections or the instrument's chassis.

Do not turn the instrument on if you suspect that it has incurred any type of electrical damage. Instead, disconnect the power cord and contact a Thermo Fisher Scientific representative for a product evaluation. Do not attempt to use the instrument until it has been evaluated. Electrical damage might have occurred if TriPlus 500 HS shows visible signs of damage, exposure to any liquids or has been transported under severe stress.

Damage can also result if the instrument is stored for prolonged periods under unfavorable conditions: for example, subjected to heat, moisture, and so on. Ensure that the power supply/controller unit is always placed in a clean and dry position. Avoid any liquid spills in the vicinity.

Before attempting any type of maintenance work, always disconnect the power cords from the power supply if optional devices are installed. Capacitors inside the instrument might still be charged also if the instrument is turned off.
To avoid damaging electrical parts, do not disconnect an electrical assembly while power is applied to TriPlus 500 HS. After the power is turned off, wait approximately 30 seconds before you disconnect an assembly.

The instrument includes a number of integrated circuits. These circuits might be damaged if exposed to excessive line voltage fluctuations, power surges or electrostatic charges, or both.

The power supply for TriPlus 500 HS has the symbols **I/O** on the label for the power switch to indicate On/Off. It is important that the power On/Off switch is accessible to unplug the AC power cord from the power supply/wall outlet in case of emergency.

Other Hazards

Danger of crushing to fingers and hands. To avoid injury keep your hands away from moving parts during operation. Turn off the power to TriPlus 500 HS if you must reach inside a mechanically powered system with moving parts.

To avoid injury, observe safe laboratory practice when handling solvents, changing tubing, or operating the TriPlus 500 HS. Know the physical and chemical properties of the solvents you use. See the Safety Data Sheets (SDS) from the manufacturer of the solvents being used.

When using TriPlus 500 HS, follow the generally accepted procedures for quality control and method development.

Do not operate on the instrument components that form part of the work area of TriPlus 500 HS when it is in motion.

Do not use vials without a sealing cap. Vapor phase from organic solvents can be hazardous and flammable. Acidic vapor phase can cause corrosion to critical mechanical parts.

Use high quality vials and closures as depending on the application conditions, high pressure can build up in the vial. Do not reuse headspace vials. Repeated heating of reused vials may increase the chance of vial breaking.

Hazardous Substances Precautions

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

AVERTISSEMENT Avant d'utiliser des substances dangereuses (toxiques, nocives, etc.), veuillez lire attentivement les indications et informations relatives au risque reprises sur la fiche de données de sécurité adéquate. Utilisez un équipement de protection individuelle conformément aux exigences de sécurité.

Biological Hazard Warning Note

In laboratories where samples with potential biological hazards are handled, the user must label any equipment or parts which might become contaminated with biohazardous material.

The appropriate warning labels are included with the shipment of the instrument. It is the user's responsibility to label the relevant parts of the equipment.

When working with biohazardous materials, you are responsible for fulfilling the following mandatory requirements:

- Providing instructions on how to safely handle biohazardous material.
- Training operators must to be aware of potential hazards.
- Providing personal protective equipment.
- Providing instructions for what to do if operators are exposed to aerosols or vapors during normal operation (within the intended use of the equipment) or in case of single fault situations such as a broken vial. The protective measures must consider potential contact with the skin, mouth, nose (respiratory organs), and eyes.
- Providing instructions for decontamination and safe disposal of relevant parts.

WARNING The user or operator is responsible for the safe handling of hazardous chemicals or biological compounds including (but not limited to) bacterial or viral samples and the associated waste, according to international and local regulations.

AVERTISSEMENT L'utilisateur ou l'opérateur est responsable de la manipulation sûre des composés chimiques et biologiques dangereux, y compris, sans s'y limiter, les échantillons bactériens ou viraux et les déchets associés, conformément aux réglementations internationales et locales.

Venting Toxic Gases

When analyzing toxic compounds, be aware that during the normal operation of the GC some of the sample might be vented outside the instrument through the split and purge flow vents; therefore, be sure to vent the exhaust gases to a fume hood. Consult local environmental and safety regulations for instructions in exhausting fumes from your system.

Maintenance

Any external cleaning or maintenance must be performed with TriPlus 500 HS turned off and the power cord disconnected.

Avoid using solvents and spraying on electrical parts. For the removal of potentially dangerous substances (toxic, harmful, and so on) read the hazard indications and information reported in the Safety Data Sheet (SDS) supplied by the manufacturer referring to the relevant CAS (Chemical Abstract Service) number. Use proper protective gloves.

When working with hazardous materials such as radioactive, biologically hazardous material, and so on, it is important to train all operators how to respond in case of spills or contamination.

Depending on the class of hazardous material, the appropriate measures have to be taken immediately. Therefore, the chemicals or solvents needed for decontamination have to be on hand.

Any parts of the equipment which can potentially be contaminated, such as the sample vial tray, and so on, must be cleaned regularly. The waste solvent from cleaning and any hardware which requires to be disposed of has to be properly eliminated with all the necessary precautions, abiding by national and international regulations.

When preparing for decontamination, ensure that the solvent or chemical to be used will not damage or react with the surface, dye (color) of the instrument, table or other nearby objects. If in doubt, please contact your Thermo Fisher Scientific representative to verify the compatibility of the type or composition of solvents with TriPlus 500 HS.

Disposal

Do not dispose of this equipment or parts thereof unsorted in municipal waste. Follow local municipal waste regulations for proper disposal provisions to reduce the environmental impact of waste electrical and electronic equipment (WEEE). European Union customers: Call your local customer service representative responsible for TriPlus 500 HS for complimentary equipment pick-up and recycling.

IMPORTANT The customer has to ensure that TriPlus 500 HS has not been contaminated by any hazardous chemical or biological compounds including (but not limited to) bacteria or viruses.

Any part which had direct contact with the analytical sample must be identified and must undergo an appropriate decontamination procedure prior to shipping for disposal.

Potentially dangerous components are vials and trays. Any critical parts sent for disposal must be handled according to national laws for hazardous compounds.

The customer and the service engineer are fully responsible for enforcing these requirements. Thermo Fisher Scientific will hold the representative, customer responsible, or both, if these regulations are not observed.

Contacting Us

There are several ways to contact Thermo Fisher Scientific for the information you need.

- **To find out more about our products**

  Go to http://www.thermofisher.com for information about our products.

- **To get local contact information for sales or service**

  Go to http://www.unitylabservice.com/en/home.html

1

Getting Started with Your TriPlus 500 Headspace Sampler

This chapter provides information to familiarize users with the TriPlus 500 Headspace Sampler (TriPlus 500 HS or HS sampler).

Contents

- Introduction
- Instrument Basics
- Label Locations on the Instrument
- Incubation Group
- Sampling Path
- Pneumatic Connections
- Electrical Connections
- Status Panel
- TRACE 1610 GC User Interface
- Vial Loader
- Sample Trays
- Barcode Reader
- Heated/Cooled Tray
- TriPlus 500 Web Interface

Introduction

The TriPlus 500 HS is an automatic sampler for headspace gas chromatographic (HSGC) analysis for the determination of volatile compounds in a liquid or solid matrices.

The samples are placed in headspace vials, tightly closed with a suitable cap and septum.

Each vial is heated, and the volatile compounds are transferred from the solid or liquid sample into the gaseous phase above it, called headspace, until a condition of thermodynamic equilibrium is reached.

Afterwards, an aliquot of headspace is withdrawn and injected into the gas chromatograph. Details on the principles of operation are available in the section "Analytical Cycle" on page 36.

Besides a sampling technique, the headspace technique is also an extraction and concentration technique. It offers the advantages of a reduced manipulation of the sample, a higher sensitivity than the direct injection, and a longer lifetime of chromatographic columns since only the volatile fraction is injected into the column.

The TriPlus 500 HS is directly coupled with a TRACE 1300/1600 Series GC and controlled by a Thermo Scientific Chromatography Data System, through the GC touch screen, if available, or the virtual GC user interface.

Instrument Basics

The TriPlus 500 Headspace Sampler (TriPlus 500 HS) consists of a 12-vial capacity configuration (TriPlus 500 HS-12), upgradeable to 120-vial capacity with the addition of a vial loader and a tray holder with three removable 40-vial trays (TriPlus 500 HS-120). The TriPlus 500 HS-120 can be further extended to 240-vial capacity with an additional tray holder and three 40-vial trays on the top of the GC.

See Figure 1 and Figure 2.

Figure 1. TriPlus 500 HS-12 Coupled with a TRACE 1600 GC

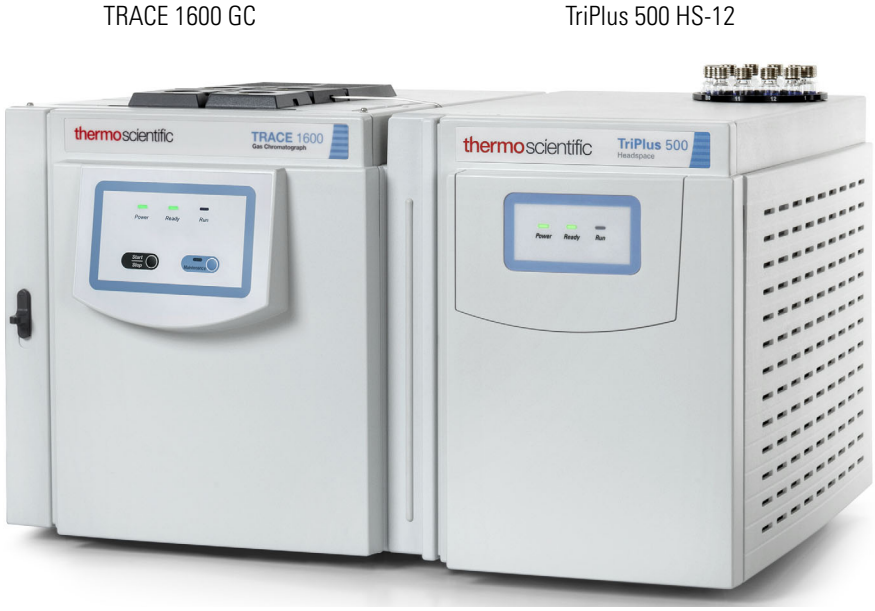


Figure 2. TriPlus 500 HS-120 Coupled with a TRACE 1600 Series GC

TRACE 1610 GC TriPlus 500 HS-120

TriPlus 500 HS-12 includes:

- **Sample Carousel** — A **12-seat** rotating carousel for 10 mL and 20/22 mL vials.
- **Incubation Oven** — A heated box including a 12-seat rotating oven carousel and mechanisms for shaking and transferring the vial. See "Incubation Group" on page 9.
- **Sampling Path** — Includes an electrically actuated 6-port gas sampling valve, a sampling needle and the GC column interface. See "Sampling Path" on page 10.
- **Pneumatic Connections** — Connect the supplies of carrier gas and auxiliary gas. See "Pneumatic Connections" on page 12.
- **Electrical Connections**— Includes electrical supply and communications ports to the GC optional devices. See "Electrical Connections" on page 13.
- **Status Panel** — Consists of three light emitting diodes (LED) showing the current status of the instrument. See "Status Panel" on page 14.

Note The TriPlus 500 HS-12 can be upgraded to a 120-sample-vial capacity at any time by adding the Vial Loader and the tray holder with three 40-vial trays.

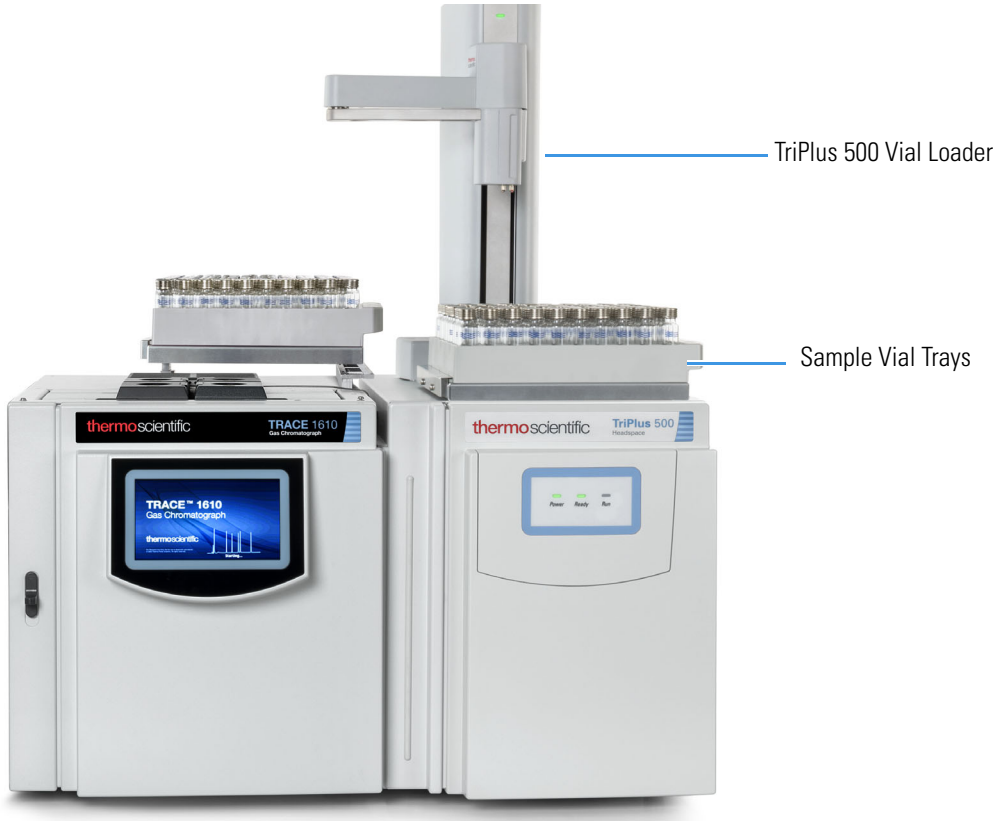
TriPlus 500 Vial Loader includes:

- One tray holder with three, 40-vial trays. See "Sample Trays" on page 20.
- **Vial Loader —** A robotic arm with a magnetic gripper that transfers the vials from each of the vial trays to the 12-position sample carousel, and vice-versa. The **Vial Loader** controls optional devices such as the **Barcode Reader** and **Heated/Cooled Tray** (Chiller). See **"Vial Loader" on page 17**, **"Barcode Reader" on page 25**, and **"Heated/Cooled Tray" on page 29**.

Note The sampling capacity of the **TriPlus 500 HS-12** can be increased to **240 vials** by adding another tray holder with three, 40-vial trays placed on the back part of a TRACE 1600 Series GC top cover.

Each sample tray has its own identification initials according to its position on the TriPlus 500 HS, on the GC, or on both. This allows for proper management of the vials during the sample sequence.

Figure 3. TriPlus 500 HS with 240 Vial Capacity

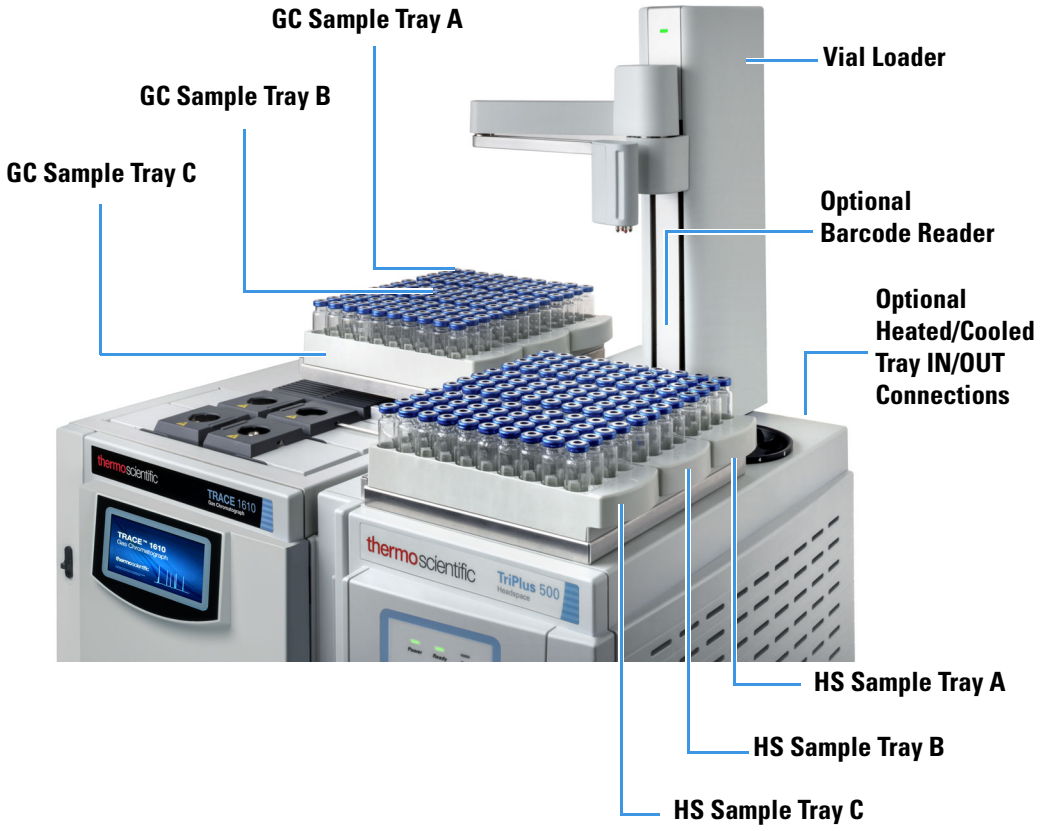


CAUTION DO NOT PLACE a vial into the 12-position rotating carousel when using the Vial Loader and the Vial Trays. The Vial Loader carries one vial at a time into the pre-defined position 1 of the carousel, while the other 11 positions are all potentially used for vial recovering.

ATTENTION NE PLACEZ PAS de flacon dans le carrousel rotatif à 12 positions lorsque vous utilisez le chargeur de flacon et les plateaux pour flacons. Le chargeur de flacon transporte un flacon à la fois dans la position 1 prédéfinie du carrousel, tandis que les 11 autres positions sont potentiellement utilisées pour la récupération des flacons.

Figure 4 shows an example of sampling system.

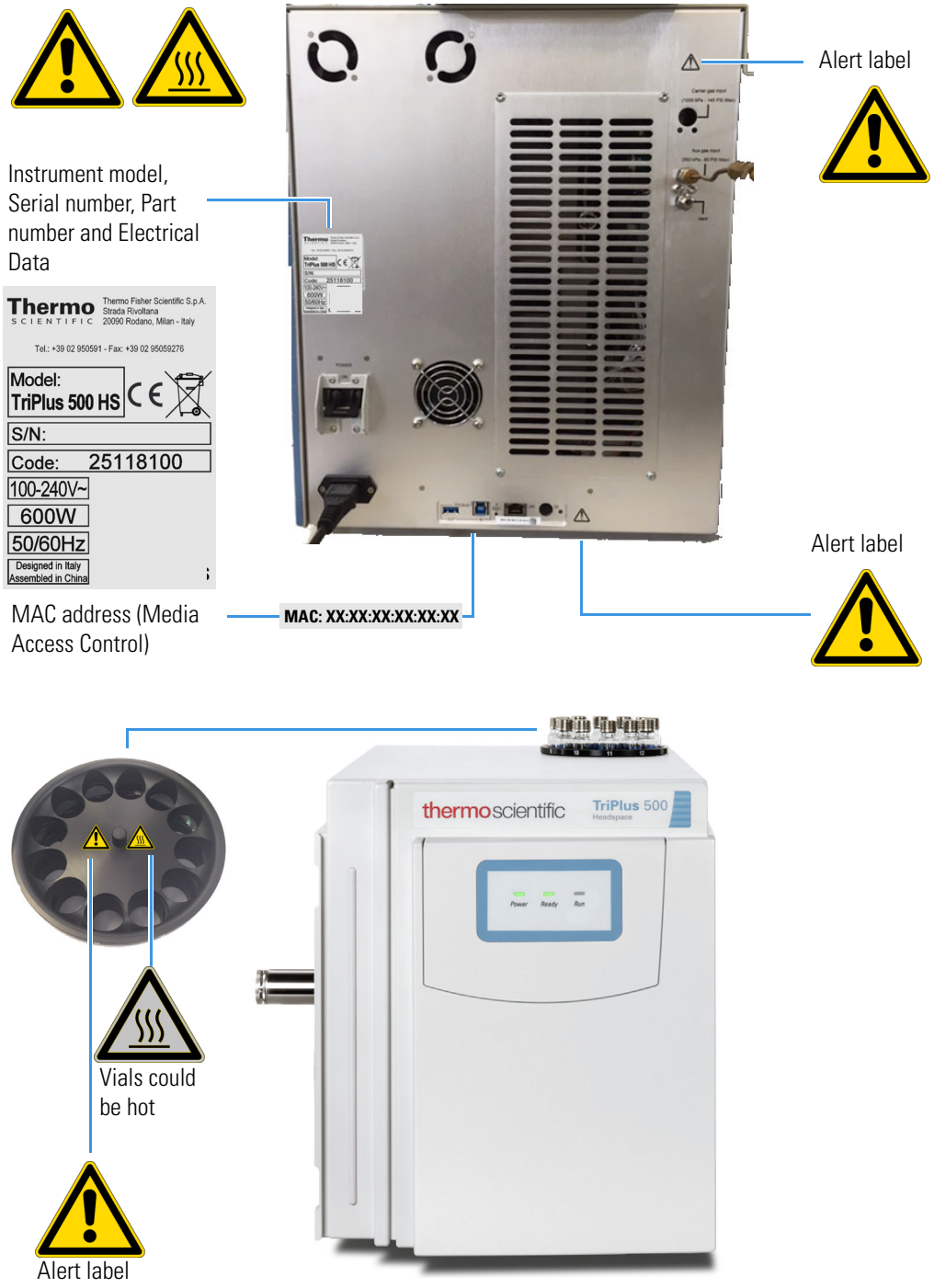
Figure 4. Example of the Sampling System at the Maximum Configuration



Label Locations on the Instrument

Figure 5 shows the locations of the labels attached to the TriPlus 500 HS.

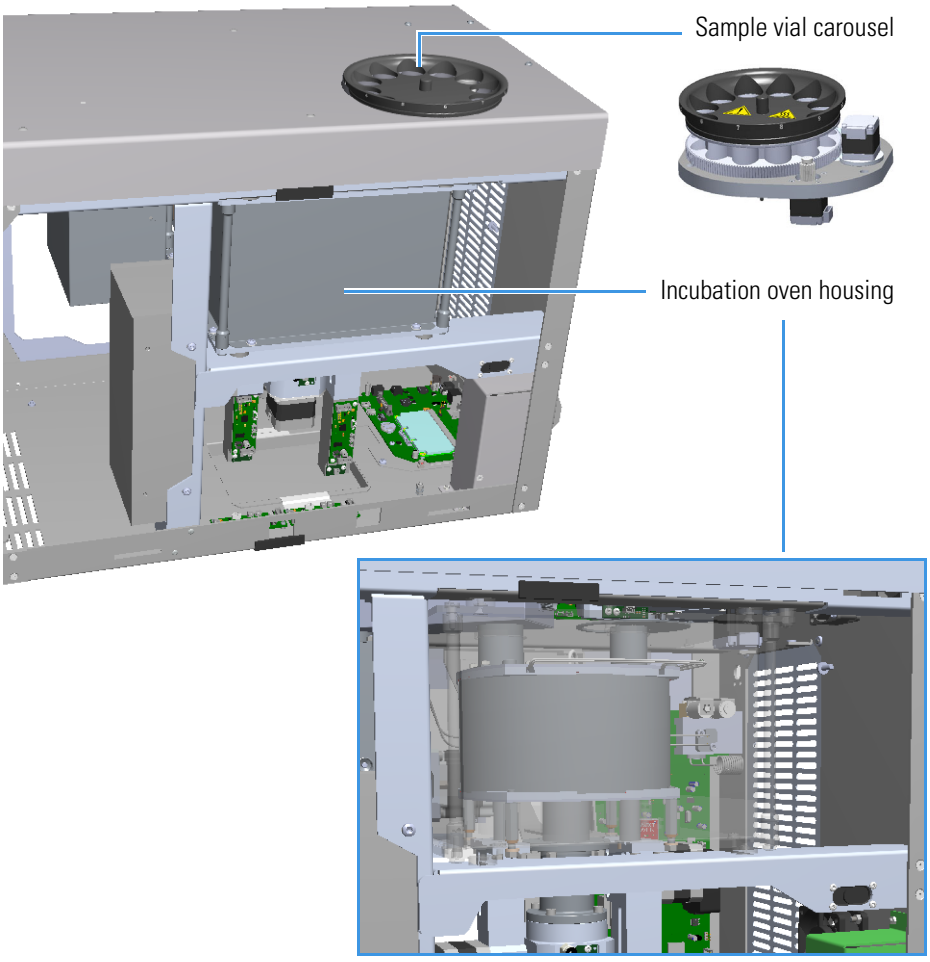
Figure 5. Labels



Incubation Group

The Incubation Group includes the **Sample Vial Carousel** and the **Incubation Oven**. See Figure 6.

Figure 6. Incubation Group



Sample Vial Carousel

The sample vial carousel consists of a **12-seat** rotating carousel, numbered from **1** to **12**, for vial housing. The carousel includes a mechanism for the automatic introduction and extraction of a vial into and from the **incubation oven** through the **incubation door**.

The incubation door has two positions: **Open** and **Closed**.

- **Open** — The door is open during the vial introduction and extraction into and from the incubation oven.
- **Closed** — The door always remains in this position except when a vial is introduced or extracted from the oven.

Incubation Oven

The incubation oven consists of a heated box including a **12-seat** rotating oven carousel.

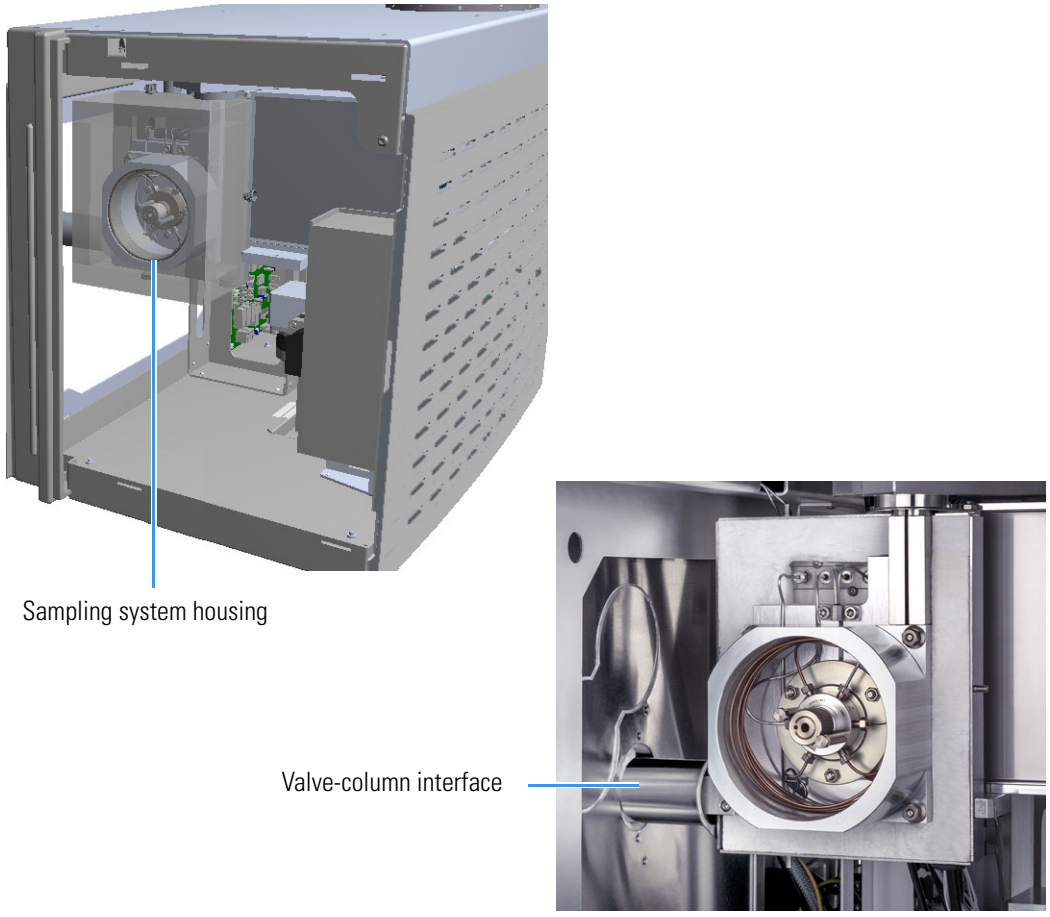
The vials are accurately thermostatted up to 300 °C and a fan provides a constant and uniform temperature. The vials are automatically inserted into the oven carousel from the sample vial carousel and can be shaken during the equilibration phase through the movements of the carousel.

Two motor-driven levers lift the vials out of the incubation carousel for sampling when they are placed in correspondence with the sampling needle, or for their recovery when they are placed under the inlet/outlet hole of the incubation oven.

Sampling Path

The sampling path consists of an electrically actuated 6-port sampling valve equipped with a deactivated stainless-steel sample loop, a sampling needle, and the valve-column interface. The valve assembly ends with a deactivated splitter, placed inside an insulating metal box, for the connection to the analytical column into the oven of the GC. See Figure 7.

Figure 7. Sampling Path



The sampling valve group is protected by a thermally insulated metal box. The valve is heated up to 225 °C with the standard factory-installed valve. An optional high temperature valve is available to heat the valve in range from 150 °C up to 300 °C.

CAUTION The valve must be used only in the specified temperature range. DO NOT EXCEED THIS RANGE.

ATTENTION La vanne doit être utilisée uniquement dans la plage de températures spécifiée. NE DÉPASSEZ PAS CETTE PLAGE.

A wide range of sample loops allows injection of different volumes of samples.

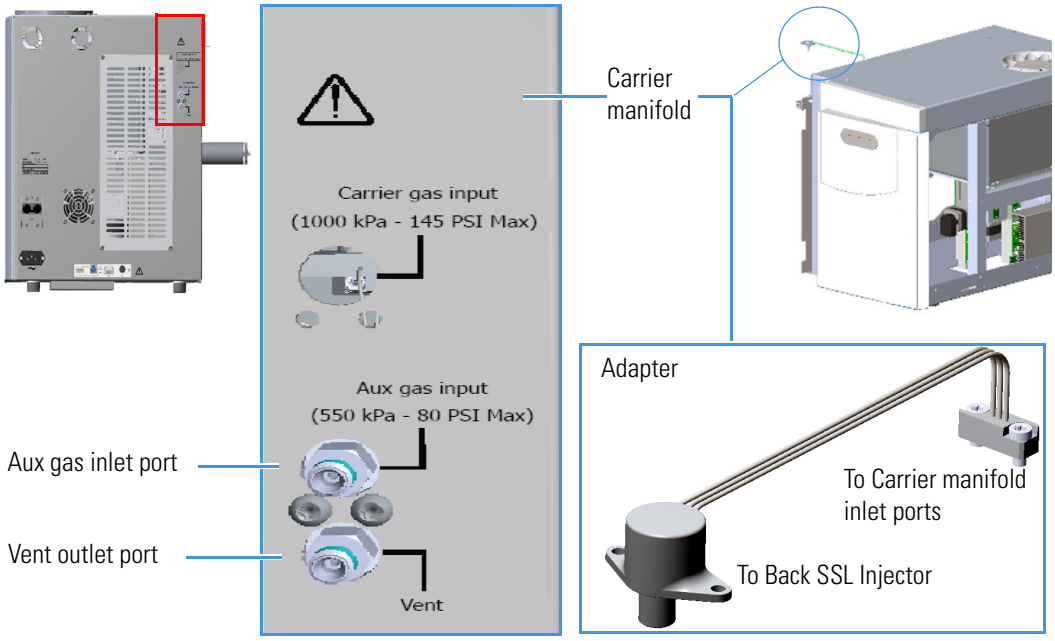
The sample loop is installed between the ports **3** and **6** of the sampling valve. The standard volume of the loop is **1.0 mL**.

The following optional sample loops are available: 25 µL, 50 µL, 100 µL, 500 µL, and 3 mL.

Pneumatic Connections

The pneumatic interface includes the inlet and outlet ports to make the pneumatic connections with the sampling path and the external devices. See Figure 8.

Figure 8. Pneumatic Interface



The pneumatic interface includes:

- A manifold for connecting the carrier gas coming from the Back SSL injector module on the GC through a dedicated adapter.

  Note For installing and connecting the adapter between the Back SSL injector module on the GC and the TriPlus 500 HS, refer to the *TriPlus 500 Headspace Sampler Hardware Manual*.

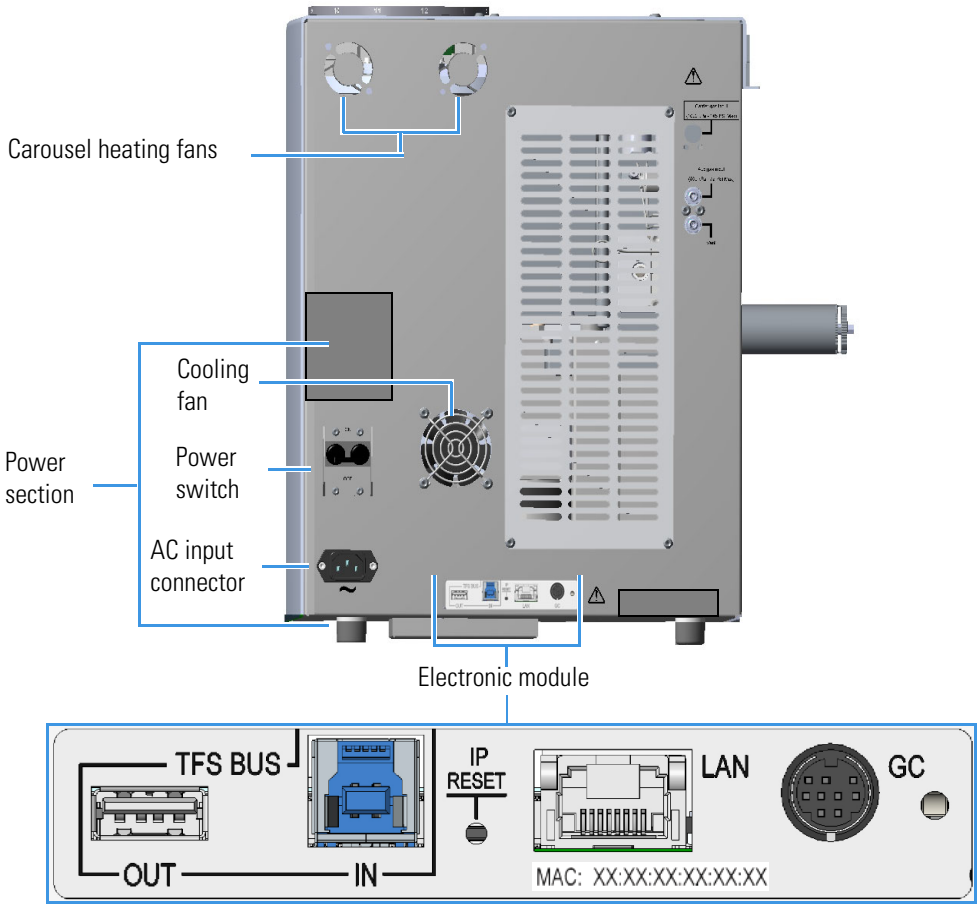
- An inlet port marked **Aux gas input** (**550 kPa - 80 PSI Max**) for connecting the auxiliary gas supply from the cylinder.
- An outlet port marked **Vent** for connecting the HS sampler to an exhaust device.

  Note **Carrier gas input** connection is not currently in use.

Electrical Connections

The electrical interface includes the **power section** and the **electronic module** to make electrical and communication connections among the HS sampler and the external devices. See Figure 9.

Figure 9. Electrical Interface



The power section includes:

- The Power Switch marked **Power** to turn the instrument On/Off.
  - Position ON = instrument powered On
  - Position OFF = instrument powered Off
- AC Input connector (Main socket) for connecting the power cable to the sampler and the wall outlet

  The power rating is: 100-240 Vac, 50/60 Hz; 600 W

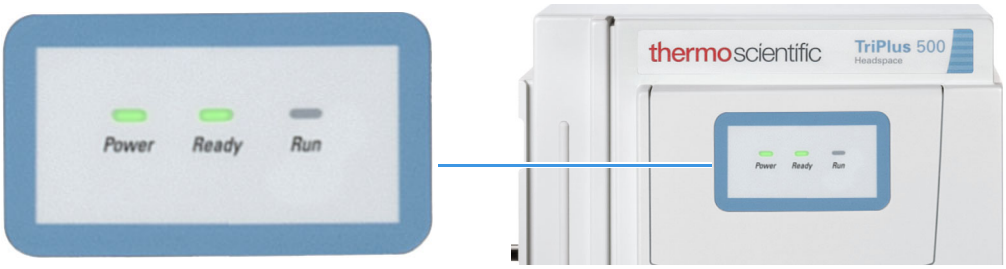
The electronic module includes:

- Two ports marked **TFS BUS (IN)** and **TFS BUS (OUT)** for the interconnection with the TriPlus 500 HS units such as the Vial Loader.
- A RJ45 connector marked **LAN** for connecting to the network.
- A button marked **IP Reset** for resetting the IP address.
  - Press the **IP Reset** button for more than 0.5 seconds to reset the Web server credentials to the default settings. A single beep will be audible.
  - Press the **IP Reset** button for more than 4 seconds to reset the network configuration to the default settings. Three beeps will be audible.
- A 9-pin connector marked **GC** to synchronize the HS sampler with the GC (**Ready In/Out** and **Start In/Out** signals). See the **Autosampler** connector on the back of the TRACE 1300/1600 Series GC.

Status Panel

The Status Panel consists of three light emitting diodes (LED), on the front door of TriPlus 500 HS, showing the instrument's current status. See Figure 10.

Figure 10. Status Panel



The three LEDs are:

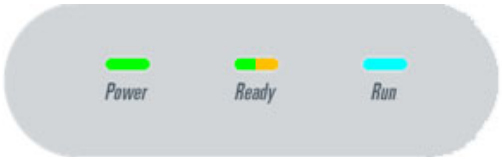
- **Power** — When the LED lights are **green**, the HS sampler is powered on.
- **Ready** — When the LED lights are **green**, the HS sampler is in **Ready** condition. When the LED blinks **orange** the HS sampler is in **Not Ready** condition.
- **Run** — When the LED lights are **blue**, the HS sampler is running an analysis.

Every instrument's event/phase is associated with a status of the LED as detailed below:

1. **Power On TriPlus 500 HS**

   All the LEDs on the status panel light up simultaneously. Next, the **Power** light becomes a solid **green**, while the **Run** light flashes during the initialization phase. See Figure 11.

Figure 11. Power On



2. **Not Ready status**

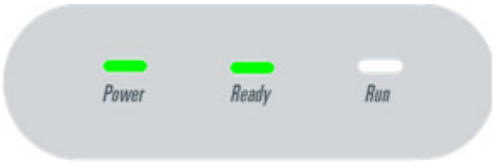
   When the HS sampler is in **Not Ready** status, the **Ready** light blinks **orange**. See Figure 12.

Figure 12. Not Ready Status

3. **Ready status**

   When the sampler parameters have reached the setpoint values, the **Ready** light becomes a solid **green**. See Figure 13.

Figure 13. Ready Status



4. **Run status**

   When the sampler is running, the **Run** lights solid **blue**, while the **Ready** lights **off**. See Figure 14.

Figure 14. Run Status

5. **Error**

   When an alarm condition is detected, the **Power** light is blinking. See Figure 15.

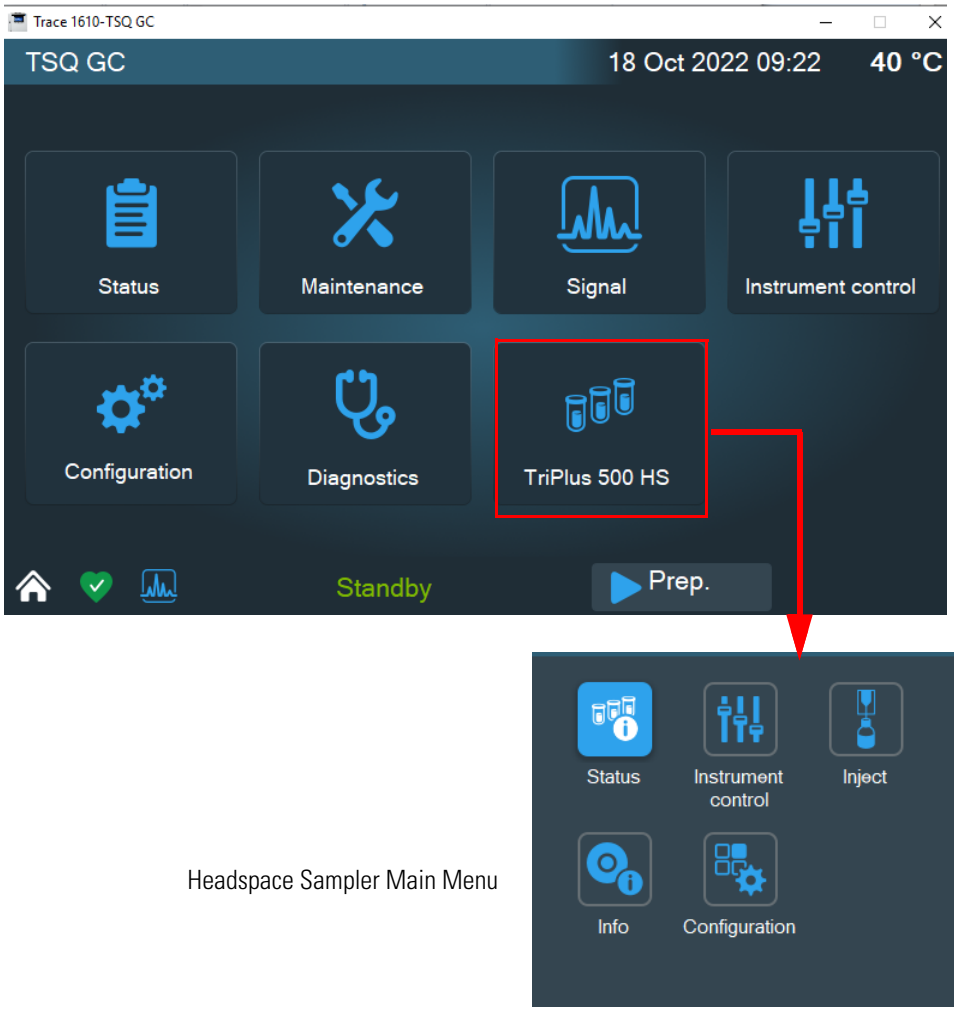
Figure 15. Error Condition

TRACE 1610 GC User Interface

The Human-Machine Interface (HMI) of the TRACE 1610 GC recognizes the connected TriPlus 500 HS. An icon is displayed on the touch screen to open the **Headspace Sampler Main Menu**. See Figure 16.

For setting the HS sampler parameters see the Chapter 3, "Setting Up Through the TRACE 1610 User Interface."

Figure 16. Touch Screen Main Menu - Headspace Sampler Icon



Headspace Sampler Main Menu

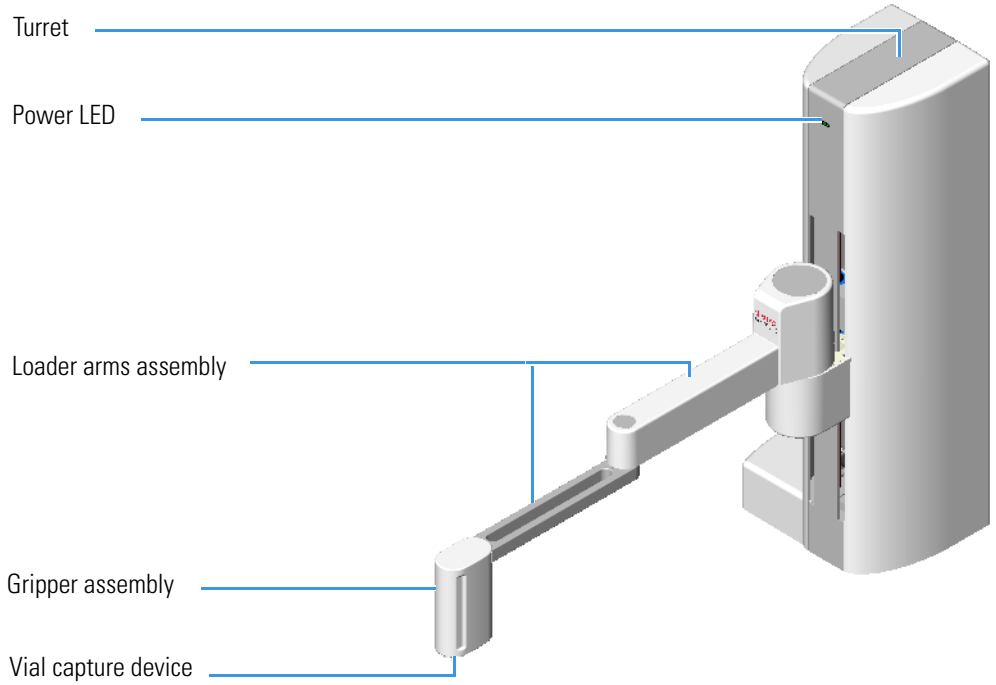
Vial Loader

The Vial Loader is a device for upgrading the sampling capacity up to 120 vials adding up to three 40-position sample trays. The sampling capacity can be further increased up to **240 vials** adding other three 40-position sample trays placed on the back part of a TRACE 1300/1600 Series GC top cover.

The device transfers one vial at a time from the sample tray to the pre-defined position **1** of the 12-position sample carousel, and vice-versa. The Vial Loader can control additional external devices such as the **Barcode Reader** and a Chiller connected to the **Heated/Cooled Tray**.

The Vial Loader consists of the following parts. See Figure 17.

Figure 17. Vial Loader



Turret — Includes the mechanisms for moving the loader arms assembly.

Power LED — When the Power LED light is solid **green**, the Vial Loader is powered on. The power LED light blinks **green** during the movements of the loader arms.

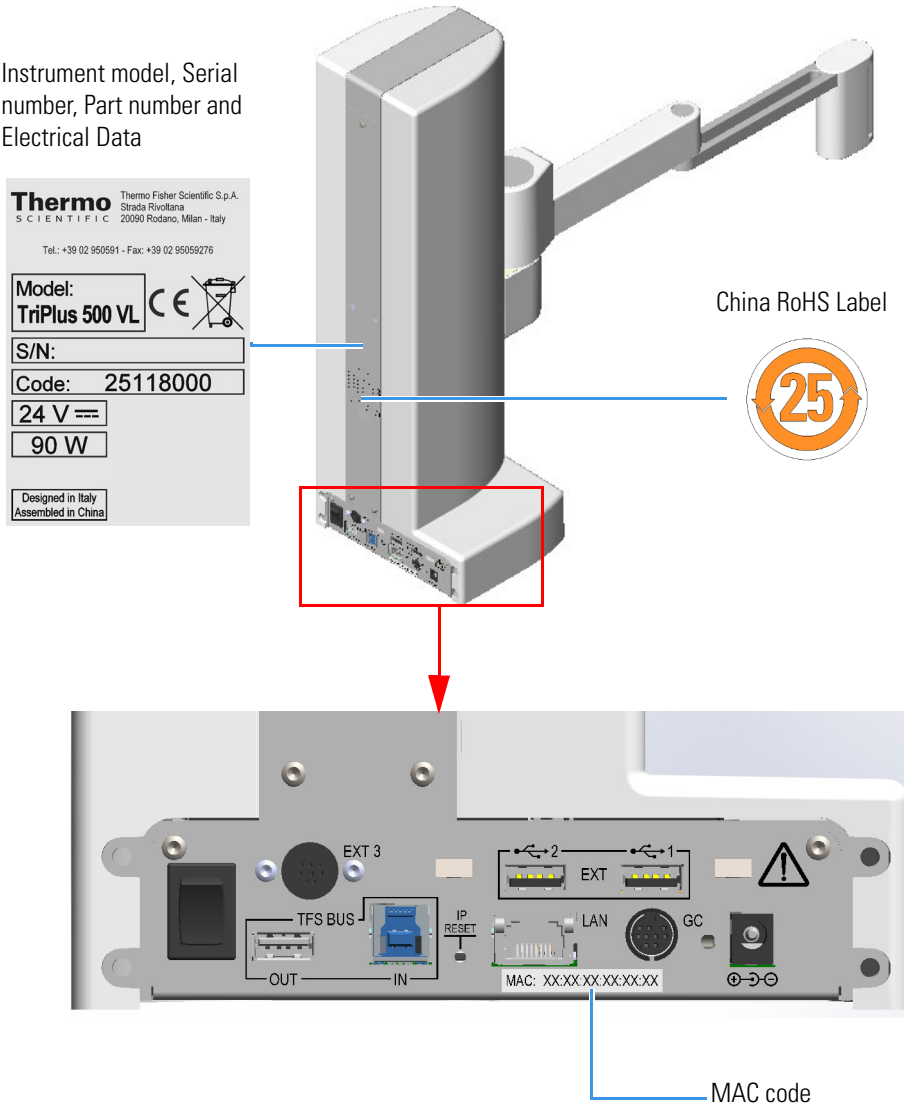
Loader arms assembly — Transfers the sample vial from a sample tray to the sample carousel and vice-versa through the **Vial capture device** located on the bottom of the **Gripper** assembly.

Gripper assembly — Captures the vial and carries it from a sample tray holder to the sample carousel and vice-versa through the four magnets of the capture device.

Vial Loader Electronic Module

The electronic module on the back of the Vial Loader includes the connectors to make electrical and communication connections. See Figure 18.

Figure 18. Vial Loader Electronic Module



The electronic module includes:

- The Power Switch to turn the instrument On/Off.
  - Position **I** = instrument On
  - Position **O** = instrument Off
- A jack socket marked ⊕⊃⊖ for the instrument power supply through a portable external power supply, level VI efficiency.

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

Input 100-240 Vac; 50/60 Hz; 1.3 A— Output 24 Vdc; Power 90 W; 3.75 A

WARNING You must only use the portable external power supply furnished with the instrument by Thermo Fisher Scientific.

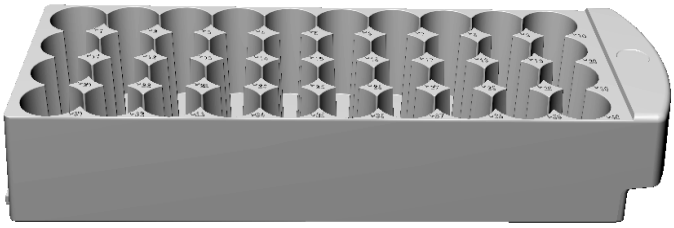
AVERTISSEMENT Vous ne devez utiliser que l'alimentation externe portable fournie avec l'instrument par Thermo Fisher Scientific.

- Two ports marked **TFS BUS (IN)** and **TFS BUS (OUT) are** for the interconnection with the units of the TriPlus 500 HS as the Vial Loader.
- A 9-pin connector marked **GC** to synchronize the HS sampler with the GC (Ready In/Out and Start In/Out signals). See the connector marked **Autosampler** on the back of the TRACE 1300/1600 Series GC.
- Two USB ports marked **EXT** **1** and **2** for the connection to external devices, controlled by the Vial Loader, as Barcode Reader and Chiller.
- A 8-pin connector marked **EXT 3** for the connection to an auxiliary external device.
- A button marked **IP Reset** to reset the IP address.

Sample Trays

The sample tray can contain up to forty 10 mL or 20/22 mL vials arranged on four rows. See Figure 19 and Figure 20.

Figure 19. 40-position Sample Tray (1)



First row = Vial **1** to Vial **10**
Second row = Vial **11** to Vial **20**
Third row = Vial **21** to Vial **30**
Fourth row = Vial **31** to Vial **40**

Figure 20. 40-position Sample Tray (2)

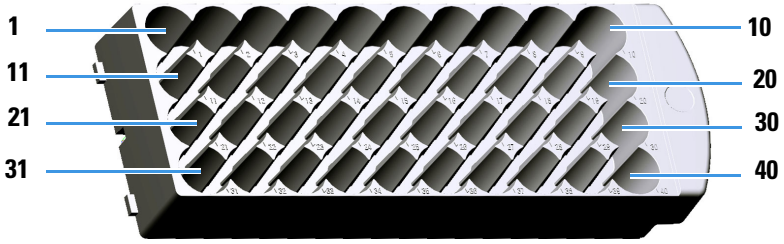
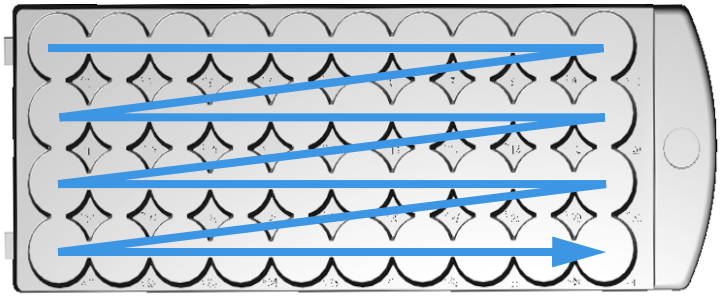


Figure 21 shows the typical sampling sequence from vial **1** to vial **40**.

Figure 21. Typical Vial Sampling Sequence

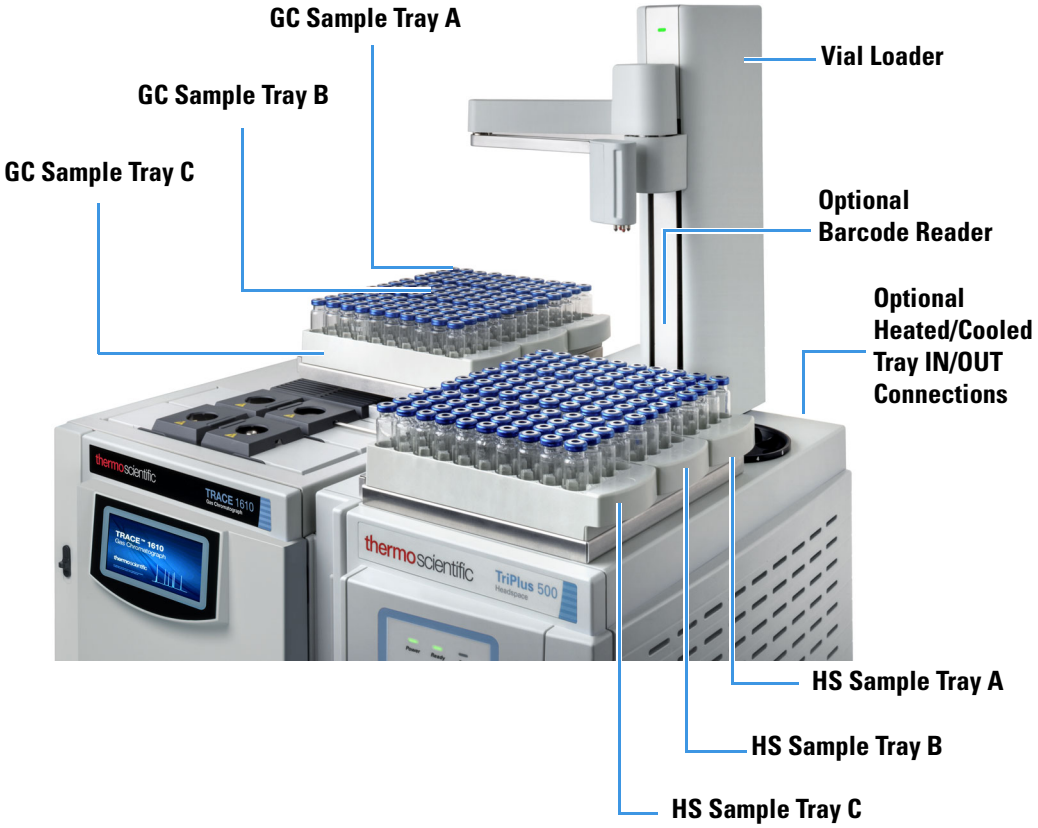


Each sample tray has its own identification initials according to the positioning on the TriPlus 500 HS, TRACE 1300/1600 Series GC, or on both the instruments:

- **HS Sample Tray A**, **B**, and **C** — Sample trays installed on the top cover of a TriPlus 500 HS
- **GC Sample Tray A**, **B**, and **C** — Sample trays installed on the top cover of a TRACE 1300/1600 Series GC

See Figure 22 and the *TriPlus 500 Headspace Sampler Hardware Manual* for more information.

Figure 22. Sample Tray Positions



CAUTION Because the sample trays have all the same vial numbering from 1 to 40, when programming a sequence of samples it is mandatory to specify the trays used and the number of vials placed in each sample tray.

DO NOT place 10 mL and 20/22 mL vials into the same sample tray.
If 10 mL and 20/22 mL vials are used simultaneously, the 20/22 mL vials can only be placed on **HS Sample Tray C** and/or **GC Sample Tray A**.

For further details see the sections "" on page 22 and "Positioning the Vials in the Sample Tray" on page 23.

ATTENTION Puisque les plateaux d'échantillons ont tous la même numérotation de flacons, allant de 1 à 40, lors de la programmation d'une séquence d'échantillons, il est obligatoire de spécifier les plateaux utilisés et le nombre de flacons placés dans chaque plateau.

Ne placez PAS les flacons de 10 ml et 20/22 ml dans le même plateau d'échantillons.

Si des flacons de 10 ml et 20/22 ml sont utilisés simultanément, les flacons de 20/22 ml peuvent être placés uniquement sur le plateau C d'échantillons HS et/ou sur le plateau A d'échantillons GC.

Pour plus d'informations, consultez les sections Syntaxe de la séquence des flacons à la page 22 et Positionnement des flacons sur le plateau d'échantillons à la page 23.

Note An optional metallic version of the sample tray is available for heating/cooling the vial. See "Heated/Cooled Tray" on page 29.

Vials Sequence Syntax

This section provides the correct syntax for identifying the position of the vials when compiling the sample table (column **Position**) for the sequence of the samples.

Syntax for Sample Carousel

Sample Carousel:

- Syntax for the vials in the carousel = **1.....12**

Syntax for Sample Trays

Sample trays installed on TriPlus 500 HS Sampler:

- Syntax for the vials in the HS Sample Tray A = **HS:A1.....HS:A40**
- Syntax for the vials in the HS Sample Tray B = **HS:B1.....HS:B40**
- Syntax for the vials in the HS Sample Tray C = **HS:C1.....HS:C40**

Sample trays installed on a TRACE 1300/1600 Series GC:

- Syntax for the vials in the GC Sample Tray A = **GC:A1.....GC:A40**
- Syntax for the vials in the GC Sample Tray B = **GC:B1.....GC:B40**
- Syntax for the vials in the GC Sample Tray C= **GC:C1.....GC:C40**

Example of vials setting in the sample table:

Only using Carousel without Vial Loader

Position
1
3
3
4
5
6
7
....

Using Vial Loader and Sample Trays

Position
HS:A1
HS:A2
HS:A23
HS:B2
HS:B40
HS:C13
GC:A7
....

Positioning the Vials in the Sample Tray

CAUTION The sample tray can contain up to 40,10 mL or 20/22 mL vials arranged on four rows. Each sample tray MUST contain vials of the same type. DO NOT place 10 mL and 20/22 mL vials into the same sample tray.

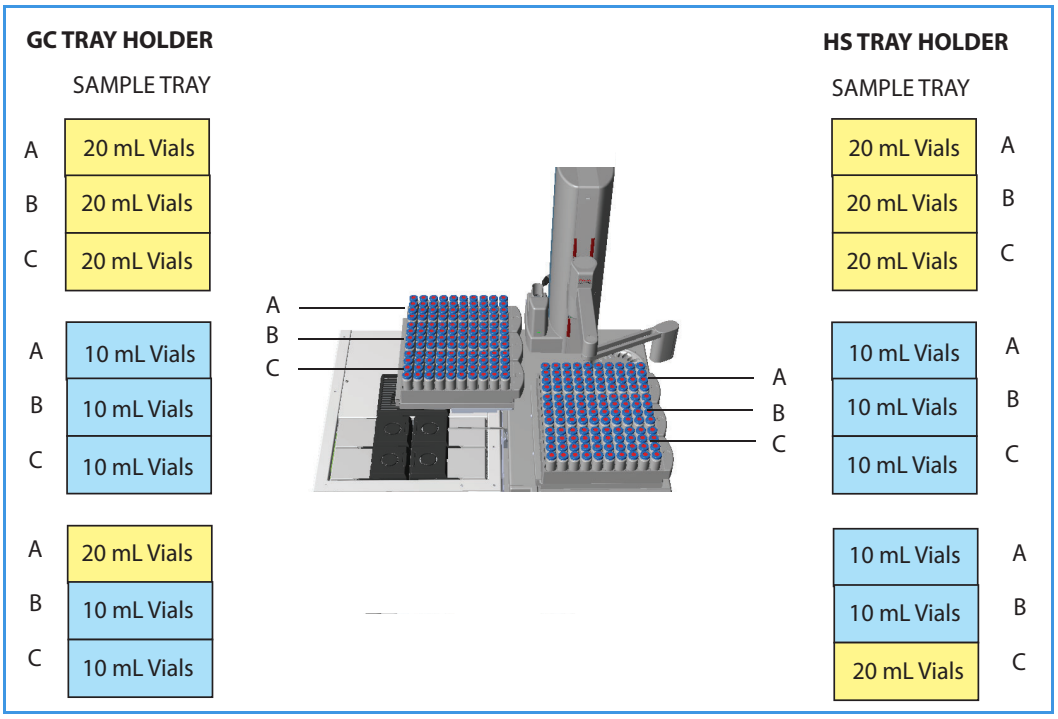
In the case both 10 mL and 20/22 mL vials are used, they must be placed in the system according to the scheme of Figure 23.

ATTENTION Le plateau d'échantillons peut contenir un maximum de 40 flacons de 10 ml ou 20/22 ml disposés sur quatre rangées. Chaque plateau d'échantillon DOIT contenir des flacons du même type. Ne placez PAS les flacons de 10 ml et 20/22 ml dans le même plateau d'échantillons.

Dans le cas où des flacons de 10 ml et 20/22 ml sont utilisés, ils doivent être placés dans le système conformément au schéma de la Figure 23.

Figure 23 shows the possible positioning of the 10 mL and 20/22 mL vials into the **HS** and **GC** Sample Trays **A**, **B** and **C**.

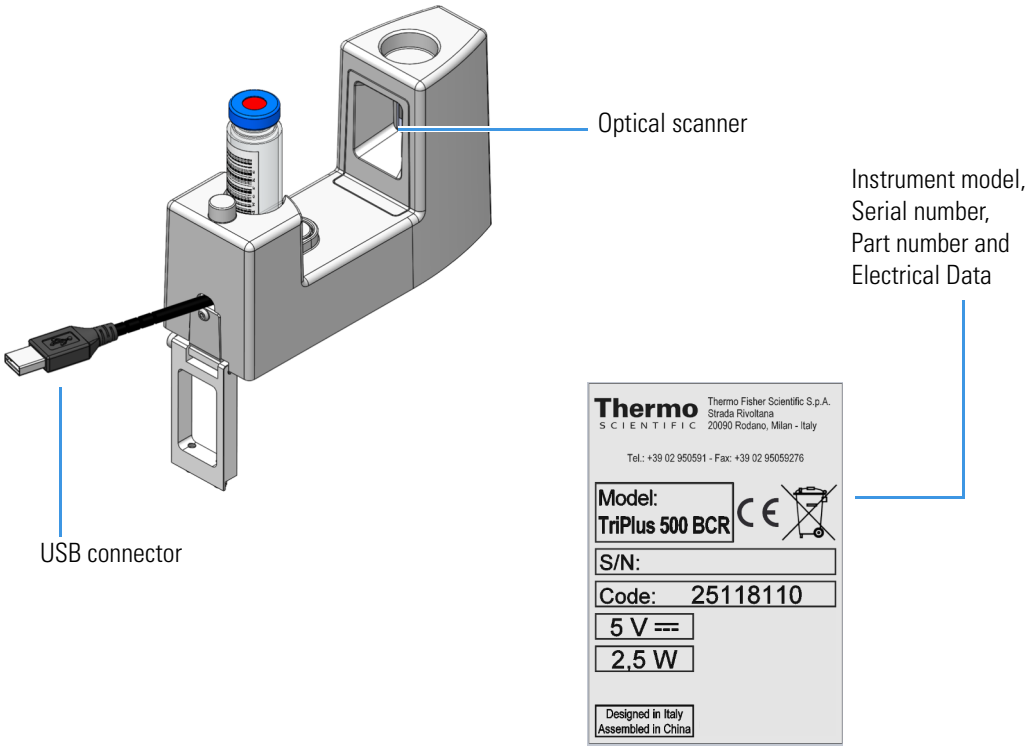
Figure 23. Sample Vials Positioning



Barcode Reader

This device allows the instrument to read barcodes on labels. Stickers with the barcode containing the codified data of the samples are glued on the wall of the relevant sample vials. See Figure 24.

Figure 24. Barcode Reader



Barcode Reader Overview

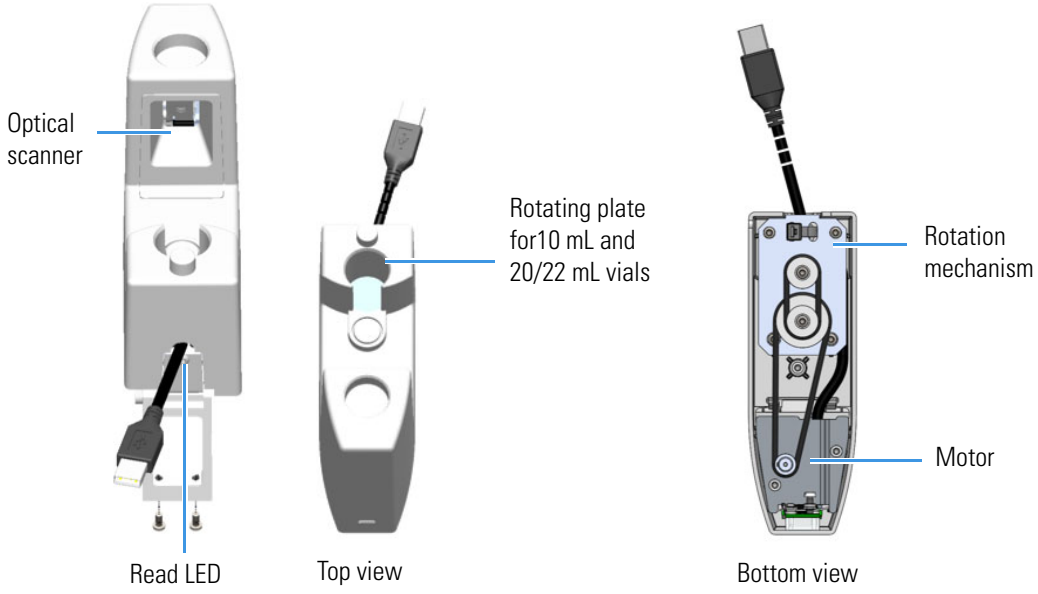
A barcode is an optical machine-readable representation of data relative to the object to which is attached.

Originally, the barcode represented data change by varying the widths and spacings of parallel lines (linear or one-dimensional), later they evolved into rectangles, dots, hexagons, and other two-dimensional (2-D) geometric patterns.

Barcode Reader Components

The Barcode Reader consists of the following components. See Figure 25.

Figure 25. Barcode Reader Components



- An optical scanner arm for enabling reading the barcode.
- A rotating plate for placing a 10 ml or 20/22 mL vial.
- A motor and a mechanism for rotating the vial for the complete reading of the barcode.
- A frontal Read LED which flashes every time a barcode label is read.

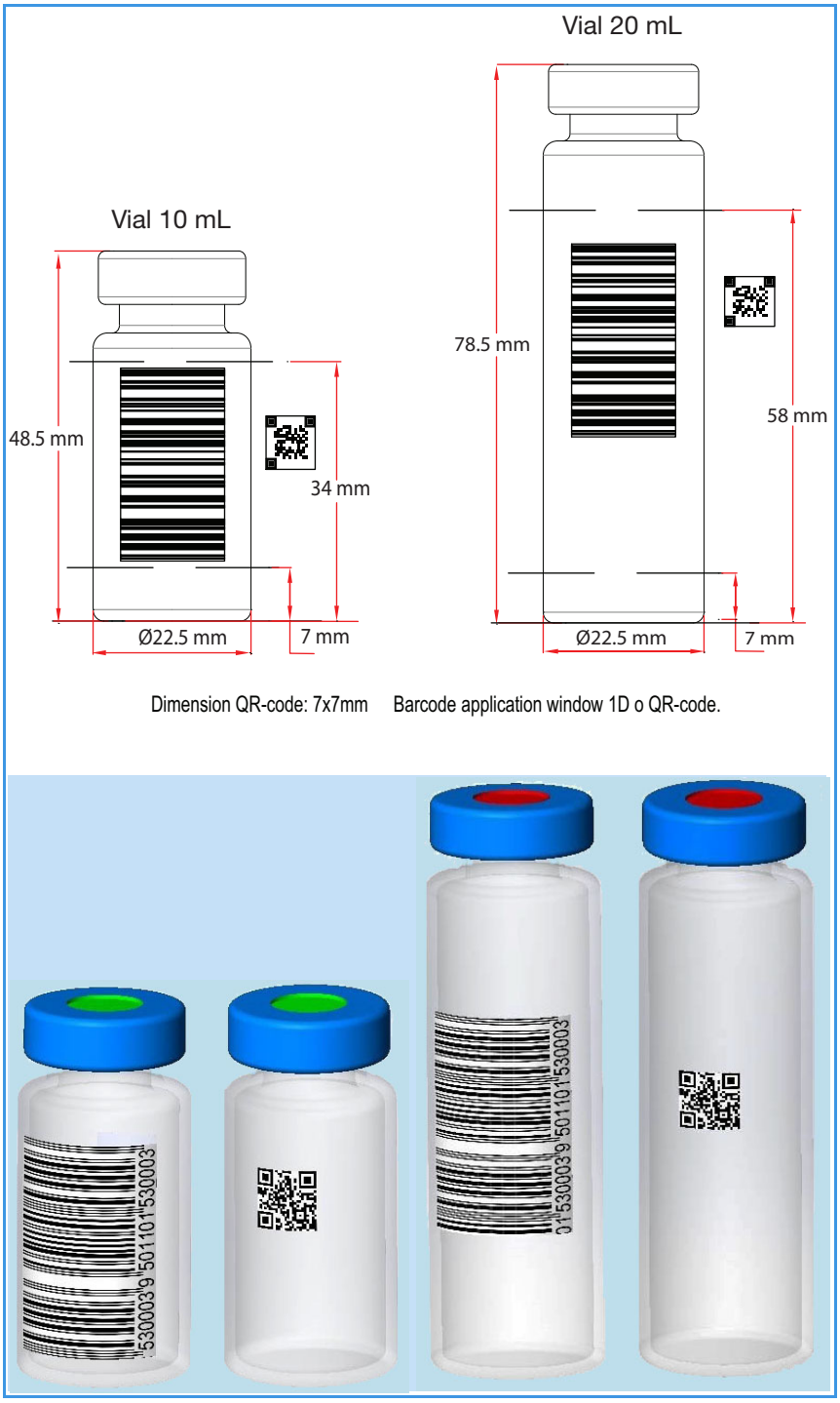
Barcode Labels

The Barcode labels should be made of polyester and not paper. Polyester can withstand high temperatures and the barcode lines will print clearly.

- **Label Width** — The minimum length of the barcode is 18 mm if used with a 20 mL vial. This dimension refers to the actual barcode length and not the label itself. Adapt the label accordingly. When possible use larger dimensions for reliable processing of the barcode.
- **Maximum tilt of the barcode label**: ±20°
- **Minimal barcode density (minimal width of a bare or a space)**: 5 mil (0.005 in.) / 0.127 mm

Place the barcode label on the vial so the barcode bars are horizontally positioned. The allowed area for the placing of the label is shown in Figure 26.

Figure 26. Examples of Barcode Label Positioning and Label Size



- The operator can test reading the barcode label on the vial through the TriPlus 500 Web Interface.
- For details see Chapter 7, "Using the TriPlus 500 Web Interface."

Supported Types of Barcode Symbols

Table 1 lists a series of barcode types that can be decoded.

Table 1. Barcode Symbology (Sheet 1 of 2)

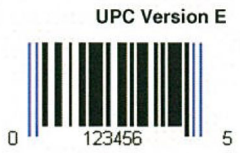
Barcode Type	Description	Minimum Length	Maximum Length	Barcode Symbology
Key for Barcode Symbology				
UPC-A	Universal Product Code, 12 numerical digits. 11 usable digits +1 check digit.	12	12	
UPC-E	Universal Product Code, Zero-compressed UPC code, 7 numerical digits. 6 usable digits + 1 check digit.	6	6	
EAN-8	Derived from the longer European Article Number (EAN-13), 8 numerical digits. 7 usable digits + 1 check digit.	7	7	
EAN-13	European (International) Article Number, 13 numerical digits. 12 usable digits + 1 check digit.	12	12	
Code-128	High density barcode for alphanumerical codes, supporting all 128 ASCII characters.	1	Unlimited	
EAN-128/GS10128	Alpha-numerical codes, supporting all 128 ASCII characters.	1	48	
Code 39 (3 of 9)	Alphanumeric code, consisting of uppercase letters (A-Z), numeric digits (0-9) and some special characters (-, ., $, /, +,%, and space).	1	Unlimited	

Table 1. Barcode Symbology (Sheet 2 of 2)

Barcode Type	Description	Minimum Length	Maximum Length	Barcode Symbology
2 of 5 Interleaved	Numerical characters.	1	Unlimited	
ISBT 128	Used for labeling of human blood.	1	Unlimited	Application specific barcodes
QR-Code	Two-dimensional 2D Barcode symbology	7	7	

Heated/Cooled Tray

The Heated/Cooled Tray is an optional accessory for heating/cooling the vials on the sample tray through the circulation of a fluid underneath the dedicated heated/cooled tray holder plate. See Figure 27.

Figure 27. Heated/Cooled Tray Holder Plate

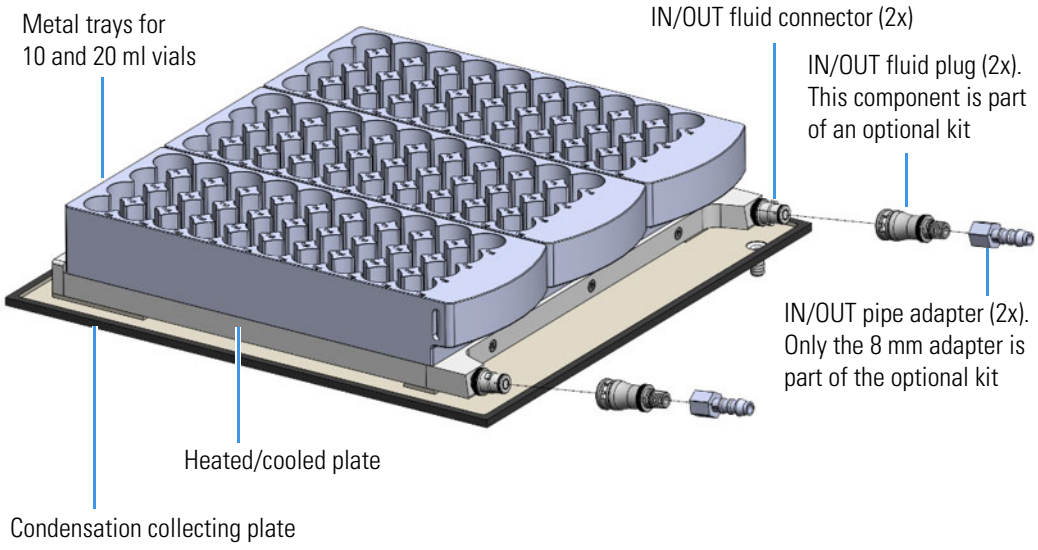
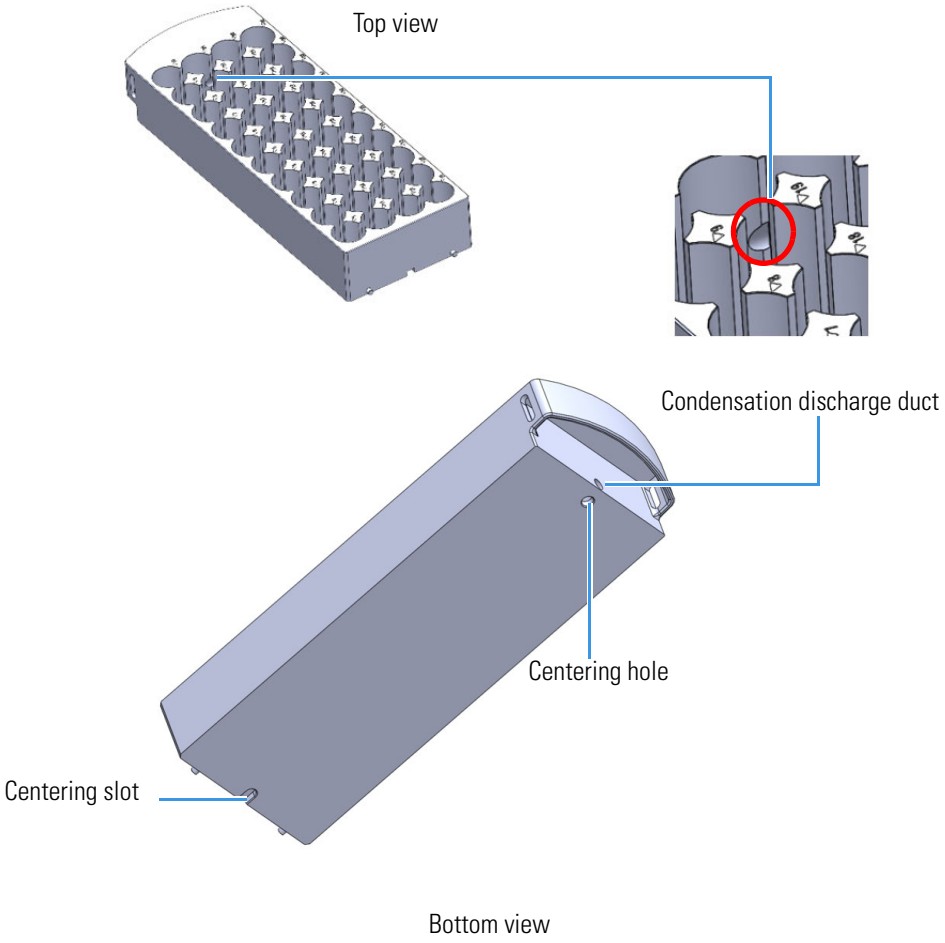


Figure 28 shows a view of the aluminum heated/cooled vial tray.

Figure 28. Heated/Cooled 40-position Vial Tray



For heating and cooling the sample vials an external recirculating device is required. All control parameters must be set on the device in use by referring to the relevant manual.

Note If you do not have an external recirculating device in your laboratory, Thermo Fisher Scientific recommends the Thermo Scientific™ Accel™ 500 LC Cooling/Heater Recirculating Chiller Part Number 223422100. This model is directly controlled by the data system. See Figure 29.

Figure 29. Accel 500 LC Optional Chiller

Working temperature range:
-10 °C to +80 °C (+14 °F to 176 °F)

Cooling capacity at 20 °C:
500 W / 1706 BTU

Heating capacity at 20 °C:
2 kW / 6829 BTU

Power supply:
230 V; 50 Hz

Dimensions (H x W x D):
62.0 x 23.2 x 48.7 cm
24.8 x 9.1 x 19.2 in.

Weight:
30 kg; 66 lb

This heating/cooling method can be controlled through the Vial Loader and the Thermo Scientific™ Chromeleon™ or TraceFinder™ Chromatography Data System.

For details, refer to the *Thermo Scientific™ Accel™ Series Cooling/Heating Recirculating Chillers Manual (P/N U01076)*.

Note Installation instructions for the **Heated/Cooled Tray** are in the *TriPlus 500 Headspace Sampler Hardware Manual*:

TriPlus 500 Web Interface

The TriPlus 500 Web Interface is a web-based application used for instrument control of the TriPlus 500 HS. The operator can check the network configuration and the status of the instrument. Also, it is possible to perform calibration of the tray holder, leak check and a reading test of the barcode label on the vial.

For further details about the use of the TriPlus 500 Web Interface see Chapter 7, “Using the TriPlus 500 Web Interface.”

2

Operating Principles

This chapter describes the operating principles of the TriPlus 500 HS.

Contents

- Introduction to the Headspace Technique
- Pneumatics
- Analytical Cycle

Introduction to the Headspace Technique

Headspace Gas Chromatography (HSGC) is an indirect method of analysis for the determination of volatile components in liquid and solid samples. The principle of operation of this technique is based on the gas chromatographic analysis of the sample gas vapor phase in thermodynamic equilibrium with the sample in a closed vial. Under these conditions, the quantity of volatile components in the headspace gas is proportional to their concentration in the sample.

The gas chromatographic analysis of the gaseous phase proved to be the most practical and reliable analytical method in the headspace technique. In fact, the combined use of this technique and gas chromatography permits full exploitation of this analytical method (high efficiency, sensitivity and selectivity) while preserving its basic simplicity, such as sample preparation and reliability of results.

HSGC eliminates all the disadvantages connected with traditional sample preparation requiring tedious and time-consuming procedures to concentrate the trace components, and it avoids possible interference from non-volatile constituents.

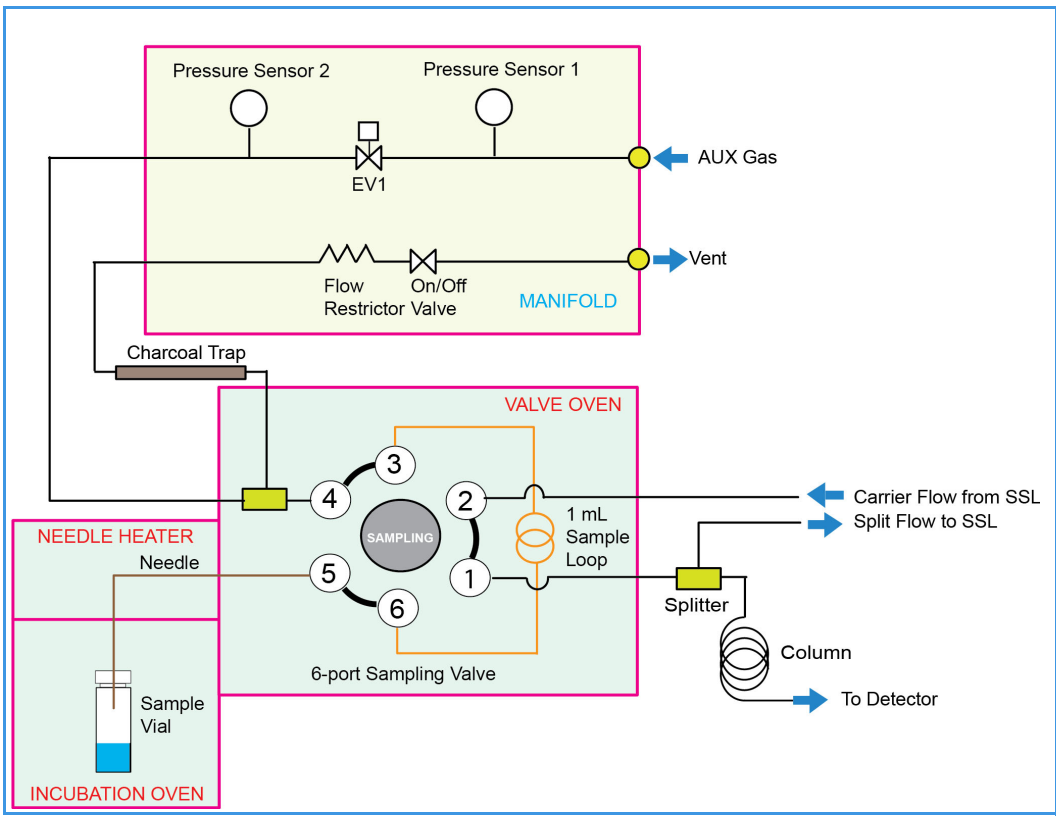
This technique is also particularly suitable for the determination of traces in samples, which cannot be injected directly into the gas chromatograph because of column overloading or contamination, decomposition or dissociation problems. The main advantages of HSGC can be summarized as follows:

- Only clean gas samples are injected, thus extending the column life time, promoting higher sensitivity and easier use of selective detectors
- Preliminary sample preparation is negligible thus reducing the analysis time, which is of primary importance in routine analysis
- Higher analytical sensitivity in the case of compounds whose partition coefficient is more favorable towards the gas phase. In this case the absolute component amount injected into the GC is larger than the maximum allowable liquid sample that might be injected into the chromatographic system
- The sample is not vaporized in the GC, therefore its partial decomposition is avoided and interference of other components eliminated
- Taking advantage of its intrinsically high modularity, TriPlus 500 HS expands the capabilities of the TRACE 1300/1600 Series GC

Pneumatics

The pneumatic diagram of TriPlus 500 HS is schematically shown in Figure 30.

Figure 30. Pneumatic Diagram



- The **carrier gas** is regulated by the GC. The carrier gas flows into the HS sampler through the **adapter gas tubing block** installed on the **Back SSL injector**. The carrier gas used with the GC can be helium, nitrogen, hydrogen, air or argon. Other gases are rarely used.
- The **auxiliary gas** pressurizes the vials and transfers the analytes from the sample into the sample loop. The auxiliary gases used with the HS sampler are helium, nitrogen or argon. The auxiliary gas is regulated by an electronic pressure control.

Analytical Cycle

The analytical cycle of a sample is made up of a sequence of operative phases starting from the vial loading into the thermostatic oven until its unloading.

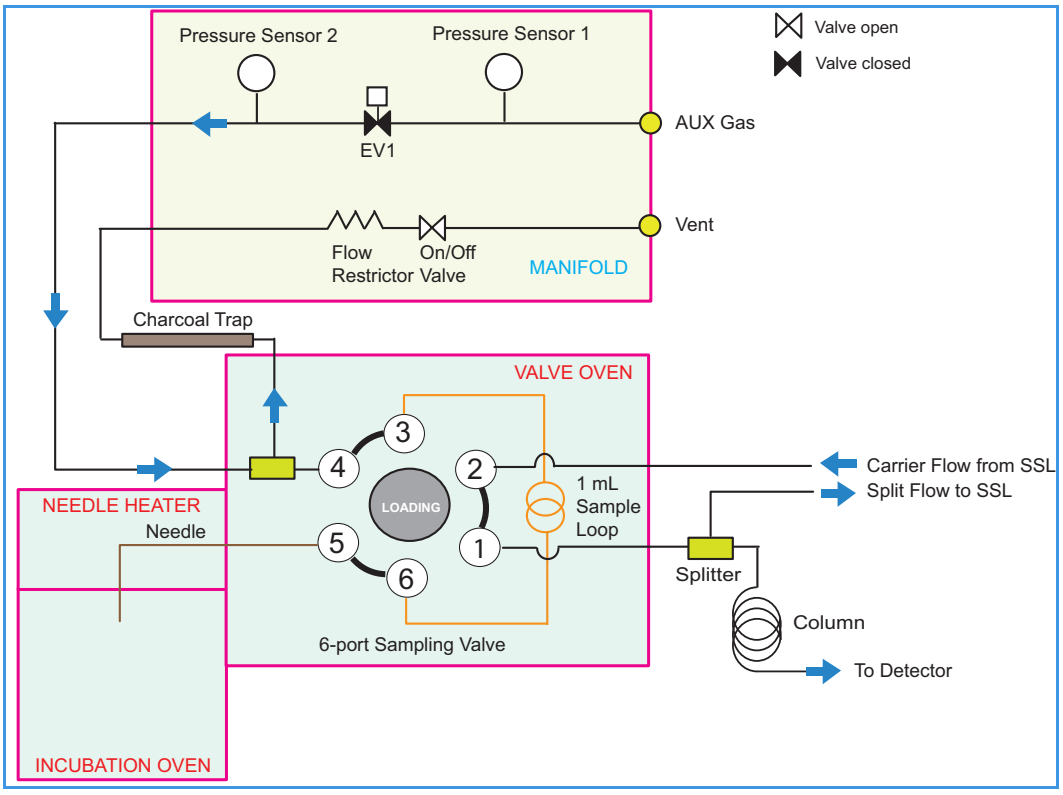
The operative phases are:

- "Stand-by" on page 36
- "Incubation" on page 38
- "Pressurization" on page 39
- "Leak Check" on page 40
- "Loop Filling" on page 41
- "Injection" on page 42
- "Purging" on page 44

Stand-by

The instrument is in stand-by condition before and after a sampling sequence, and between a sampling and the next one of the same sequence. See Figure 31.

Figure 31. Stand-by Phase

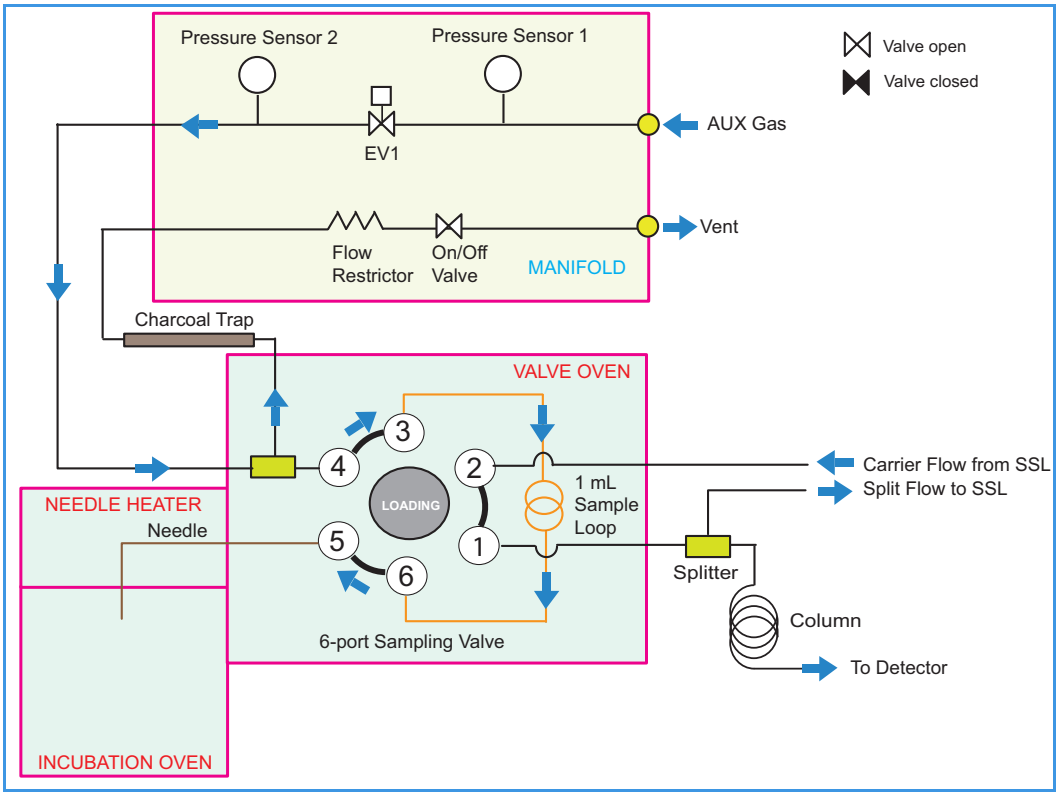


The sampling valve is in **Load** position.

The proportional valve EV1 is closed while the On/Off valve is open. The carrier gas flows to the GC through the sampling valve to feed the column.

If during the stand-by phase the parameter **Stand-by Purge** is active, EV1 and On/Off valve are open. The auxiliary gas flushes at a constant flow rate through the sampling valve, the sample loop, and the sampling needle to clean the circuit. See Figure 32.

Figure 32. Stand-by with Purge Phase

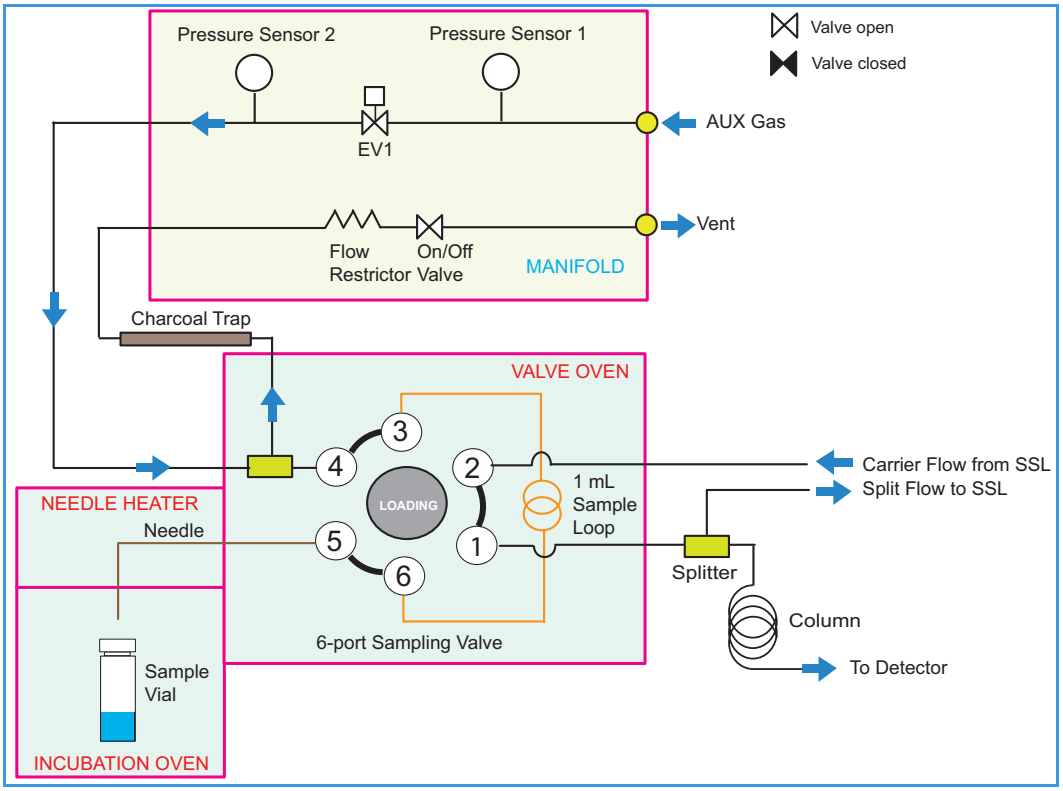


Note The flow rate used during **Stand-by**, if active, is the same as set for the **Purge** level in the method. See Table 2.

Incubation

The incubation phase retains the same sampling valve configuration as the **Stand-by** phase without purge or **Stand-by with purge** phase. See Figure 33.

Figure 33. Incubation Phase



During the incubation phase the vial remains in the incubation oven at a constant temperature.

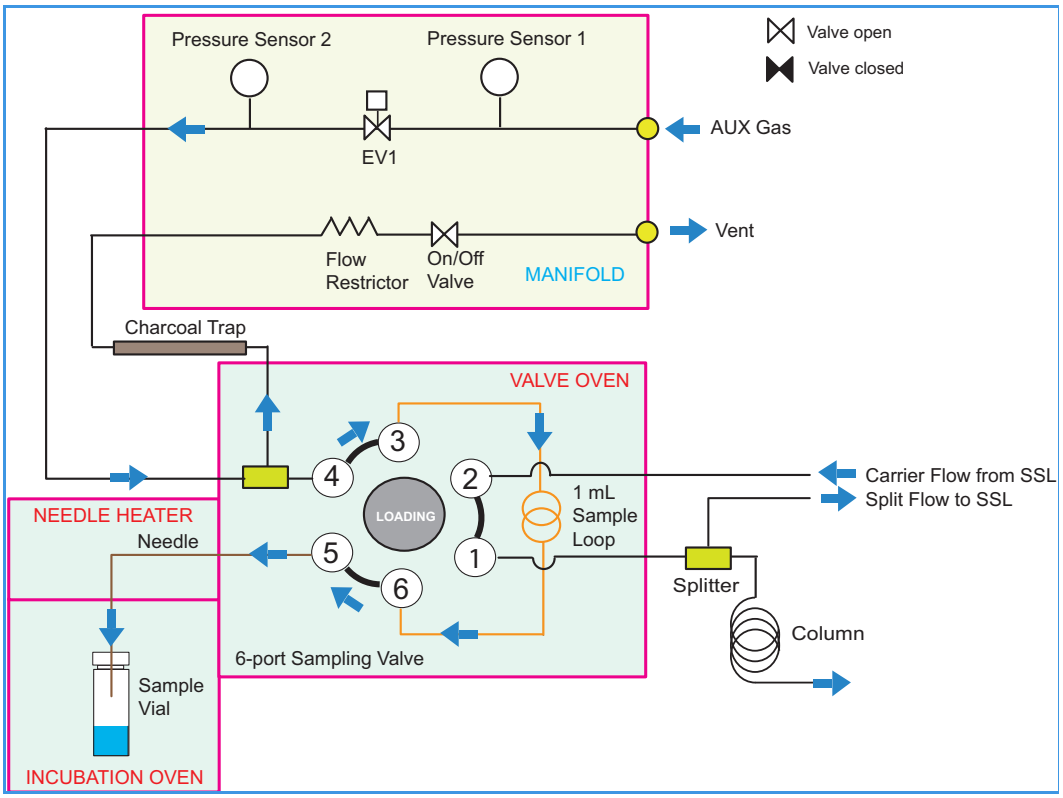
Note During the incubation phase it is possible to stir the sample in the vials choosing the appropriate vial shaking mode **Slow**, **Medium**, or **Fast**.

Pressurization

The pressurization phase starts at the end of the incubation time. If activated, the oven carousel stops the shaking.

The **Vial sampling lifter** goes up to a different position according to the type of vial used (10 mL or 20/22 mL). The sampling valve is in **Load** position, EV1 and On/Off valve are open. See Figure 34.

Figure 34. Pressurization Phase



Three types of pressurization can be chosen:

- **Pressurization at constant pressure** — The pressure P2 is controlled by EV1 in order to keep a default pressure rate increase until P2 reaches the Pressurization Pressure set.
- **Pressurization at defined pressure rate increase** — The pressure P2 is controlled by EV1 in order to keep a pressure rate increase set by the user until P2 reaches the pressurization pressure set.
- **Pressurization at defined pressurization time** — The pressure P2 is controlled by EV1 in order to keep a pressure rate increase defined by (Pressure set-Pressure initial)/Time, where the time is set by the operator, until P2 reaches the pressurization pressure set.

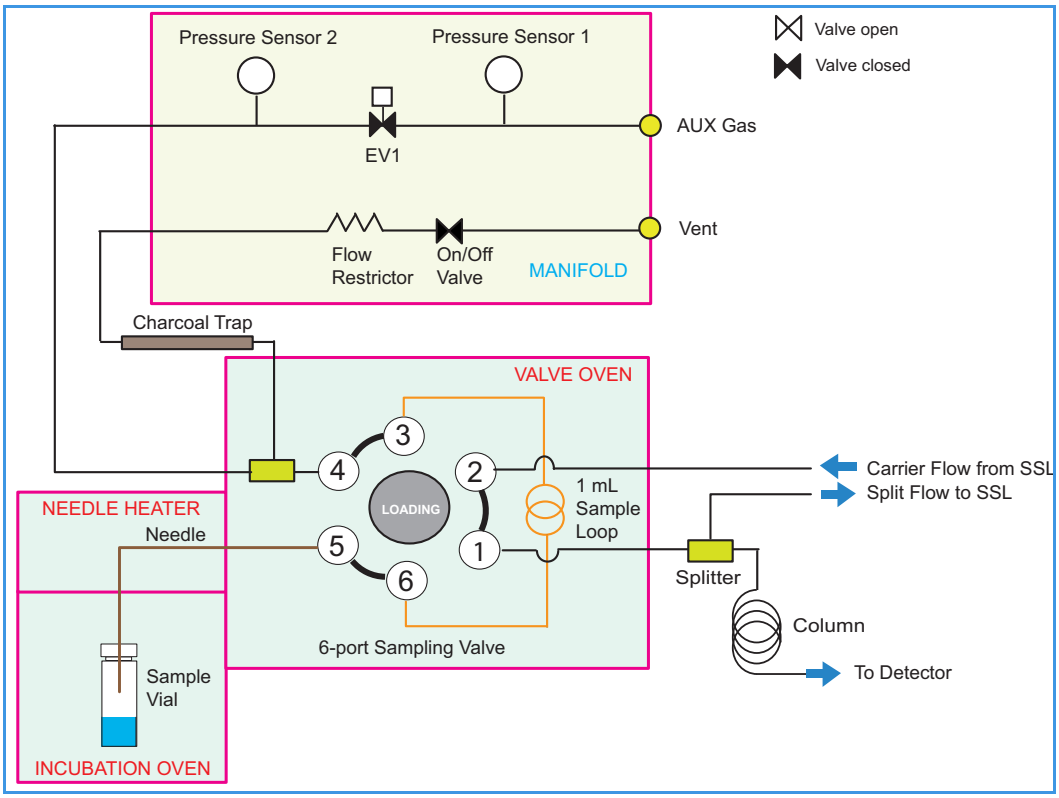
Wait the **Pressure Equilibration Time** set by the operator.

Leak Check

After the system executes the vial pressurization, EV1 and On/Off valve are closed.

The system monitors the P2 decay, calculates the decay rate and compares with a set value to assess if there is a leak in the vial. See Figure 35.

Figure 35. Leak Check Phase

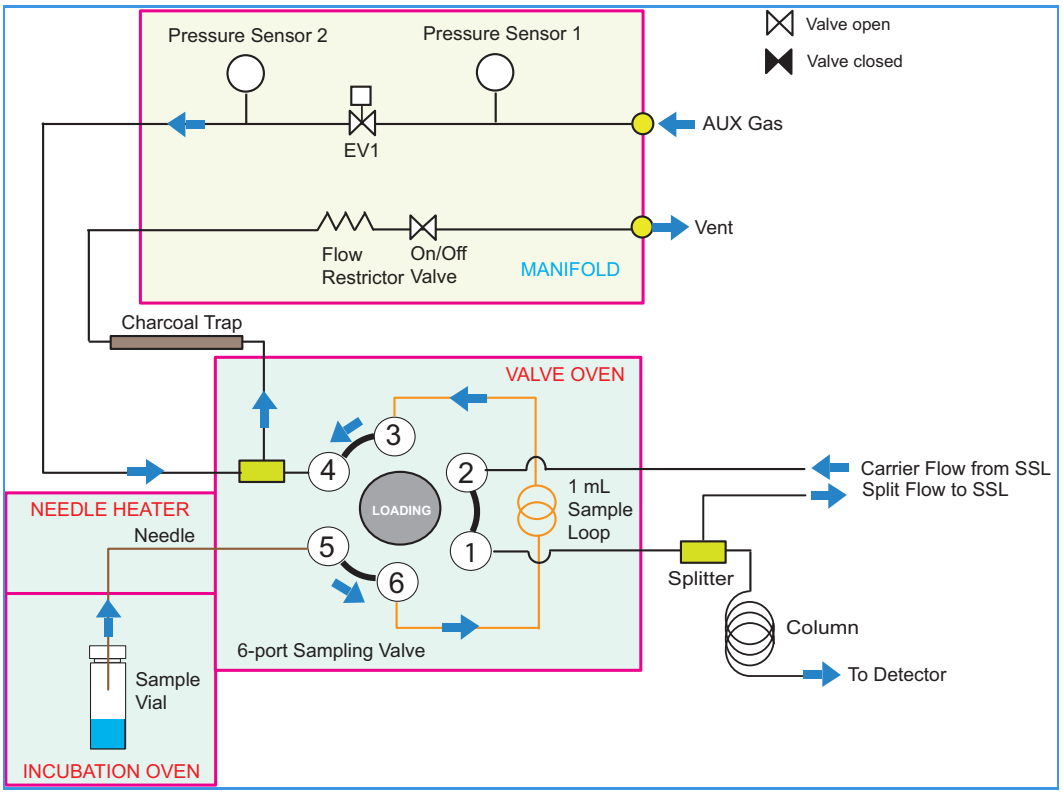


Loop Filling

The sampling valve is in **load** position. EV and On/Off valve are open allowing the pressurized sample to vent through the sample loop.

The filling stops when P2 reaches the set loop final pressure. Wait **Loop Equilibration Time** set by the operator. See Figure 36.

Figure 36. Loop Filling Phase



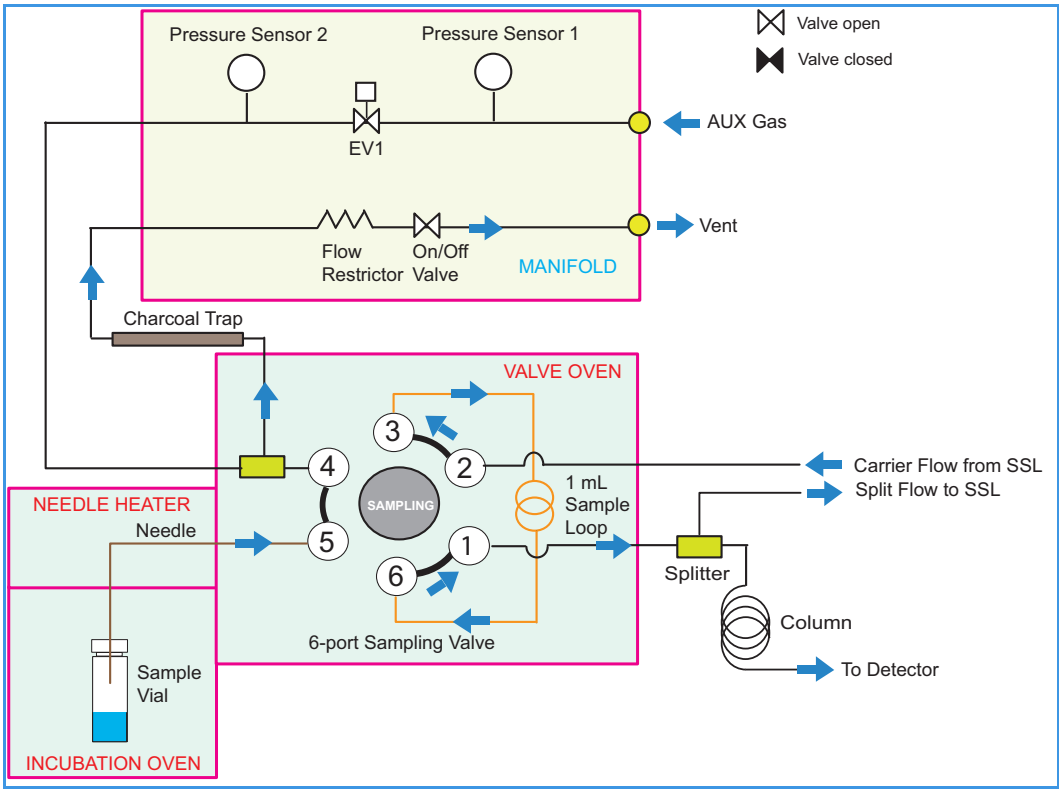
Injection

The sampling valve is in **Inject** position (Sampling). EV1 and On/Off valve are open.

The carrier gas flushes through the sampling valve and the sample loop moving the sample towards the analytical column.

The auxiliary gas flows through the rest of the circuit and goes to the vent. See Figure 37.

Figure 37. Injection Phase



The following injection phases can be chosen:

- **Single injection**
  - The sample valve is set to inject position.
  - Send Start signal to the GC.
  - Venting phase (only if vial venting option is selected).
  - The On/Off valve is open to vent the residual pressure of the vial.
  - The sampling valve is set to load position when injection time is expired.
  - The needle is removed from the vial.

- **Enrichment**
  - The sampling valve is set to **Inject** position.
  - The sampling valve is set to **Load** position when injection time is expired.
  - The vial is removed from the sampling needle insertion position.
  - Repeat for N of enrichments -1.
  - Wait for enrichment time (shaking ON).
    - Pressurization.
    - Sampling.
    - The multi-position valve is set to inject position.
    - The multi-position valve is set to load position when injection time is expired.
    - The needle is removed from the vial.
  - Last enrichment.
  - Send Start signal to the GC. The sampling valve is set to **load** position when injection time is expired.
  - Venting phase (only if vial venting option is selected).
    - The On/Off valve is open to vent the residual pressure of the vial.
    - The sampling valve is in a position depending on the injection time.
  - The needle is removed from the vial.
- **MHE** (Multiple Headspace Extraction)
  - Same as single injection, but venting is always selected.

Purging

The **Purging** is a post-injection phase that starts after the selected sampling phase is completed. This function allows to reduce the carryover.

The sampling valve is in **Load** position and the On/Off valve is open. The pressure P2 is controlled by EV1 to maintain the purge flow level. This phase ends the analytical sequence.

Table 2 lists the values of pressure and approximated purge flows according to the levels of the purge.

Table 2. Aux Pressure and Approximated Flow Values

Purge Level	Aux Pressure	Total Purge Flow	Vent Line Flow	Needle Flow
1	5 kPa	**10 mL/min**	3 mL/min	7 mL/min
2	10 kPa	**20 mL/min**	5 mL/min	15 mL/min
3	30 kPa	**75 mL/min**	20 mL/min	55 mL/min
4	50 kPa	**135 mL/min**	35 mL/min	100 mL/min
5	120 kPa	**400 mL/min**	100 mL/min	300 mL/min
Total purge = vent line flow + needle flow				

Note Flow rates might change slightly depending on operating temperatures.

Note The purge level set for the post-injection purging is also applied to the Stand-by purge, when selected. See Table 2. If purging is not necessary, consider not checking this option to reduce gas consumption during Stand-by.

3

Setting Up Through the TRACE 1610 User Interface

This chapter contains the instructions to configure your TriPlus 500 HS and to edit the parameters through the TRACE 1610 GC user interface (touch screen) or through the TRACE 1610 Virtual Touch Screen software.

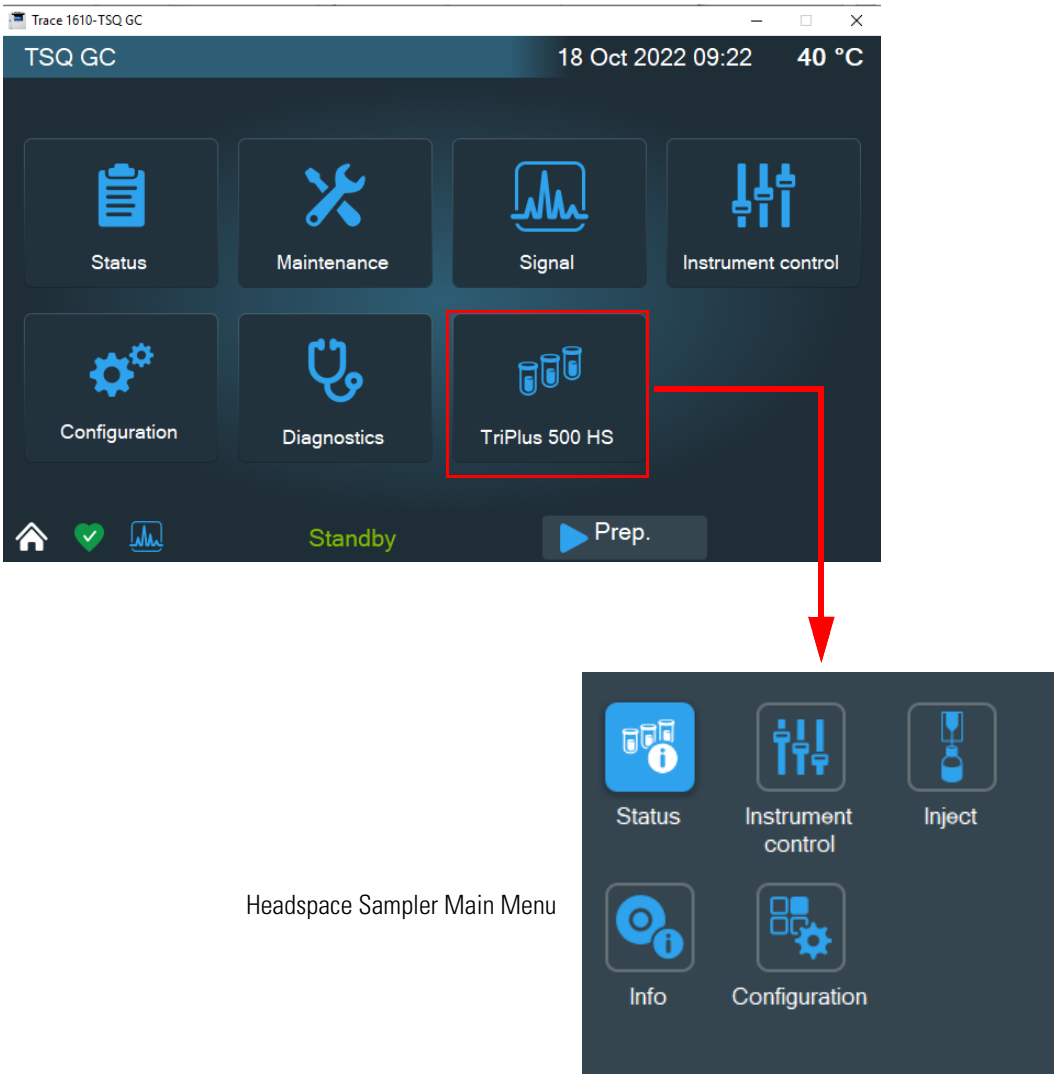
Contents

- User Interface Overview
- Configuring the Instrument
- Editing the Instrument Method
- Performing Sample Sequences
- Monitoring the Instrument Status
- Information Page

User Interface Overview

The HMI (Human-Machine Interface) of the TRACE 1610 GC recognizes the connected TriPlus 500 Headspace sampler. The relevant icon is displayed on the touch screen and must be selected to open the **Headspace Sampler Main Menu**. See Figure 38.

Figure 38. Touch screen Main Menu - Headspace Sampler Icon



Headspace Sampler Main Menu

Menu Icons

Each instrument's function is associated with an icon that opens the relevant menu:

- Select the **Configuration** icon to configure your system. See "Configuring the Instrument" on page 48.
- Select the **Instrument Control** icon to program and display method parameters for TriPlus 500 Headspace Sampler. See "Editing the Instrument Method" on page 50.

- Select the **Inject** icon to set the injection parameters and to perform the sequence of samples. See "Performing Sample Sequences" on page 53.
- Select the **Status** icon to monitor the status of the instrument. See "Monitoring the Instrument Status" on page 55.
- Select the **Info** icon to display the network parameters, software version, and the serial number of the system. See "Information Page" on page 56.

Data Entry Keys

A **keyboard** displays in the right pane of the **Instrument Control** menu when you select a parameter, an actual, or setpoint field.

The numeric keyboard includes numbers from 0–9. The numeric keyboard includes a decimal point, and a minus key. See Figure 39.

Figure 39. Data Entry Keyboard

- Use the **Enter** key to confirm the entry or the change made.
- Use the key to erase the last digit typed.
- Use the **On/Off** key to set the setpoint value to Off or On.
- Use the < key to close the keyboard.
- Use the **?** key to display the range of the selected parameter.

❖ How to enter or modify a parameter

To enter or modify a parameter, complete the following steps:

1. Press the name of parameter to enter or modify, for example **Incubator temperature**. The value in the setpoint field is highlighted.
2. Type the desired value, for example **250**, and press **Enter** to confirm the modification.

Note If the **Actual** or **Setpoint** field has been selected, before typing the desired value, erase the highlighted value by pressing **Clear**.

Shortcut Keys

Shortcut keys are located on the status/message bar of each menu and submenu.

The shortcut keys are:

Go to Main Menu— to return to the **Main** menu.

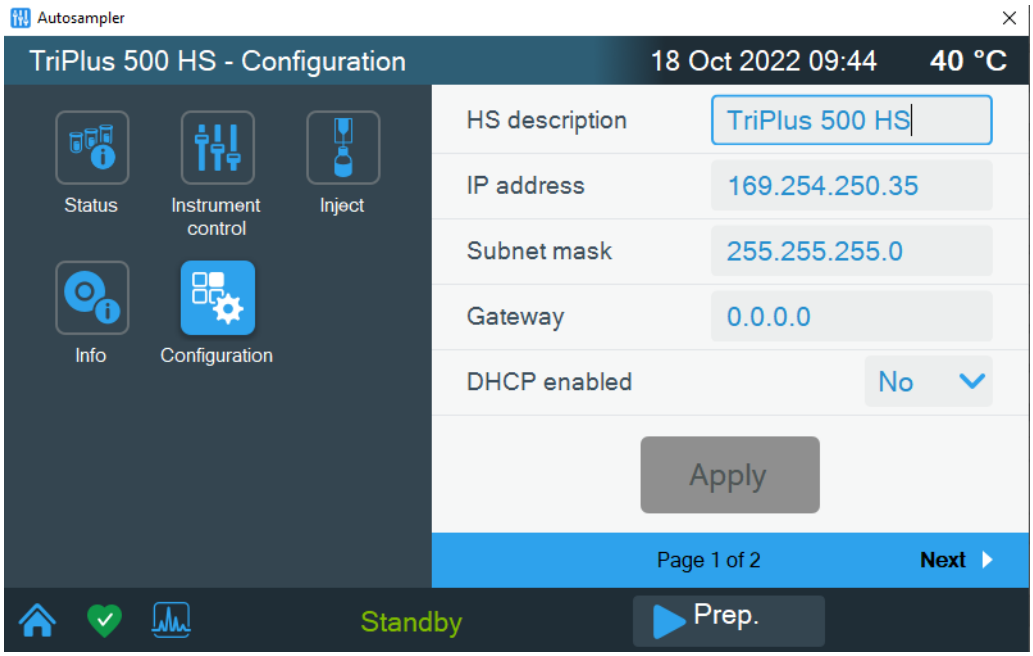
Go to Counter — to jump immediately to the **Counter** menu.

Go to Status — to jump immediately to the **Status** menu.

Configuring the Instrument

Press the **Configuration** icon on the touch screen Headspace Sampler Main Menu to configure the TriPlus 500 Headspace Sampler. See Figure 40.

Figure 40. Configuration Menu (Page 1)

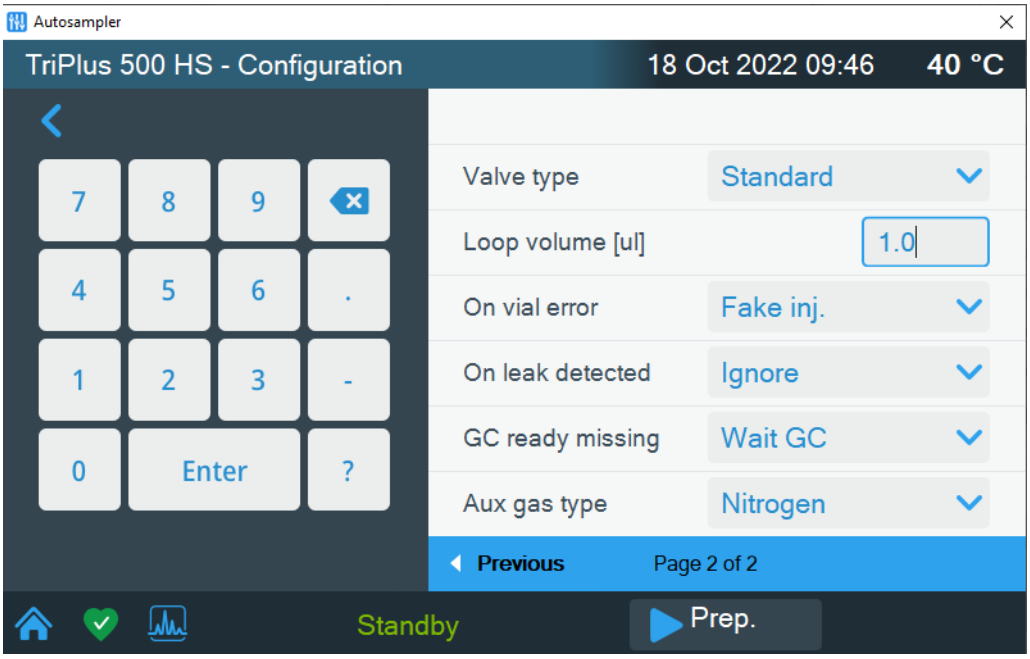


- **HS description** — Type a name you want to assign to your instrument.
- **IP address** — Displays the IP address for LAN control of the TriPlus 500 HS through the Thermo Scientific™ Chromeleon™ or TraceFinder™ Chromatography Data System (CDS). The IP address can be modified according to LAN requirements.

- **Subnet mask** — Displays the subnet mask address. It can be modified.
- **Gateway** — Displays the gateway address. It can be modified.
- **DHCP enabled** — Enables or disables the Dynamic Host Configuration Protocol (DHCP). Choose **On** to enable the function, or **Off** to disable it. The default setting is **Enabled**.

The second page of the Configuration menu. See Figure 41.

Figure 41. Configuration Menu (Page 2)



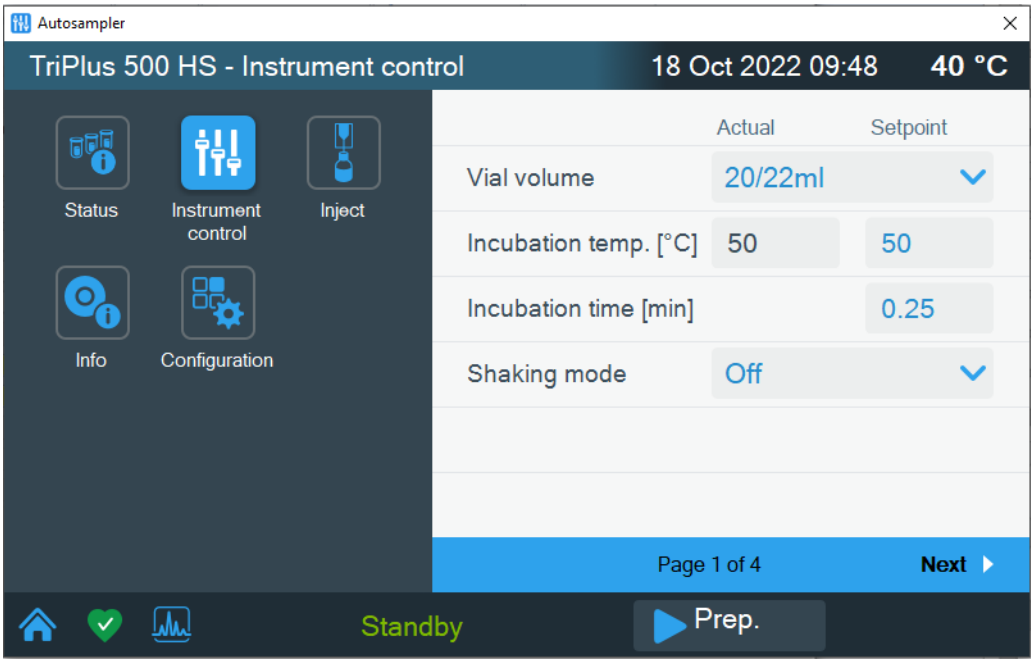
- **Valve type** — Specifies the type of sampling valve installed into your HS sampler. From the dropdown list choose one valve between **LT** (Low Temperature) and **HT** (High Temperature). **LT** is the standard valve on the instrument.
- **Loop volume** — Specifies the volume of the loop installed on the sampling valve. The standard loop volume is 1 mL.
- **On Vial Error** — From the dropdown list choose among the following actions in case of a vial error:
  - **Fake inj.**: wrong vials not processed.
  - **Stop**: complete current incubating vials and stop sequence.
  - **Abort**: stop current sample and sequence immediately.
- **On leak detected** — From the dropdown list choose among the following actions in case a vial leak is detected:
  - **Ignore**.
  - **Fake injection**: Injection Valve is not switched but vial processing is completed.
  - **Abort**: stop current sample and sequence immediately.

- **GC ready missing** — From the dropdown list choose between the following actions in case the GC Ready is missing:
  - **Abort**.
  - **Wait GC**.
- **Aux gas type** — From the dropdown list choose: **Helium**, **Nitrogen**, or **Argon**.

Editing the Instrument Method

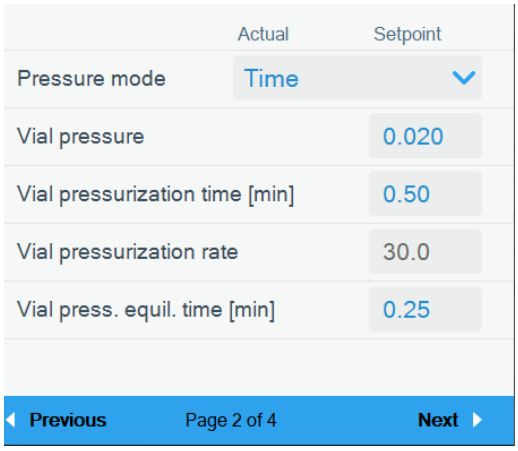
Press the **Instrument Control** icon on the touch screen Headspace Sampler Main Menu to program the parameters of the analytical method. See Figure 42.

Figure 42. Instrument Control Menu (Page 1)



The Instrument Control menu appears. See Figure 43 and Figure 44

Figure 43. Instrument Control Menu (Pages 2 and 3)



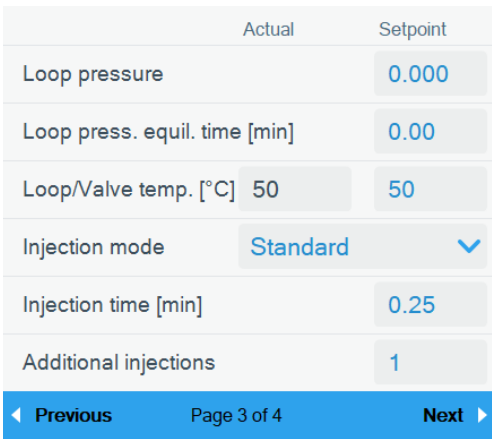
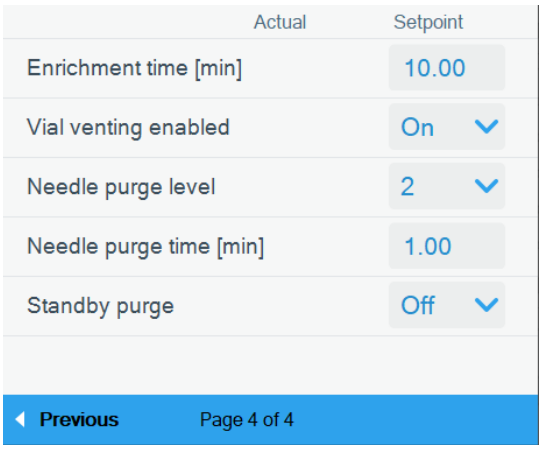


Figure 44. Instrument Control Menu (Page 4)



- **Vial Volume** — Defines the volume of the vial to incubate. From the dropdown list select **10 mL** or **20/22 mL**.
- **Incubator temp.** [°C]—The **Actual** box indicates the temperature of the incubator, while **Setpoint** box indicates the control of the temperature is set **On** or **Off**.
  - When the **Setpoint** box is set **Off**, the control of the temperature is disabled. The Actual box shows the room temperature.
  - When the **Setpoint** box is set **On**, the control of the temperature is enabled. Type in the **Actual** box the temperature of the incubation oven in the range **0–300 °C**.

> **Note** When the Actual box is green, the temperature set is **ready**. When the Actual box is red, the temperature set is **NOT ready**.

- **Incubation time** [**min**]— Defines the time during which the vial is conditioned in the oven at a fixed temperature before the pressurization phase starts. Set a value in the range **0.50–999.00 minutes**.

- **Shaking mode** — Selects the desired shaking mode. Choose one shaking mode from the dropdown list among **Off**, **Low**, **Medium**, or **Fast**. If a shaking option is enabled, the incubation carousel will keep moving during the incubation in order to stir the samples.
- **Pressure mode** — Select how the vial pressurization must be performed. Choose one mode from the dropdown list among **Pressure, Time**, and **Rate.** The selected mode activates the associated parameters.
- **Vial pressure** — Defines the target pressure level at which the vial is pressurized. Set a value in the range **0.00–500.00 kPa**.
- **Vial pressurization time** [**min**]— Displays when the **Press mode** is set to **Time**. Defines the duration of the pressurization. Set a value in the range **0–10 minutes**.
- **Vial pressurization rate** — Displays when the **Press mode** is set to **Rate**. Defines the rate of the pressure change in the range **30–3000 kPa/min**.
- **Vial press. equil. time** [**min**]— Defines the time for equilibrating the vial pressure after the pressurization step. Set a value in the range **0.00–5.00 minutes**.
- **Loop Pressure** — Defines the pressure target that the loop must achieve. Set a value in the range **0.00–500.00 kPa**.
- **Loop press. equil. time** [**min**]— Defines the time for equilibrating the loop pressure after the pressurization step. Set a value in the range **0.00–5.00 minutes**.
- **Loop/Valve temp**. [**°C**] — The **Actual** box indicates the temperature of the loop/valve path. The **Setpoint** box indicates the control of the temperature is set **On** or **Off**.
  - When **Setpoint** box is set to **Off**, the control of the temperature is disabled. The Actual box shows the room temperature.
  - When **Setpoint** box is set to **On**, the control of the temperature is enabled. Type in the **Actual** box the temperature of the incubation oven from **0–225 °C** if a low-temperature valve is installed or **150–300 °C** if a high-temperature valve is installed.

    Note When the Actual box is **green**, it means that the temperature set is **ready**. When the Actual box is **red**, it means that the temperature set is **NOT ready**.
- **Injection mode** — Select the type of injection. Choose a mode from the dropdown list among **Standard, Enrichment**, and **MHE.**
  - **Standard** — Equilibrates the vial, fills the sample loop (single extraction), then starts a run while injecting the sample into the Back SSL injector of the GC. The loading time of the vials into the incubation oven is automatically calculated to guarantee the same equilibration time for all the samples, and the optimization of the analysis times. According to the equilibration time and GC time, more vials can be contemporaneously present inside the incubation oven.
  - **Enrichment** — The sample from the same sample vial is injected the number of time selected. The parameters **Additional injection** and **Enrichment time** are enabled.

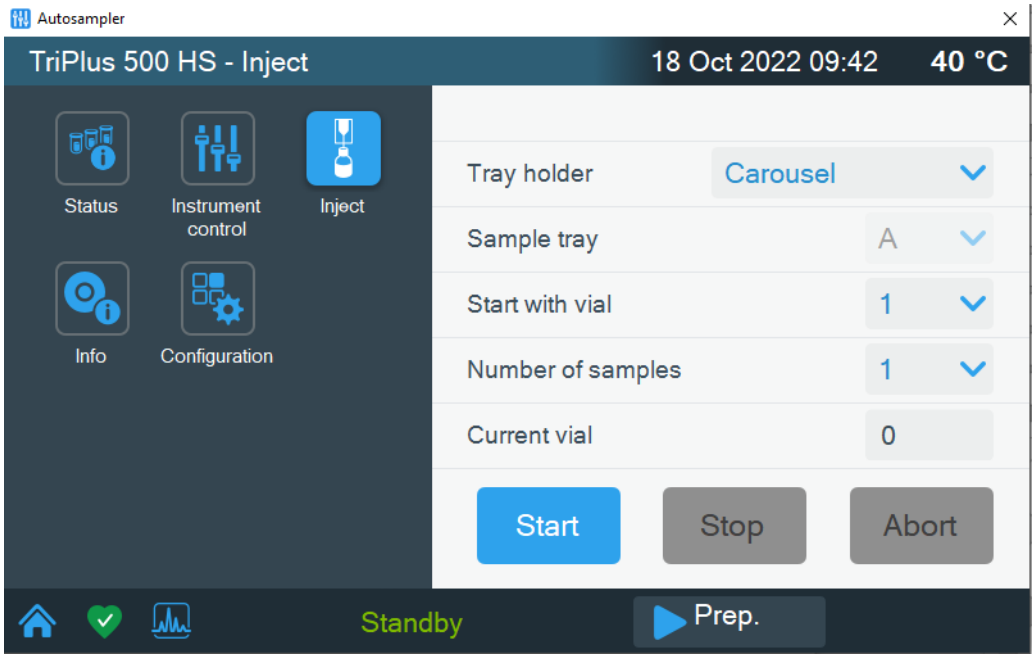
  - **MHE** — Multiple Headspace Extraction. Each vial is pressurized, sampled and vented multiple times. Each vial is incubated first for a time equal to the equilibration time set in the method, while the incubation time of the subsequent samplings corresponds to the longest time set in the method (equilibration time or GC time). At each sampling an analysis is performed.

- **Injection time** [**min**]— Defines the time of the sample transfer to the GC. Set a value in the range **0.00–999.00 minutes**.
- **Additional injections** — Enabled when **Injection Mode** is set to **Enrichment**. Specifies the number of samplings to be carried out from the same sample vial. Set a value in the range **1–100**.
- **Enrichment time** [**min**]— Enabled when **Injection Mode** is set to **Enrichment**. Specifies the time between one enrichment of and the next. Set a value in the range **0.5-–999.00 minutes**.
- **Vial venting enabled** — Enables or disables the venting of the vial. Choose **On** to enable the function, or **Off** to disable it.
- **Needle purge level** — Specifies a target flow rate of the purge gas for cleaning the sampling valve and the needle. From the dropdown list select a value from **1** to **5**. The default value is **2**.
- **Needle purge time** [**min**]— Defines the duration of the purging phase. Set a value in the range **0.00–999.00 minutes**.
- **Standby purge** — The purge gas flows continuously in the system. Choose **On** to enable the function, or **Off** to disable it.

Performing Sample Sequences

Press the **Inject** icon on the touch screen Headspace Sampler Main Menu to set the injection parameters and to perform the sample sequence. See Figure 45.

Figure 45. Inject Menu



The **Inject** menu appears.

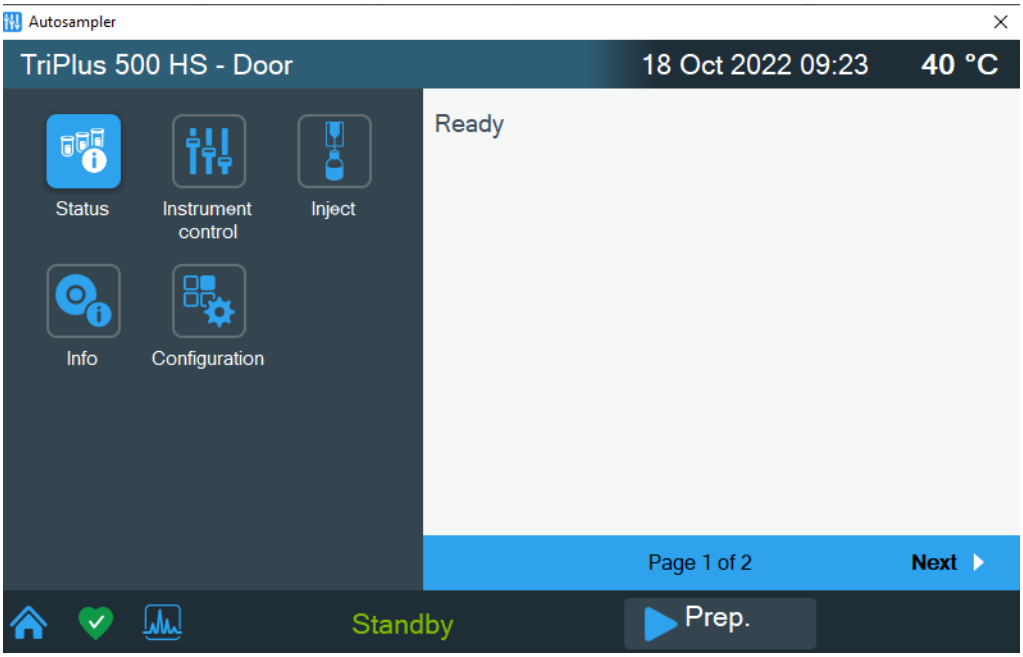
- **Tray Holder** — Define the tray holder in use.
  - Select **Carousel** when the vials are inserted in the 12-seal rotating carousel of the TriPlus 500 Headspace Sampler.
  - Select **Loader** when the vials are inserted in the 40-position sample trays installed on the top cover of the TriPlus 500 Headspace Sampler.
- **Sample tray** — Enabled only when **Tray Holder** is selected to **Loader**. Select among the sample trays **A**, **B**, or **C**.
- **Start with vial** — Defines the start vial of the sequence.
  - Select the position of the start vial from **1** to **12** when **Tray Holder** is selected to **HS**.
  - Select the position of the start vial from **1** to **40** when **Tray Holder** is selected to **Loader**.
- **Number of samples** — Defines the number of samples to inject.
  - Select the number of samples from **1** to **12** when **Tray Holder** is selected to **HS**.
  - Select the number of samples from **1** to **40** when **Tray Holder** is selected to **Loader**.
- **Current vial** — Displays the current vial in execution.
- **Start** — Press this button to begin the sample sequence.
- **Stop** — Press this button to end the sample sequence.
- **Abort** — Press this button to abort the sample sequence.

Monitoring the Instrument Status

Press the **Status** icon on the touch screen Headspace Sampler Main Menu to monitor the status of the TriPlus 500 Headspace Sampler.

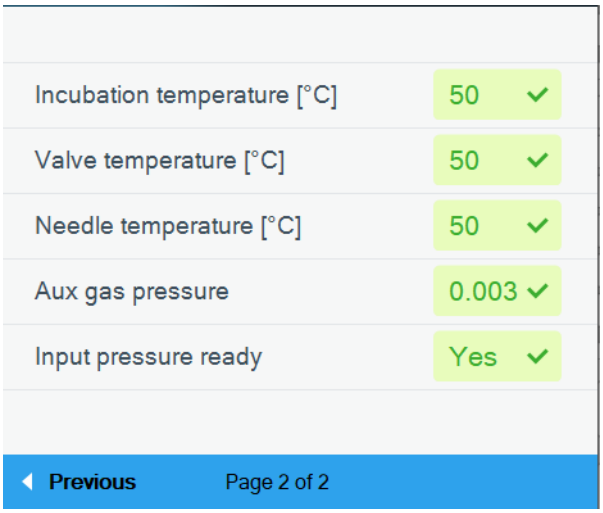
The status can be **Not Ready**, **Ready**, **Running**, or **Error**. See Figure 46.

Figure 46. Status Menu (Page 1)



The **Status** menu appears. See the example of Figure 47.

Figure 47. Status Menu (Page 2)



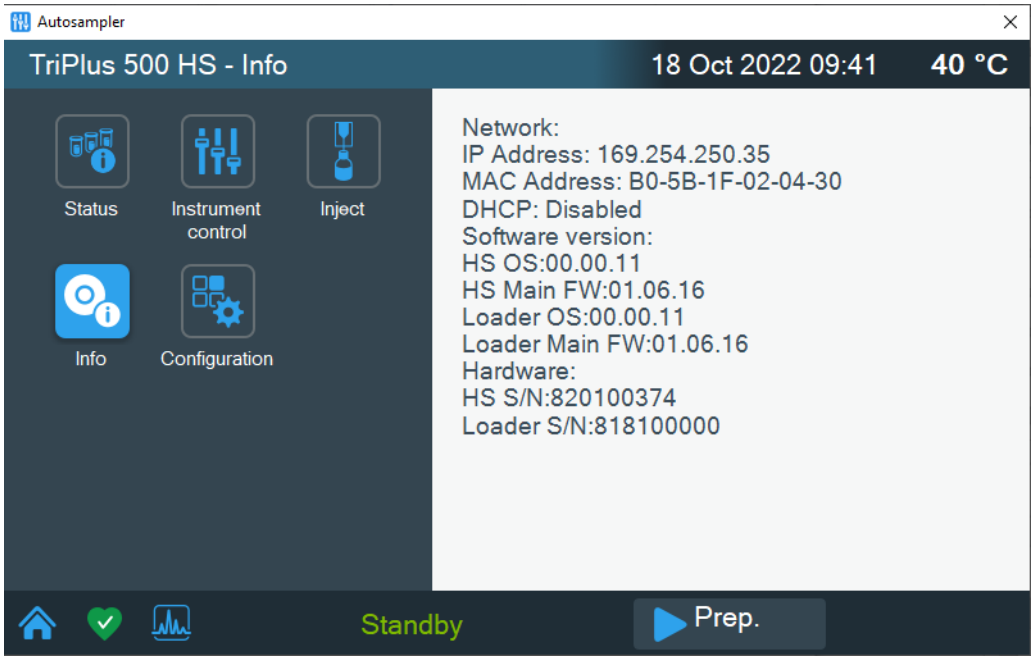
- **Incubator temperature** [°C] —Indicates the temperature of the incubator,
- **Valve temperature** [°C] —Displays the temperature of the valve.

- **Needle temperature** [°C]—Displays the temperature of the needle.
- **Aux gas pressure**—Shows the aux gas pressure.
- **Input pressure ready**—Shows status of the input pressure.

Information Page

Press the **Info** icon on the touch screen Headspace Sampler Main Menu to display information about the TriPlus 500 Headspace Sampler. See Figure 48.

Figure 48. Info Page



The **Information Page** appears.

Network — Indicates the network connection specifications (IP address, MAC Address, and DHCP).

Software — Indicates the software and firmware version of the TriPlus 500 HS and the Vial Loader when present.

Hardware — Indicates the serial number of the TriPlus 500 HS and the serial number of the Vial Loader when present.

4

Setting Up Through Chromeleon CDS

This chapter contains the instructions to configure your TriPlus 500 HS and to edit the parameters through the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS).

Contents

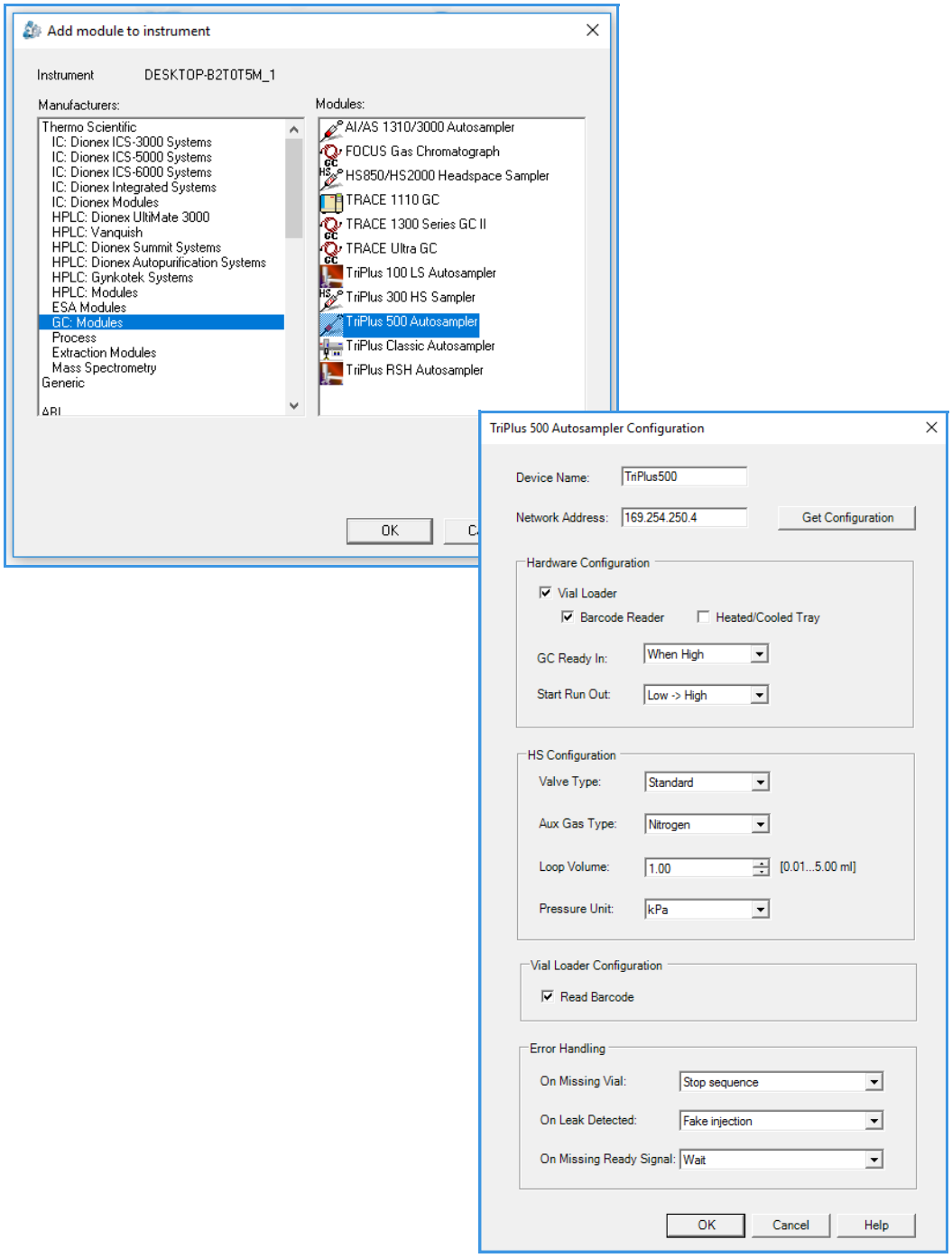
- Configuring the TriPlus 500 HS Through Chromeleon CDS
- Editing Method Parameters Through Chromeleon CDS
- Chromeleon HS Sampler Control Panel
- Vials Sequence
- Vial Leak Checking
- Barcode Reading

Configuring the TriPlus 500 HS Through Chromeleon CDS

Run the Chromeleon Configuration Manager, and open the TriPlus 500 HS **Configuration** window.

Figure 49 shows the Configuration Manager dialog window of Chromeleon™ CDS for the TriPlus 500 HS.

Figure 49. Chromeleon™ Configuration Manager Dialog Window for the TriPlus 500 HS



The Configuration dialog window comprises the following settings and areas:

- "Hardware Configuration" on page 61
- "HS Configuration" on page 62
- "Error Handling" on page 63

Device Name — Displays the name used to identify the autosampler in Chromeleon. In general, accept the default name (e.g. TriPlus500). If you enter a different name, you may need to re-link controls or edit the device name in Instrument Methods. For details, refer to Naming Devices in the Instrument Configuration Manager.

Network Address — Enter the network address to allow LAN control of the autosampler through Chromeleon.

The network address is made up of four integers written in the format xxx.xxx.xxx.xxx, for example: 192.168.127.10

The TriPlus 500 Autosampler is shipped with a default network address, which might not match the needs of the LAN where the autosampler is installed. To change the default values, contact your LAN administrator and ask for the network address to be assigned.

Get Configuration — Click this button to download the hardware configuration from the autosampler to Chromeleon.

When you click the **Get Configuration** button, Chromeleon attempts to connect to the autosampler and communicate with it using the specified Network Address:

- If Chromeleon successfully communicates with the autosampler and downloads its hardware configuration, the parameters in the configuration dialog box are automatically populated with the downloaded settings.
- If Chromeleon fails to communicate with the autosampler, the following error message is displayed: The driver failed to connect to the sampler.
- If Chromeleon communicates with the autosampler but fails to get the configuration, the following error message is displayed: The driver failed to query the configuration.

Hardware Configuration

In case of off-line editing, in this area you set the hardware configuration of your TriPlus 500 HS.

Vial Loader— Select the check box to configure the **Vial Loader** if it is installed on the autosampler.

Barcode Reader — (Available only if the Vial Loader check box is selected.) Select the check box to configure the **Barcode Reader** if it is installed on the autosampler.

Heated/Cooled Tray — (Available only if the Vial Loader check box is selected.) Select the check box to configure the **Heated/Cooled Tray** if it is installed on the autosampler.

GC Ready In — To allow other devices to run properly, specify how the signal will change to indicate to the autosampler that the GC is ready (the GC Ready signal). Select one of the following options from the list:

- **When Low** — Signals to the autosampler that the GC is ready when the signal is low.
- **When High** — Signals to the autosampler that the GC is ready when the signal is high.

Start Run Out — To allow other devices to run properly, specify how the signal will change to indicate to the GC that a run has started. Select one of the following options from the list:

- **Low -> High** — The signal should change from low to high to indicate to the GC that a run has started.
- **High -> Low** — The signal should change from high to low to indicate to the GC that a run has started.

HS Configuration

In this area you set the type of sampling valve, the volume of the sample loop, the pressure units and the type of gas used for pressurizing the HS sampler.

Valve Type — Specify the type of sampling valve installed on the autosampler. Select one of the following valve types from the list:

- **Standard**
- **High Temperature**.

Aux Gas Type — Specify the type of auxiliary gas in use. Select one of the following gas types from the list:

- **Helium**
- **Nitrogen**
- **Argon**.

Loop Volume — Enter the volume of the sample loop installed on the sampling valve. The available range is **0.01** to **5.00 mL**.

Pressure Unit — Specify the unit of measurement for pressure. Select on of the following units from the list: **kPa**, **bar**, or **psi**.

Read Barcode — Select the check box to enable reading of sample barcodes.

Error Handling

In this area you configure how to treat vial errors. By default a recoverable error such as vial missing, wrong vial, or leak detection, is treated with a blank injection. Nevertheless, there are cases where the instrument stops injecting samples and needs to inform the user that it has stopped. In this case, users must select instrument actions in case of configuration errors. The three selections are:

On Missing Vial — Specify the action to occur if a vial is missing or is the wrong size. Select one of the following options from the list:

- **Fake injection**— A fake injection (nothing is injected into the inlet) is performed.
- **Stop sequence** — Any current vial incubation is completed, and then the sequence is stopped.
- **Abort sequence** — The current sample and sequence are stopped immediately.

On Leak Detected — Specify the action to occur if a leak is detected. Select one of the following options from the list:

- **Ignore and inject** — The leak is ignored and the sample is injected as normal.
- **Fake injection** — A fake injection (nothing is injected into the inlet) is performed.
- **Abort**—The current sample and sequence are stopped immediately.
- **Abort after 3 consecutive leaks** — The current sample and sequence are stopped after three leaks in succession.

On Missing Ready Signal — Specify the action to occur if there is no GC Ready signal to indicate to the autosampler that the GC is ready. Select one of the following options from the list:

- **Abort sequence after 5 seconds** — The sequence is aborted after 5 seconds.
- **Wait** — The system waits until the GC is ready.

The Configuration dialog window includes the following buttons:

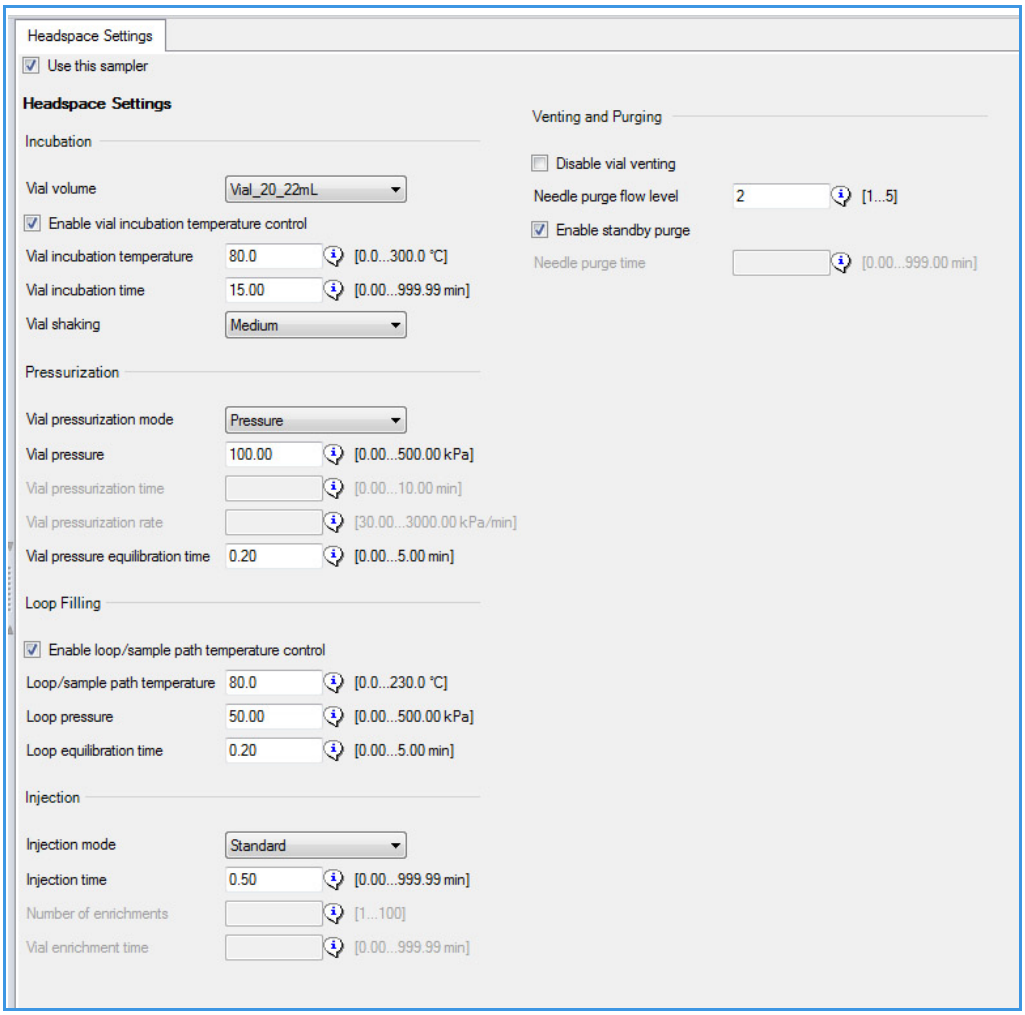
- **Ok** — Closes the dialog window and confirms your selections.
- **Cancel** — Clears all modifications and restores previous settings.
- **Help** — Opens help instruction.

Editing Method Parameters Through Chromeleon CDS

This section provides instruction for creating and saving an instrument method for the TriPlus 500 HS.

Figure 50 shows the **Headspace Settings** page of the Chromeleon Chromatography Data System (CDS). Select the **Use this sampler** check box.

Figure 50. Chromeleon™ Headspace Setting Page



The Headspace Settings page contains the following areas:

- "Incubation" on page 65
- "Pressurization" on page 65
- "Loop Filling" on page 66
- "Injection" on page 66
- "Venting and Purging" on page 67

Incubation

In this area you set all the temperatures for the sample incubation.

Vial Volume — Defines the volume of the vial to incubate: **10 mL** or **20-22 mL**.

Enable vial incubation temperature control — Select this check box to enable the temperature control of the headspace incubation oven.

Vial incubation temperature — (Available when **Enable vial incubation temperature control** is selected.) Enter a value for the vial incubation temperature. Set a value in the range 0.0–300.0 °C.

Vial incubation time — Defines the time during which the vial is conditioned in the oven at a fixed temperature before the pressurization starts. Set a value in the range 0.00–999.99 min.

Vial shaking: — Select the desired shaking mode from the drop-down list. The following modes are available: **Off**, **Slow**, **Medium**, and **Fast**. If a shaking mode is enabled, the incubation carousel will keep moving during the incubation in order to stir the samples.

Pressurization

In this area set the pressurization parameters:

Vial pressurization mode — Defines how vial pressurization will be performed. Select the desired mode from the dropdown list. The following modes are available: **Pressure, Time,** and **Rate.** The selected mode enables the associated parameters.

Vial pressure — Defines the target pressure level at which the vial is pressurized. Set a value in the range 0.00–500.00 kPa.

Vial pressurization time — (Available when **Vial pressurization mode** is set to **Time.**) Defines the duration of the pressurization. Set a value in the range 0.00–10.00 min.

Vial pressurization rate — (Available when **Vial Pressurization Mode** is set to **Rate.**) Defines the rate of the pressure change. Set a value in the range 30.00–3000.00 kPa/min.

Vial pressure equilibration time — Defines the time for equilibrating the vial pressure after the pressurization step. Set a value in the range 0.00–5.00 min.

Loop Filling

Enable loop/sample path temperature control — Select this check box to enable the temperature control of the loop/sample path.

Loop/sample path temperature — (Available when **Enable loop/sample path temperature control** is selected.) Enter the temperature of the loop/sample path. For the standard valve, set a value in the range **0–225 °C.** For the high temperature valve, set a value in the range **150–300 °C**.

Loop pressure — Defines the pressure target that the loop must achieve. Set a value in the range 0.00–500.00 kPa.

Loop equilibration time — Defines the time for equilibrating the loop pressure after the pressurization step. Set a value in the range 0.00–5.00 min.

Injection

In this area set the injection parameters:

Injection mode — Select the type of injection. The following injection modes are available**:**

- **Standard** — Incubates the vial, fills the sample loop, then starts a run while injecting the sample into the GC. Vial venting is enabled by default and can be turned off using the **Disable vial venting** option (see further below). The loading time of the vials into the incubation oven is automatically calculated to guarantee the same equilibration time for all the samples, and the optimization of the analysis times. Depending on the equilibration time and GC time, more vials can be present at the same time inside the incubation oven.
- **Enrichment** — Incubates the vial, fills the sample loop, injects into the GC as many times as selected, then starts the run. The vial remains in the incubation oven between one extraction and the next. Set the time between two consecutive extractions in the **Vial Enrichment Time** box. **Number of Enrichments** and **Enrichment Time** are enabled.
- **MHE** — Multiple Headspace Extraction. Each vial is pressurized, sampled, and vented multiple times. The incubation time for all the extractions is equal to the incubation time set in the method. At each sampling an analysis is performed. In MHE mode, vial venting cannot be turned off.

Injection time — Defines the time of the sample transfer to the GC. Set a value in the range 0.00–999.99 min.

Number of enrichments — (Available when **Injection Mode** is set to **Enrichment**). Specifies the number of samplings to be carried out from the same sample vial. Set a value in the range 1–100.

Vial enrichment time — (Available when **Injection mode** is set to **Enrichment**). Specifies the time between one enrichment and the next. Set a value in the range 0.00–999.99 min.

Venting and Purging

In this area set the venting and purging parameters.

Disable vial venting — (Available when **Injection mode** is set to **Standard**). Select this check box to disable the vial venting performed after sampling. Vial venting is enabled or disabled depending on the injection mode.

Note Vial venting cannot be disabled in **MHE** injection mode.

Needle purge flow level — Specifies a target flow rate of the purge gas for cleaning the sampling valve and the needle. Set a value from 1 to 5. The default value is **2**.

Enable standby purge — Select the check box if you want the purge gas to flow continuously in the system.

Needle purge time — Defines the duration of the purging phase after sampling. Set a value in the range 0.00–999.99 min.

Advanced

Enable heated/cooled tray temperature control— Select this check box to enable temperature control of the sample tray.

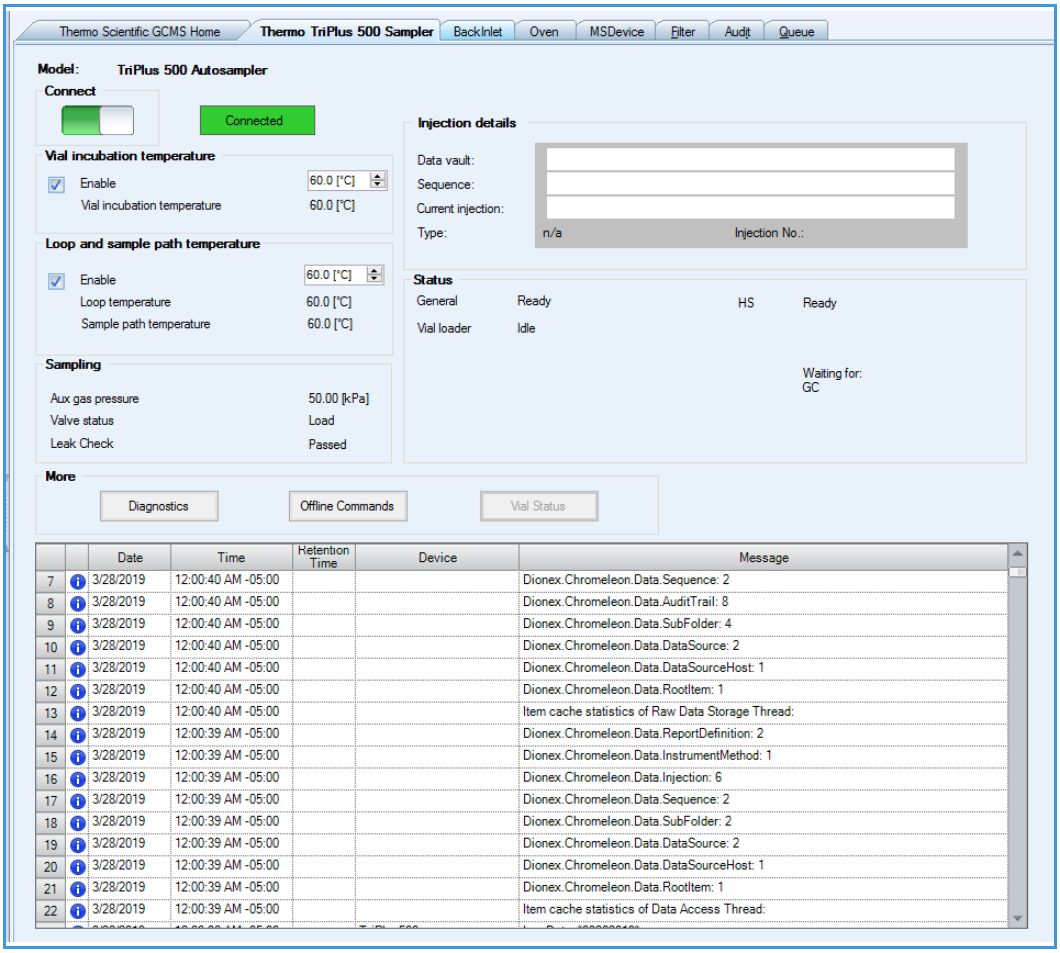
Heated/cooled tray temperature — (Available when **Enable heated/cooled tray temperature control** is selected.) Specify the tray temperature. Set a value in the range 4.0–70.0 °C.

Chromeleon HS Sampler Control Panel

You can check the current status of the TriPlus 500 HS using the HS Sampler Control Panel.

Figure 51 shows an example of the **Status** window for the HS Sampler.

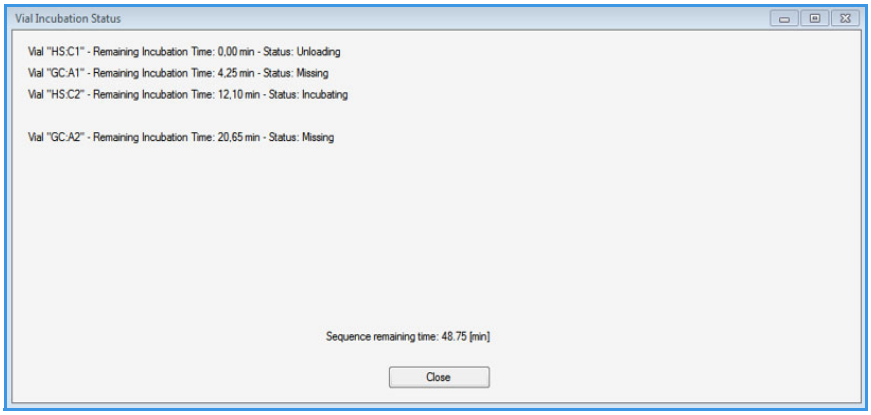
Figure 51. Chromeleon HS Sampler Status Window



The **More** area of the Status window includes the following buttons:

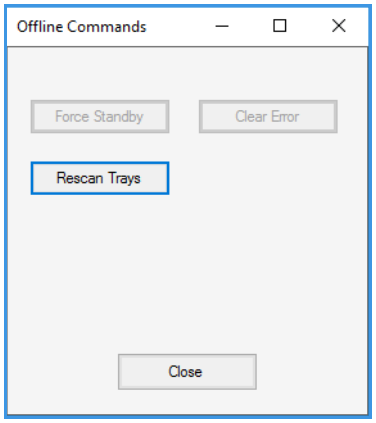
- **Vial Status** — Displays the incubation status of the vials. See the example in Figure 52.

Figure 52. Vial Incubation Status Page



- **Offline Commands**

Figure 53. Offline Commands Page



This window includes the following commands:

– **Force Standby** — Forces TriPlus 500 HS in standby condition.

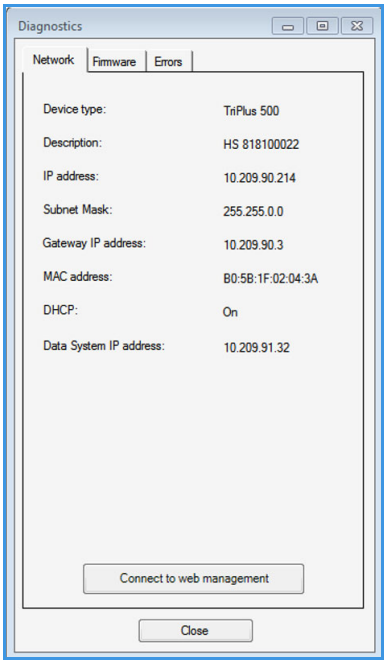
– **Clear Error** — Clears a possible error that has occurred.

– **Rescan Trays** — Rescans the trays present in the system.

– **Close** — Exits Offline Commands window.

- **Diagnostics** — By selecting this button the following page appears and allows you to find diagnostics information. See the example in Figure 54.

Figure 54. Diagnostics Page

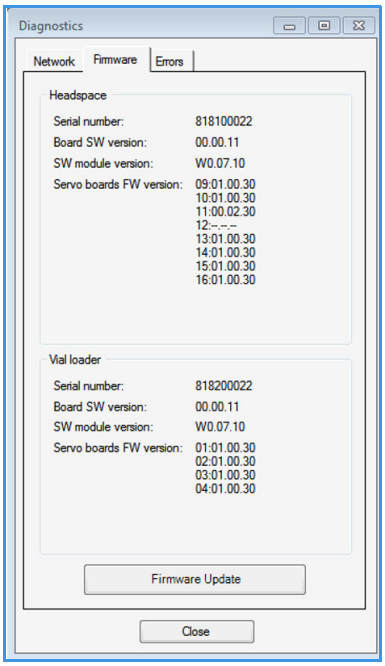


The **Diagnostics** page includes the following tabs:

– **Network** — Indicates the network connection specifications (IP address, Subnet mask, Gateway IP address, MAC address, etc).

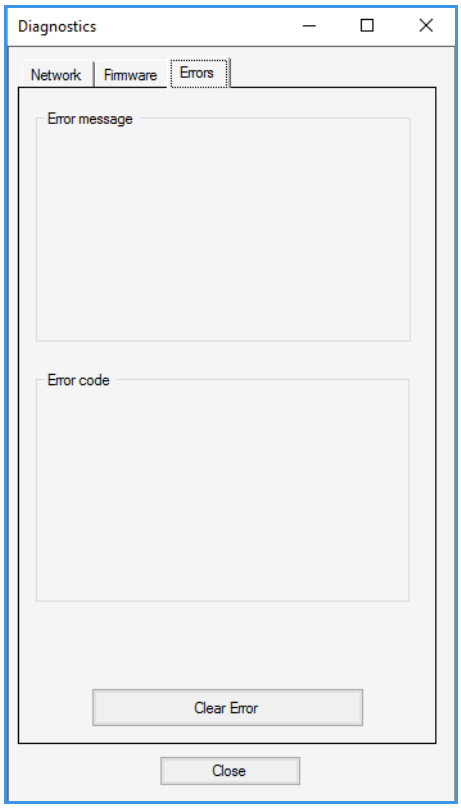
– **Firmware** — Indicates Serial number, Firmware version, and other information of the HS sampler and of the Vial Loader when present. See the example of Figure 55.

Figure 55. Firmware Info Tab



– **Errors** — Indicates the diagnostic of the possible errors. See the example in Figure 56.

Figure 56. Errors Tab



Note For details please refer to the Chromeleon documentation.

Vials Sequence

The correct sequence vial syntax to be used to set up a vial sequence, depending on the system configuration, is described in the section "" on page 22. This section shows an example of vials sequences according to the configuration of TriPlus 500 HS: **TriPlus 500 HS-12**, **TriPlus 500 HS-120**, and **TriPlus 500 HS-120+120**.
See Figure 57, Figure 58, and Figure 59.

Figure 57. TriPlus 500 HS-12 Vials Sequence Page

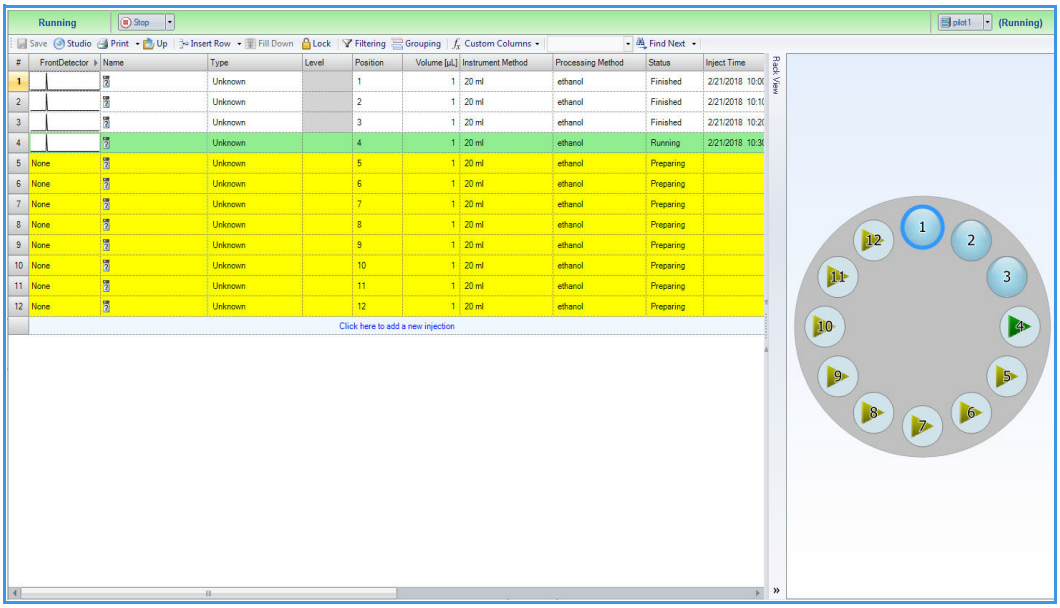


Figure 58. TriPlus 500 HS-120 Vials Sequence Page

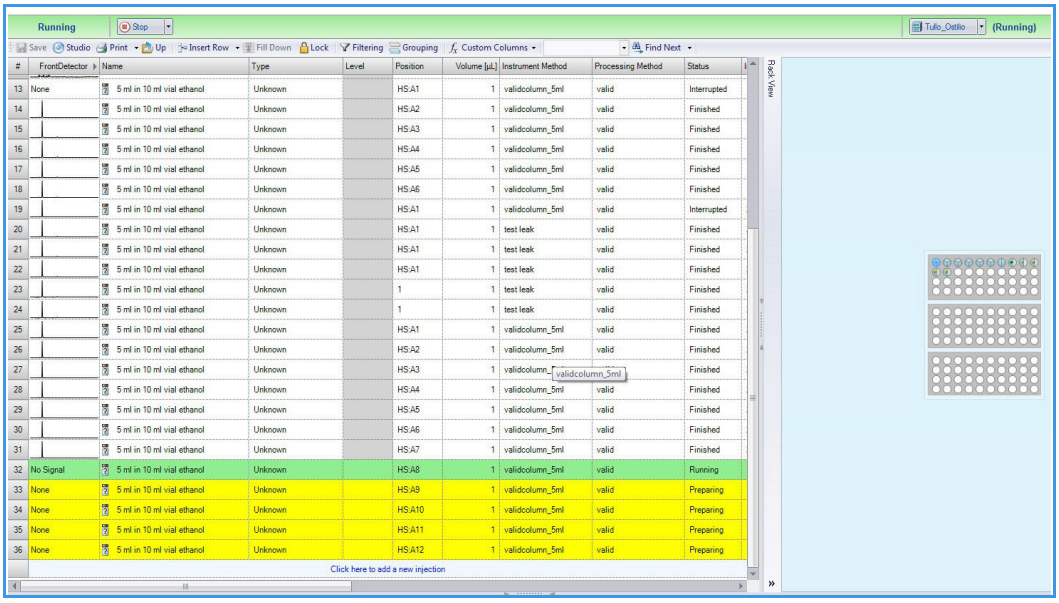
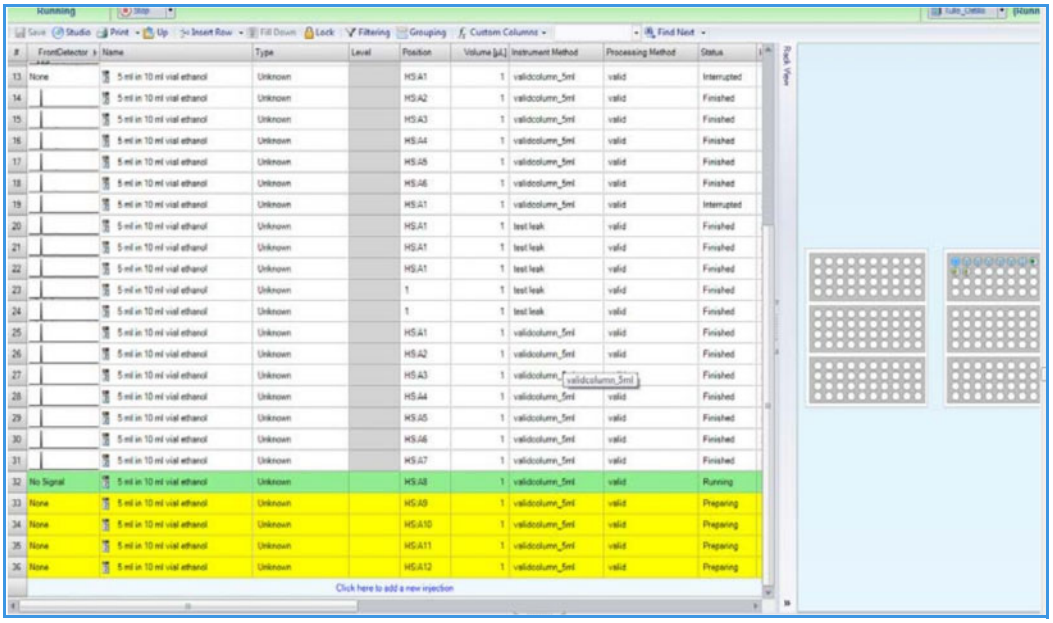


Figure 59. TriPlus 500 HS-120 +120 Vials Sequence Page



Vial Leak Checking

During the injection process, a vial leak check is performed. Depending on the **On Leak Detected** configuration setting, a sequence can be aborted or continue:

Ignore and inject — The leak check fails, the sample is injected.

Fake injection — The leak check fails, the injection process continues, but the valve is not switched.

Abort — The leak check fails, and the sequence is aborted.

Abort after 3 consecutive leaks — The leak check fails three consecutive injections (using different vials) within a subsequence, the sequence is aborted.

If the leak check fails, a **Vial Leak Detected** warning is reported for:

- Ignore and inject
- Fake injection
- Abort after 3 consecutive leaks (if it is not the third consecutive leak)

A **Vial Leak Detected** error is reported for:

- Abort
- Abort after 3 consecutive leaks (if it is the third consecutive leak)

Note Every time a Vial Leak Detected warning or error is reported, that warning or error is logged in the audit trail.

In addition to a warning or error logged in the audit trail, a **Vial Leak Check: Failed** or **Vial Leak Check: Succeeded** message is logged at the time of the injection response.
A dedicated VialLeakDetected property is set to Yes or No and also logged in the audit trail.

The VialLeakDetected property is logged in the audit trail, and can be reported. Its value can also be shown in the sequence by setting up a dedicated custom column, using a formula to search the audit trail for the VialLeakDetected property.

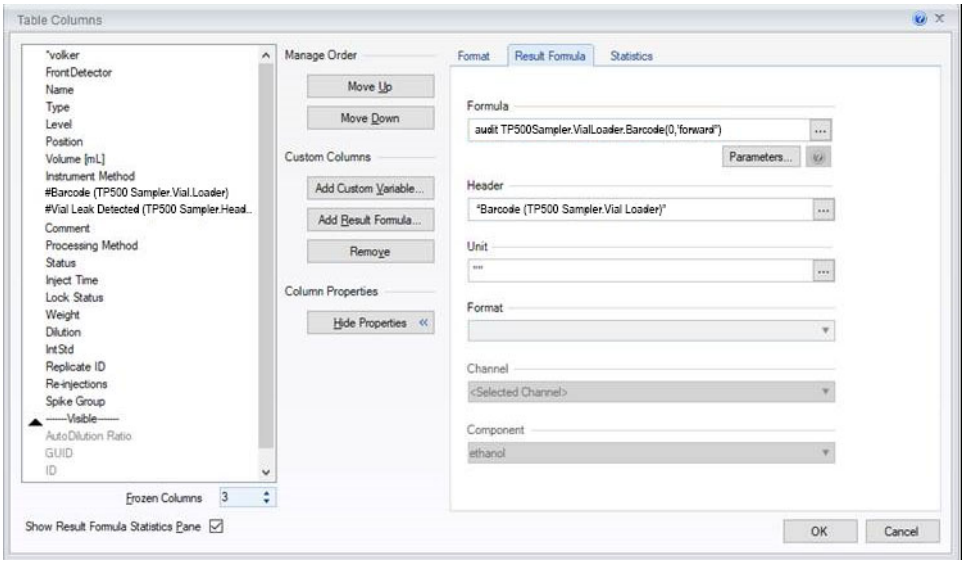
Note You can create a custom column by right-clicking on a column header of the sequence and choosing the **Custom Columns>>Insert Custom Variable**, this will start the Custom Variables Wizard.

Figure 60. Vial Leak Detected custom column

#	#Vial Leak Detected (Tri Plus500.Headspace)	BackDetector	Name	Position	Volume [mL]	Instrument Method	Processing Method	Status
1	No	None		HS:A22	1.00	Method1		Finished
2	No	None		HS:A2	1.00	Method1		Finished
3	No	None		HS:A1	1.00	Method1		Finished
4	Yes	None		HS:A2	1.00	Method1		Finished
5	Yes	None		HS:A3	1.00	Method1		Interrupted
6	n.a.	None		HS:A4	1.00	Method1		Interrupted

The audit trail can be searched with a formula and values displayed in dedicated custom column.

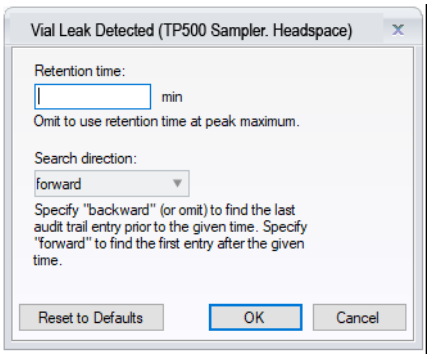
Figure 61. Creating a formula to display a custom column



Note The formula audit.<device name>.VialLoader.VialLeakDetected(0;"forward") will report the VialLeakDetected property in a dedicated custom column; <device name> is a user defined name.

For example, audit.TP500Sampler.VialLoader.VialLeakDetected(0;"forward").

Figure 62. Formula parameters



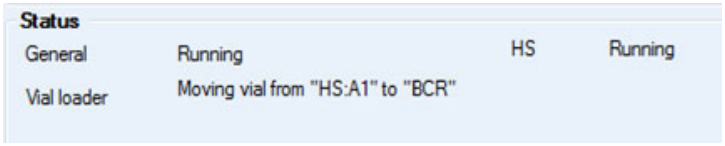
Note For more details about custom columns refer to the *Chromeleon Help*.

Barcode Reading

Barcode reading is available if the barcode reader is configured and the **Read Barcode** configuration option is enabled.

Prior to a vial being placed into the incubation oven, the vial loader moves it from its tray position to the barcode reader slot. During this process, the vial loader status indicates the vial is moving from the tray to the barcode reader. See the example in Figure 63.

Figure 63. Vial loader status



Barcode reading takes a few seconds. The Barcode property is updated with the barcode string at the time of injection (i.e. not immediately after the barcode is read). The value of the barcode (the Barcode property's name and value) is logged in the audit trail at this time.

If a barcode is not read, a warning or error message is logged in the audit trail indicating the barcode reading failed. The injection is not aborted and continues.

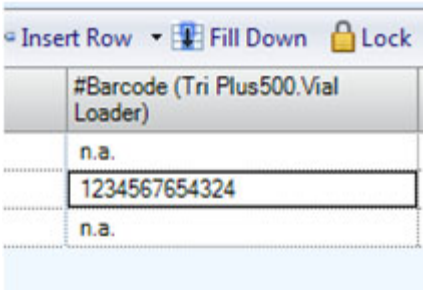
Next, the vial loader moves the vial from the barcode reader slot to the incubation oven. During this process, the vial loader status indicates the vial is moving from the barcode reader to the tray position.

The Barcode property is logged in the audit trail, and the barcode string is reported.

The barcode string can be reported by setting up a dedicated custom column, and using a formula to search the audit trail for the Barcode property.

Note You can create a custom column by right-clicking on a column header of the sequence and choosing the **Custom Columns>>Insert Custom Variable**, this will start the Custom Variables Wizard.

Figure 64. Barcode property custom column



Note The formula audit.<device name>.VialLoader.Barcode(0;"forward") will report the Barcode property in a dedicated custom column; <device name> is a user defined name.

For example, audit.TP500Sampler.VialLoader.Barcode(0;"forward").

Note For more details about custom columns refer to the *Chromeleon Help*.

5

Setting Up Through TraceFinder CDS

This chapter contains the instructions to configure your TriPlus 500 HS and to edit the parameters through the Thermo Scientific™ TraceFinder™ Chromatography Data System (CDS).

Contents

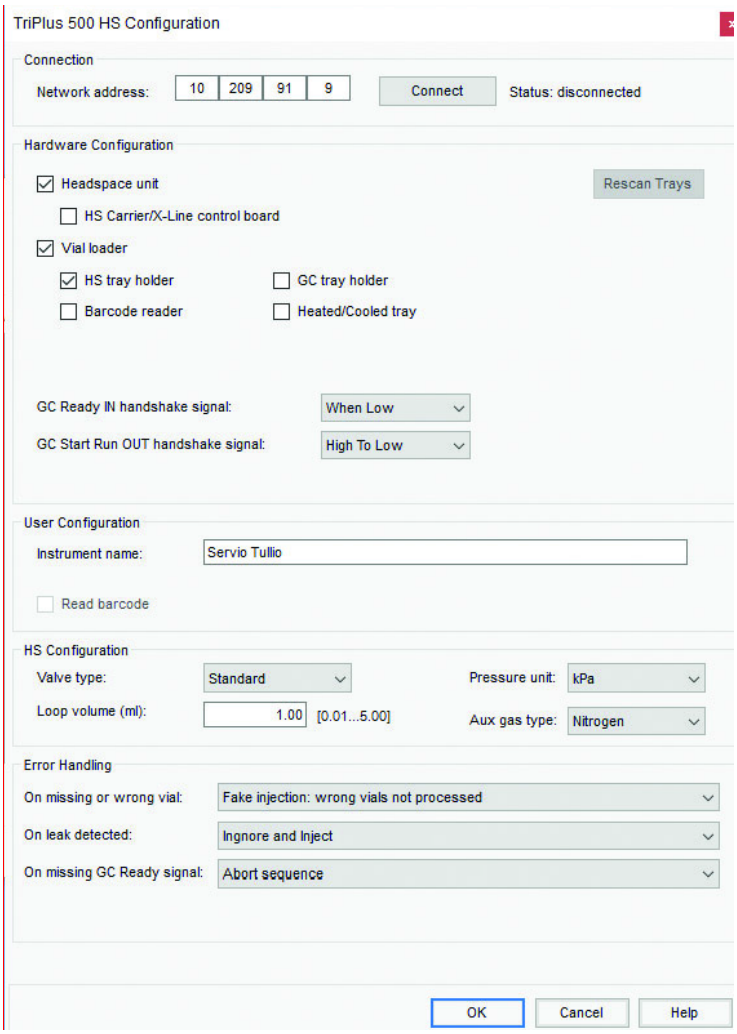
- Configuring TriPlus 500 HS Through TraceFinder CDS
- Editing Method Parameters Through TraceFinder CDS
- TraceFinder Road Map Home Page Status Tabs

Configuring TriPlus 500 HS Through TraceFinder CDS

Run your Thermo Scientific™ TraceFinder™ Chromatography Data System (CDS), then open TriPlus 500 HS **Configuration** window.

Figure 65 shows the Configuration dialog window of TraceFinder CDS for TriPlus 500 HS.

Figure 65. TraceFinder™ Configuration Dialog Window for TriPlus 500 HS



The Configuration dialog window is subdivided into the following areas:

- "Connection" on page 79
- "Hardware Configuration" on page 79
- "User Configuration" on page 80
- "HS Configuration" on page 80
- "Error Handling" on page 80

Connection

In this area you can configure LAN communication between the TriPlus 500 HS and the TraceFinder Chromatography Data System (CDS).

- **Network Address** — The TriPlus 500 HS is shipped from the factory with Dynamic Host Configuration Protocol (DHCP) enabled. If the DHCP is unable to acquire an IP address from the server it will use the following default settings:
  - The default IP address is 169.254.250.4.
  - The default netmask is 255.255.255.0.
  - The default gateway is 169.254.250.1.
  - The port is a number given by the network administrator - for example 2551.

  To change the default settings, contact your LAN administrator.
- **Connect** — By clicking this button the configuration is read automatically by the HS sampler when connected. Connection status is shown to the right of the button (e.g: disconnected, connected).

Hardware Configuration

In case of off-line edit, in this area you set the hardware configuration of your TriPlus 500 HS.

Headspace Unit — Select this check box to choice your instrument. The following sub-selections are available:

HS Carrier/X-Line control board — Select the relevant check box in case the instrument is equipped with the optional local carrier gas control or external transfer line.

Vial loader — Select this check box if your instrument is provided with the Vial Loader. The following sub-selections are available:

HS tray holder / GC tray holder / Barcode reader / Heated-Cooled tray — Select the relevant check box according to the objects present on the instrument.

GC Ready IN handshake signal — Signals to the HS sampler that the GC is ready. To allow other devices to run properly, select the correct option from the drop down menu, depending on how the signal will change. **When Low** is the default value for the TRACE 1300/1600 Series GC.

GC Start Run handshake signal — Signals to the GC that the run has started. To allow other devices to run properly, select the correct option from the drop down menu, depending on how the signal will change. **High -> Low** is the default value for the TRACE 1300/1600 Series GC.

Rescan Trays — Click this button to rescan the trays present in the system.

User Configuration

In this area you specify the instrument name and enable/disable the reading of the barcode.

Instrument name — Type a name you want to assign to your HS sampler.

Read barcode — Select the check box to enable reading of sample barcodes.

HS Configuration

In this area you set the type of sampling valve, the volume of the sample loop, the pressure units and the type of gas used for pressurizing the HS sampler.

Valve type — Specify the type of sampling valve installed into your HS sampler. Select one of the following valve types from the list: **Standard** or **High Temperature**.

Loop volume — Enter the volume of the sample loop installed on the sampling valve. The range is from 0.01 to 5.00 mL.

Pressure Unit — Specify the unit of measurement for pressure. Select one of the following units from the list: **kPa**, **bar, or psi**.

Aux gas type — Specify the type of auxiliary gas in use. Select one of the following gas types from the list: **Helium**, **Nitrogen**, or **Argon**.

Error Handling

In this area you configure how to treat vial missing errors. As the default a recoverable error as vial missing or wrong vial, leak detection and GC ready signal missing, is treated with a fake injection. Nevertheless, there are cases where the instrument stops injecting samples and needs to inform the user that it has stopped. In this case, users must select instrument actions in case of configuration errors. The three selections are:

On missing or wrong vial — Specify the action to occur if a vial is missing or has the wrong size. Select one of the following options from the list:

- **Fake injection** — A fake injection (nothing is injected into the inlet) is performed.
- **Stop sequence** — Any current vial incubation is completed, and then the sequence is stopped.
- **Abort sequence** — The current sample and sequence are stopped immediately.

On leak detected — Specify the action to occur if a leak is detected. Select one of the following options from the list:

- **Ignore and inject** — The leak is ignored and the sample is injected as normal.
- **Fake injection** — A fake injection (nothing is injected into the inlet) is performed.
- **Abort** — The current sample and sequence are stopped immediately.

On Missing Ready Signal — Specify the action to occur if there is no GC Ready signal to indicate to the HS that the GC is ready. Select one of the following options from the list:

- Abort sequence.
- Wait

Configuration dialog window includes the following buttons:

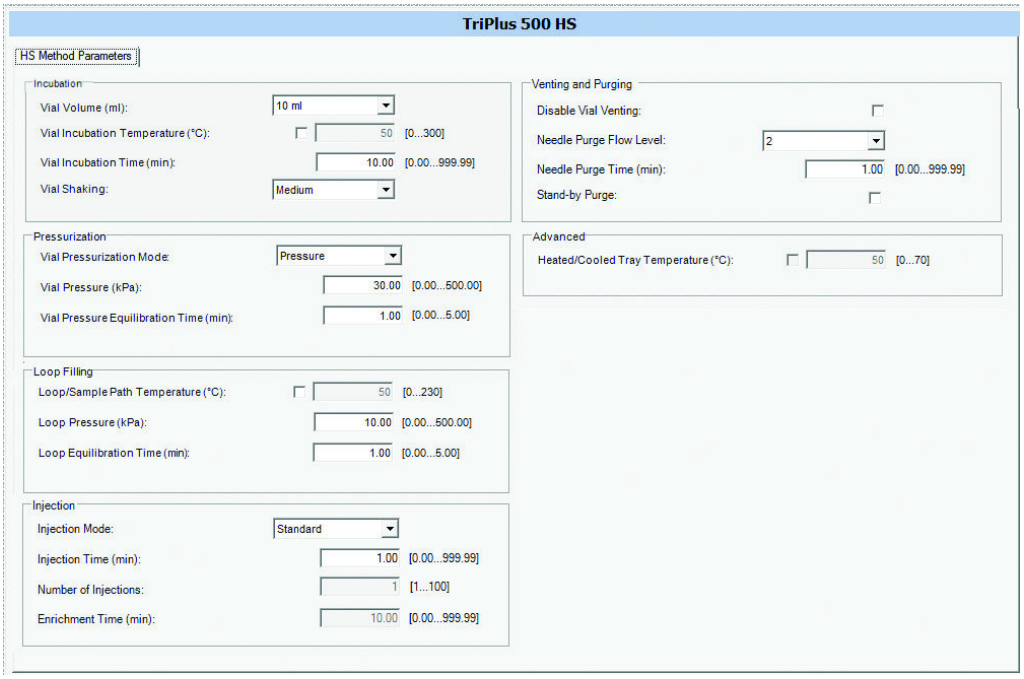
- **Ok** — Closes the dialog window and confirm your selections.
- **Cancel** — Clears all modifications.
- **Help** — Opens help window.

Editing Method Parameters Through TraceFinder CDS

This section provides instruction for creating and saving an instrument method for TriPlus 500 HS.

Figure 66 shows the **HS Sampler Instrument Method Setup** page of the TraceFinder Chromatography Data System.

Figure 66. TraceFinder HS Sampler Instrument Method Setup Page



The Method Setup page contains the following areas:

- "Incubation" on page 82
- "Pressurization" on page 82

- "Loop Filling" on page 83
- "Injection" on page 83
- "Venting and Purging" on page 84
- "Advanced" on page 84

Incubation

In this area you set all the temperatures for the sample incubation.

Vial Volume (mL) — Defines the volume of the vial to incubate. **10 mL** or **20/22 mL**.

Vial Incubation Temperature (°C) — Select this check box to enable the temperature control of the incubation oven. In the adjacent box type the temperature of the incubation oven. Set a value in the range 0–300 °C.

Vial Incubation Time (min) — Defines the time during which the vial is conditioned in the oven at a fixed temperature before the pressurization starts. Set a value in the range 0.00–999.99 minutes.

Vial Shaking: — Select the desired shaking mode. The following modes are available: **Off**, **Slow**, **Medium**, and **Fast**. If a shaking mode is enabled, the incubation carousel will keep moving during the incubation in order to stir the samples.

Pressurization

In this area set the pressurization parameters:

Vial Pressurization Mode — Defines how the vial pressurization will be performed. Select the desired mode from the dropdown list. The following modes are available: **Pressure, Time,** and **Rate.** The selected mode activates the associated parameters.

Vial Pressure (kPa) — Defines the target pressure level at which the vial is pressurized. Set a value in the range 0.00–500.00 kPa.

Vial Pressure Equilibration Time (min) — Defines the time for equilibrating the vial pressure after the pressurization step. Set a value in the range 0.00–5.00 minutes.

Vial Pressurization Time (min) — Defines the duration of the pressurization. Set a value in the range 0.00–10.00 minutes.

Vial Pressurization Rate (kPa/Min) — Defines the rate of the pressure change. Set a value in the range 30.00–3000.00 kPa/min.

Loop Filling

In this area set the following parameters:

Loop/Sample Path Temperature (°C) —Enter the temperature of the loop/sample path. For a standard valve, set a temperature in the range 0–225 °C. For the high-temperature valve set a value in the range 150–300 °C.

Loop Pressure (kPa) — Defines the pressure target that the loop must achieve. Set a value in the range 0.00–500.00 kPa.

Loop Equilibration Time (min)— Defines the time for equilibrating the loop pressure after the pressurization step. Set a value in the range 0.00–5.00 minutes.

Injection

In this area set the injection parameters:

Injection mode — Selects the type of injection. Choose one mode from the dropdown list among **Standard, Enrichment**, and **MHE.**

- **Standard** — Incubates the vial, fills the sample loop, then starts a run while injecting the sample into the GC. The loading time of the vials into the incubation oven is automatically calculated to guarantee the same equilibration time for all the samples, and the optimization of the analysis times. According to the equilibration time and GC time, more vials can be contemporaneously present inside the incubation oven.
- **Enrichment** — Incubates the vial, fills the sample loop and inject into the GC as many times as the number of times selected, then starts the run. The vial remains in the incubation oven between one extraction and the next. The time between two consecutive extractions is set with the parameter **Vial Enrichment Time**. The parameters **Number of Enrichments** and **Enrichment Time** are enabled.
- **MHE** — Multiple Headspace Extraction. Each vial is pressurized, sampled, and vented multiple times. The incubation time for all the extractions is equal to the incubation time set in the method. At each sampling an analysis is performed.

Injection time — Defines the time of the sample transfer to the GC. Set a value in the range 0.00–999.99 minutes.

Number of enrichments— Enabled when **Injection Mode** is set to **Enrichment**. Specifies the number of samplings to be carried out from the same sample vial. Set a value in the range 1–100.

Vial enrichment time — Enabled when **Injection Mode** is set to **Enrichment**. Specifies the time between one enrichment of and the next. Set a value in the range 0.00–999.99 minutes.

Venting and Purging

In this area set the venting and purging parameters.

- **Disable Vial Venting** — Select this check box to disable the vial venting performed after sampling.
- **Needle Purge Flow Level** — Specifies a target flow rate of the purge gas for cleaning the sampling valve and the needle. Set a value from 1 to 5. The default value is **2**.
- **Needle Purge Time** — Defines the duration of the purging phase after sampling. Set a value in the range 0.00–999.99 minutes.
- **Stand-by Purge** — Check this box if you want that the purge gas flows continuously in the system when in stand-by condition.

Advanced

In this area set the temperature of the optional object when present.

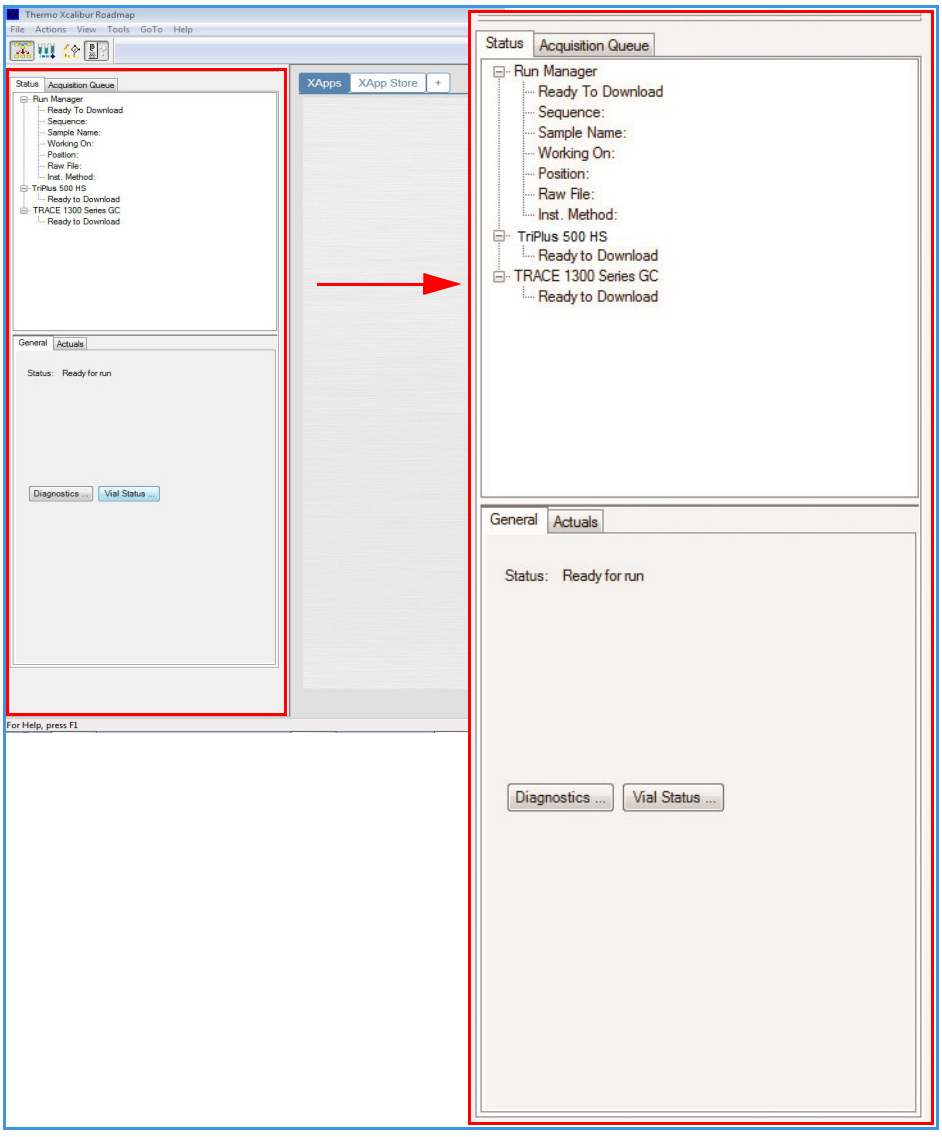
Heated/Cooled Tray Temperature (°C) — Specify the tray temperature. Set a value in the range 4–70 °C.

TraceFinder Road Map Home Page Status Tabs

This section describes how to check on the current status of the TriPlus 500 HS while working in **TraceFinder CDS**.

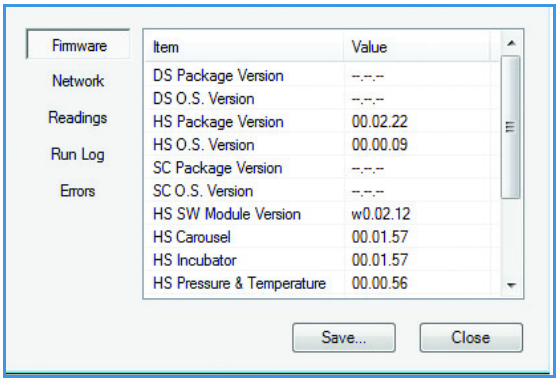
Status pages are located on TraceFinder Roadmap-Home page. Just highlight TriPlus 500 HS from the Roadmap Status tab scroll list and see the TriPlus 500 HS sampling system tab. See Figure 67.

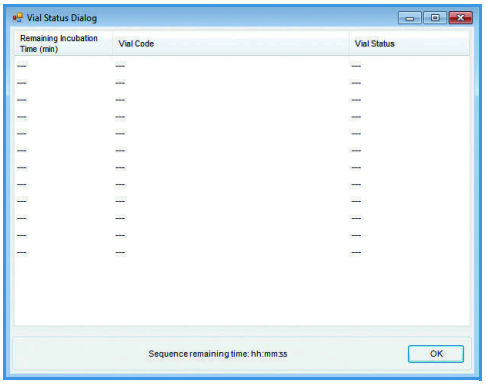
Figure 67. TraceFinder Status Tabs



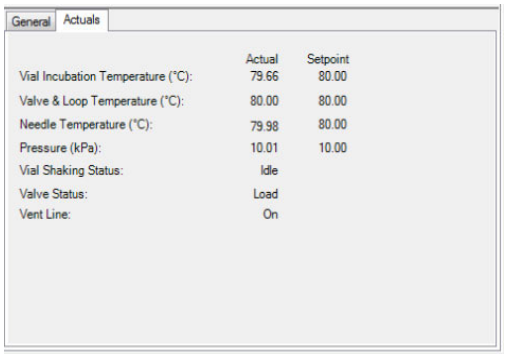
This tab comprises the following options:

General — Displays **Diagnostics** and **Vial Status** information.





Actuals — Displays status of temperatures, pressures, vials, and valve status currently in the system.



Note For details please refer to the *Thermo Xcalibur User Guide -TriPlus 500 Headspace Sampler Setup* PN 31716109.

6

Method Development

This chapter provides information for developing a method with the TriPlus 500 HS.

Contents

- Usage Notes
- Processing a Sample Vial
- Method Developing Workflow
- Analytical Troubleshooting General Guidelines

Usage Notes

This section provides some advice about the headspace technique for optimizing parameters that can affect the sensitivity, precision, and accuracy of an analysis.

The related topics are:

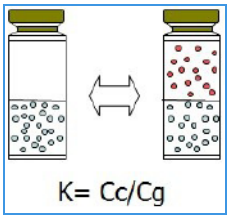
- "Temperatures" on page 88
- "Carrier Gas Optimization" on page 88
- "Auxiliary Gas Pressure Optimization" on page 88
- "Shaking Conditioning" on page 89
- "Sample Vial Septa" on page 89
- "Vial Filling" on page 90
- "Vial Closure" on page 90
- "Missing and Wrong Size Vials" on page 91
- "Matrix Effects" on page 91
- "Principles of Multiple Headspace Extraction (MHE)" on page 92

Temperatures

The oven temperature can have a profound effect on the concentration of analyte that passes into the gas phase.

In general as the temperature increases, the amount of analyte in the gaseous phase increases, and consequently the sensitivity of the method.

The tendency of a compound to go into the gaseous phase is determined by the partition coefficient **K**:



where: **Cc** is the concentration of the analyte in the condensed phase (matrix phase), and **Cg** is the gaseous phase.
The partition coefficient **K** is highly dependent on temperature as demonstrated by the following equation:

dK/dT = 1/T2

Consider the following when choosing the oven temperature:

- The best results are achieved when the temperature is maintained at the minimum level sufficient to ensure the desired analytical sensibility.
- Do not set the oven temperature within 10 °C of the boiling point of any solvent, except in special cases.
- Thermally labile compounds may degrade at elevated temperatures.

The sampling path temperatures must be equal to or higher than the oven temperature to avoid the condensation of the analytes in the pneumatic system, and consequently avoid the presence of residuals.

Carrier Gas Optimization

The carrier gas flow rate should be set high enough to transfer the headspace sample out of the loop into the GC without causing peak broadening. For a capillary column that generates a 1-second-wide peak, the flow rate should be higher than 60 mL/min. In this case the flow rate will be the total flow (column flow + split flow).

Auxiliary Gas Pressure Optimization

The pressure developed in the vial allows you to transfer the head space sample to the sample loop. The auxiliary gas pressure should be set at the minimum value to ensure the loop filling. To optimize your selection of vial pressurization, run a series of vials using different pressures, and interpret the optimum condition from peak areas versus vial pressurization.

Note Optimization is valid only for analysis of a specific sample at a specific oven temperature.

Shaking Conditioning

In some cases, and especially for aqueous samples, shaking during the equilibration phase reduces the time required to reach the equilibrium.

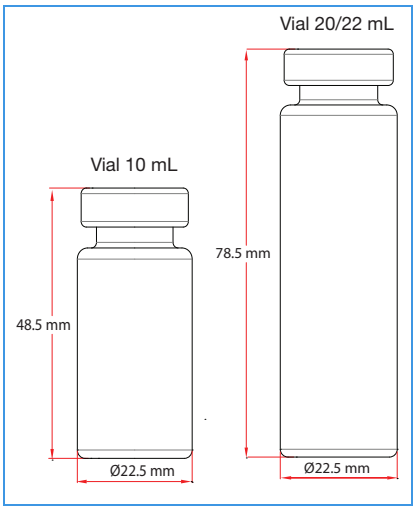
The shaking benefits samples with a larger amount of liquid, liquid samples with high viscosity, and analytes with high partition coefficients K.

Sample Vials

TriPlus 500 HS can use 10 mL and 20/22 mL vials. The vial size may change with each method in use in a sequence.

The vial can have crimp or screw caps with flat or rounded bottoms and can have clear or amber glass. Use amber glass for light–sensitive samples.

Vials must conform to the following specifications:



A larger sample size may give greater sensitivity. Peak areas are influenced by the relative amount of the gas and condensed phases in the vial.

If the sensitivity is not a fundamental requirement, small vials may be preferred because a shorter equilibration time is required.

The bottom profile of a headspace vial may be round or flat. Generally a round-bottomed vial tends to withstand higher pressures and is more suitable for elevated temperature.

Make sure to use pre-cleaned vials. Dirty or re-washed vials can cause ghost peaks or erroneous results. See Table 3 for allowable sample vial dimensions.

Table 3. Allowable Sample Vial Dimensions

Vial Capacity (including septum and cap)	Minimum Height (mm)	Maximum Height (mm)	Minimum Outer Diameter (mm)	Maximum Outer Diameter (mm)
10 mL	46.5	49.5	22.25	23.5
20/22 mL	75.5	79.0	22.25	23.5

Sample Vial Septa

Use septa suitable for the temperature of the system. Poor septa can bleed and contaminate the headspace.

The use of septa with the lower layer in PTFE is recommended to avoid contamination of the sample. Always confirm that there are no peaks in the blank analysis chromatogram.

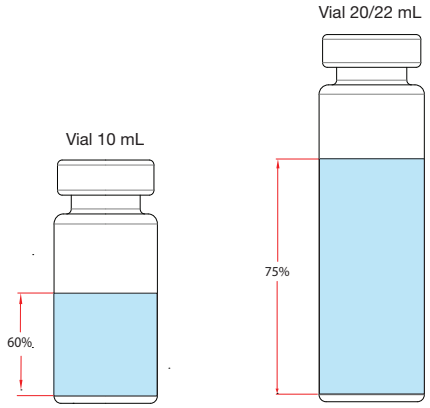
Suggested septum thickness = minimum 2 mm.

Vial Filling

To avoid liquid under the septum during shaking and to leave a minimum head space volume, it is suggested to fill the vial according to the following limits.
See Figure 68.

- Maximum **60%** (6 mL) of the vial capacity for the 10 mL vial.
- Maximum 75% (15 mL) of the vial capacity for the 20/22 mL vial.

Figure 68. Recommended Maximum Filling Percentage of Sample in the Vial.



Vial Closure

A frequent, and non-negligible cause of error, is a leak due to incorrect closure of the vial. To close the vials, use a crimper then check each vial for proper crimping.

- The vial requires the use of a **metallic** cap.
- If the cap is loose the vial may leak.
- Adjust the crimper and close the vial again.
- The cap should be flat.

The deformation of the cap can generate some problems for the HS sampler during the pick-up phase of the vial from the incubation oven or from the tray holder when present.

		
Correct crimp	**Over crimp**	**Under crimp**
Undeformed sides, good seal	Deformed sides, upward bulge, probably problem with the HS sampler	Loose seal, caps twists easily

Missing and Wrong Size Vials

If the instrument does not find the vial, or the vial is of a different size than the one set in the method, the HS sampler performs the action selected in **Error Handling**.

CAUTION While the TriPlus 500 HS recognizes the type of the vial by its height, the instrument does not actually distinguish between 20 mL and 22 mL vials. There are commercial 22 mL vials that are not compatible with the TriPlus 500 HS. Follow the guidelines in the section "Sample Vials" on page 89 to choose the correct sample vials.

ATTENTION Puisque le TriPlus 500 HS reconnaît le type du flacon par sa hauteur, l'instrument ne fait pas de distinction entre un flacon de 20 ml et un de 22 ml. Certains flacons commerciaux de 22 ml ne sont pas compatibles avec le TriPlus 500 HS. Respectez les consignes de la section Flacons d'échantillon à la page 89 pour choisir les bons flacons d'échantillon.

Matrix Effects

The composition of the sample matrix may affect the amount of the analyte that is transferred to the headspace.

Adding an inorganic salt to an aqueous sample increases the concentration of organic molecules in the gas phase. The salt makes these compounds less soluble in the sample matrix, and sensitivity increases.

Usually the matrix is not a pure compound, but a complex mixture of compounds, some of which may not be volatile. The interaction of the matrix components with the analyte influences its solubility. This is called matrix effect.

Matrix effects are important when an external standard must be developed. The composition of the matrix in the standard must resemble as much as possible that of the sample.

The effect of the matrix on a standard can be compensated by quantifying results using Multiple Headspace Extraction (MHE).

Principles of Multiple Headspace Extraction (MHE)

The multiple headspace technique is used in instances where interfering matrices interact with the analyte, such as by partial adsorption, or if a solid analyte contains low concentrations of moisture. Whenever it is impossible to prepare the calibration standard using an identical matrix to that of the actual sample, single point calibration using the static headspace technique will fail.

The term multiple headspace extraction (MHE) is partially self-descriptive. To determine the quantity of an analyte, sampling is carried out by repeated extraction of gas from the same vial above a sample solution containing the analyte (known as the headspace).

The method thus approaches continuous gas extraction but is carried out in a stepwise fashion. In concept, the procedure is analogous to the multiple extraction of a sample from a simple elution column, where each passage of eluate through the column reduces the amount of analyte contained in a subsequent passage.

The peak area from any given extraction will be smaller than that of the previous extraction step. The sum of the peaks will be proportional to the total content of the analyte. A quantitative determination of the total amount of analyte present in a sample depends on the relationship between peak area and the amount of analyte. The sum of the amounts of analyte that are removed will eventually equal the total amount of analyte in the original sample if the extractions are continued to infinity.

Theoretically, summing up the peaks obtained from an infinite number of column extractions would allow the calculation of the absolute amount of analyte in a sample. In practice, it is sufficient to carry out a limited number of extractions, permitting a determination of the underlying exponential relationship between the measured area and the number of extractions, in turn allowing for the determination of the total amount of analyte in the sample.

The effect of the sample matrix is eliminated by extracting the entire amount of the analyte. Contrary to the situation that exists in single-headspace extraction, it is possible to use calibration standards that are not prepared with the same matrix as the actual samples.

For MHE to provide meaningful results, the only prerequisite is that equilibrium must exist with respect to the analytes in terms of the distribution between the phases in the headspace vial.

Instrumentmation in MHE

In a normal single extraction, the following steps are employed in order to sample an aliquot of the headspace gas:

- The vial is thermostatted in order for equilibrium to be established between the phases in the headspace vial.
- Loop-injection headspace samplers use a pressurization step to start the headspace cycle. The vial is pressurized to a preselected value. The pressure is selected so that it is always greater than the pressure increase due to sample heating and vapor pressure of the sample.
- The sample is introduced into the GC column for analysis and the system is ready for the next sampling after cleaning.

In MHE, equilibrium must be re-established in the headspace following each extraction.

The following steps would normally be employed:

- The vial is thermostatted in order for the equilibrium to be established between the phases in the headspace vial.
- The vial is pressurized.
- An aliquot of headspace gas is introduced in the GC column.
- The vial is vented. The pressurized headspace gas vents to atmospheric pressure.
- Equilibrium is established again before the next analysis.
- This procedure is repeated a number of times.

Examples

In the following examples, we will briefly go through the basics of multiple headspace extraction (MHE).

We will look at four examples (situations) of equilibrium between the condensed (solid or liquid) phase and the gas phase (the headspace). The examples will be shown in increasing complexity with respect to the equilibrium distribution and removal of the headspace between subsequent extractions.
In each of the four cases described below, the complexity of the equilibrium distribution and of the removal of the headspace between subsequent extractions will be increased. The final conclusion is that the MHE method will work in most cases, with the only prerequisite that the distribution of the analyte between the gas and the condensed phases is actually governed by an equilibrium.

Situation 1

The equilibrium between the solid phase and the gas phase is assumed to be a 50/50-distribution. In the course of each extraction, **all** of the headspace is removed/exchanged.

The measured area of the analyte peak is **A**.

A(ref) — Area we would measure if all of the analyte were in the headspace (corresponding to the measured signal from a similar gas standard).

A(0) — Measured area of the analyte peak from the first extraction.

With 50% of the analyte present in the headspace, this amounts to:

A(0) = ½ * A(ref)

The total area that is determined over a number of subsequent extractions is:

A(tot) = A(0) + ½ * A(0) + ¼ * A(0) + 1/8 * A(0) + ……, or

A(tot) = A0 / (1-½) = 2 * A0

However, A(0) = ½ * A(ref) and therefore:

A(tot) = A(ref)

Thus, it would be correct to determine the total amount of analyte by comparing the measured sum of analyte areas to a calibration measurement with data from a single determination of a gas standard.

Situation 2

The equilibrium between the condensed phase and the headspace is assumed to be 25/75 (with 75% in the gas phase). Furthermore, we assume that the entire headspace is exchanged during the course of each extraction step.

The measured area of the analyte peak is **A**.

A(ref) — Area that would be measured if all of the analyte were present in the headspace (again corresponding to the measured signal from a similar gas standard).

A(0) — Actual area measured from the first extraction with 75% of the analyte in the gas phase:

A(0) = ¾ * A(ref)

The total area determined by the MHE procedure is:

A(tot) = A(0) + ¼ * A(0) + 1/16 * A(0) + ……, or

A(tot) = A(0) / (1-¼) = 4/3 * A(0)

However, A(0) = ¾ * A(ref) and therefore:

A(tot) = A(ref)

In this case, you also arrive at the correct value by determining the total amount of analyte by comparing the measured sum of analyte areas to a calibration measurement using data from a single determination of a gas standard.

The conclusion is: when in equilibrium, the relative ratio of the condensed phase versus the headspace plays no role in the amount of analyte content that is determined from an MHE-series. Furthermore, it can be concluded that quantification based on a single determination from a gas standard will, in this particular case, yield the correct result, assuming that the entire headspace is removed/exchanged during each extraction step.

Situation 3

The equilibrium between the condensed phase and the headspace is assumed to be 50/50.

Furthermore, we assume that only a part of the headspace is removed between each sampling. The relative amount of the headspace removed between each successive extraction constant and is denoted α.

The measured area of the analyte peak is **A**.

A(ref) — Area that would be measured if all of the analyte were present in the headspace (again corresponding to the measured signal from a similar gas standard).

A(0) — Actual area measured from the first extraction with 50% of the analyte in the gas phase:

A(0) = ½ * A(ref)

The total area determined by MHE is:

A(tot) = A(0) + (1-? * ½) * A(0) + (1-? * ½)2 * A(0) + ……, or

A(tot) = A(0) / (1-(1-? * ½)) = A(0) * 2/α

However, A(0) = ½ * A(ref) and thus:

A(tot) = ½ * A(ref) * 2/a, or

A(tot) = A(ref)/a

If α =1 (the entire headspace is exchanged between each extraction) then:

A(tot) = A(ref)

If α = ½ (half of the headspace is exchanged between each extraction step) then:

A(tot) = 2 * A(ref)

In this case A(tot) will be over-determined by a factor of two relative to A(ref). In other words, the content of analyte will be over-determined by a factor of two.

Situation 4

Similar to Situations 1 to 3 as described above, the general description for an equilibrium distribution with β being the relative amount of the analyte in the gas phase (in the headspace) and ? being the relative amount of the headspace that is exchanged between each successive extraction, is given by:

$$\mathbf{A(tot) = \beta * A(ref) * (1 + (1-\alpha * \beta) + (1-\alpha * \beta)2 + \ldots)}$$

Using the expression for the geometric sum yields:

$$\mathbf{A(tot) = \beta * A(ref)/(1-(1-\alpha * \beta)) = \beta * A(ref) * 1/(\alpha * \beta)}$$

or:

$$\mathbf{A(tot) = A(ref)/\alpha}$$

That is: the total area, A(tot), will be over-determined by a factor of 1/α relative to A(ref). In other words, the content of analyte will be over-determined by the factor 1/α. Thus the total area will depend on the amount of headspace removed during each successive extraction, but it will not depend on the position of the equilibrium (β).

Conclusion

If the headspace is only partially removed/exchanged between each extraction, the total area that results from the MHE determination will be greater than A(ref), where A(ref) denotes the area from a pure gas standard measured only once). Therefore, if MHE quantification is performed based on a singly-determined gas standard, the amount of analyte in the sample will be overestimated by a factor of 1/α, with α being the amount of the headspace that is removed/exchanged between each successive extraction. If the MHE quantification is based on gas standards that are subjected to the same MHE extraction sequences as the actual samples, there will be no overestimation. The amount calculated will be correct.

References

For more details, please refer to the corresponding publication on the static headspace technique and, in particular, on the MHE technique.

Title: 'Static Headspace-Gas Chromatography – Theory and Practice'
Authors: Bruno Kolb and Leslie S. Ettre, Second Edition.
Published by: John Wiley & Sons, Inc.

Processing a Sample Vial

Figure 69 and Figure 70 show how the TriPlus 500 HS processes a sample vial according to the configuration HS-12 and HS-120.

Figure 69. TriPlus 500 HS-12 - Vial Processed Workflow

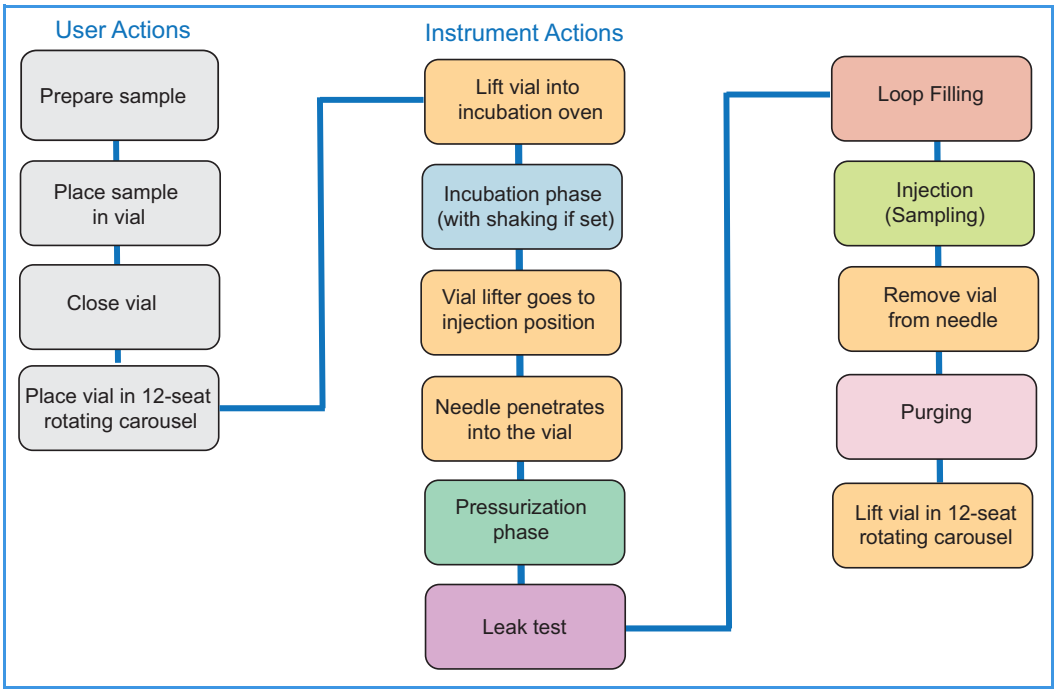
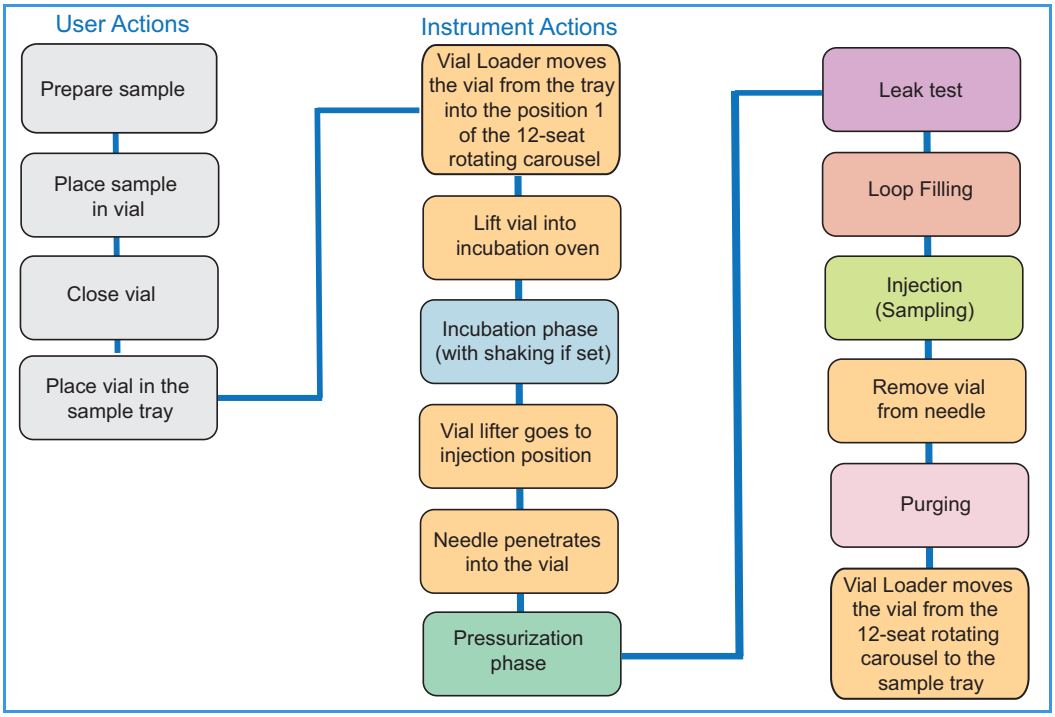


Figure 70. TriPlus 500 HS-120 - Vial Processed Workflow



User Actions

1. Prepare the sample in such a manner as to maximize the concentration of the volatile components in the headspace.
2. Quickly pour the sample into a pre-cleaned vial without shaking it. Fill the vial, paying attention that the maximum filling limit is 6 mL for 10 mL vials and 15 mL for 20/22 mL vials.
3. Quickly close the vial using the proper septa and metallic cap, then crimp the vial correctly to avoid leaks (or screw the cap on according to the vial type).
4. Place the vial into the 12-seat rotating carousel or into a 40-position sample tray according to version of your TriPlus 500 HS-12 or HS-120.
5. Repeat the previous steps for all the required sample vials to analyze.

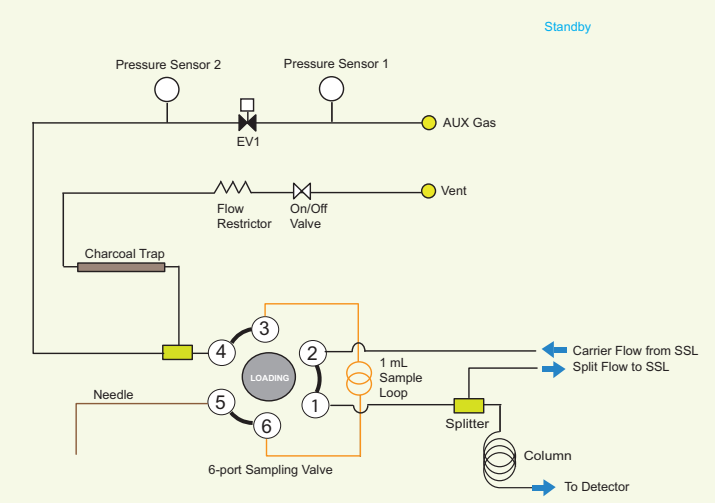
Instrument Actions

Note At this point it is assumed that GC and TriPlus 500 HS are properly installed, all the required gas supplies are open, the method parameters for the GC (oven, injector and detector) and the sampler have been properly set, and the sequence of samples has been defined.

For more details refer to the *TRACE 1300/1310 GC User Guide* or *TRACE 1600/1610 User Guide*.

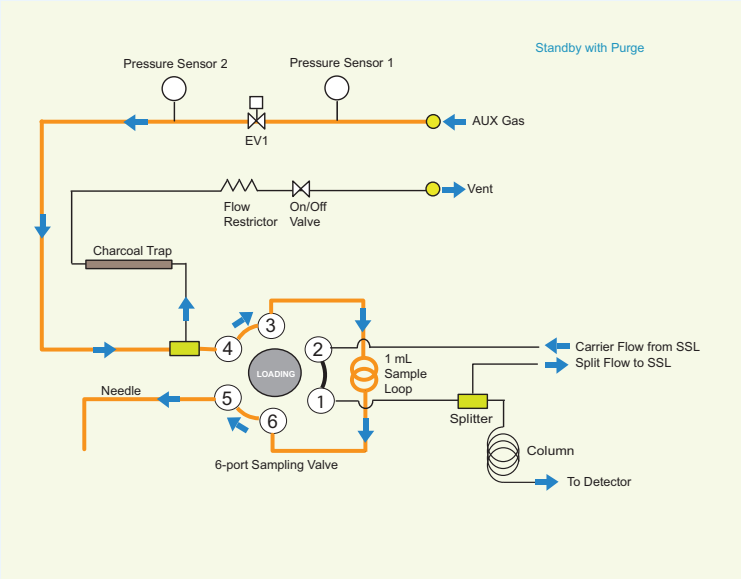
When waiting the **Ready** signal from the GC, the TriPlus 500 HS is in **Standby** condition. See Figure 71.

Figure 71. Standby Condition



Note If in the sampler method the **purging** function has been enabled, during the Standby phase a constant flow of purge gas is circulating in the system. See Figure 72.

Figure 72. Standby with Purge



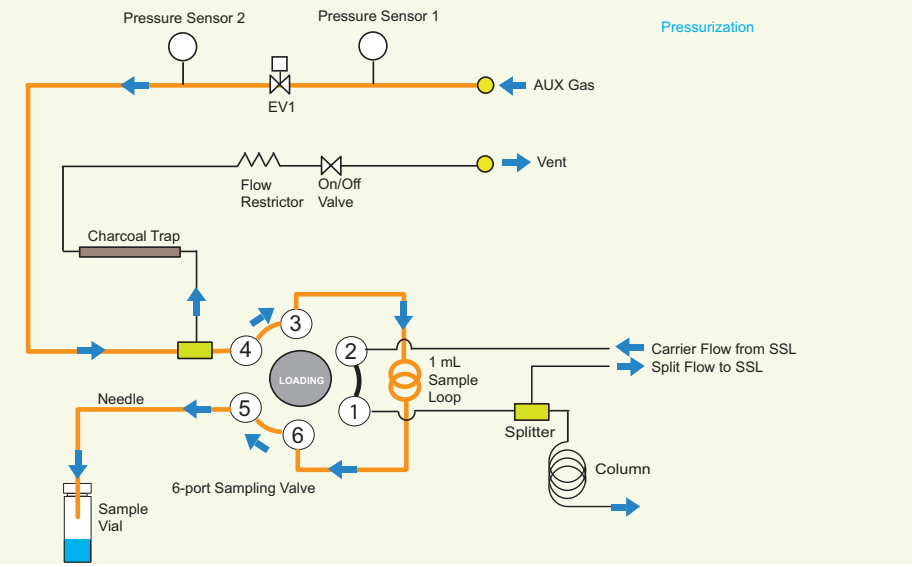
1. When the GC sends the **Ready** signal, the sampler begins the vial loading.
   - In the case of HS-12 version, the sample vials are into the 12-seat rotating carousel.
   - In the case of HS-120 version, the sample vials are into the sample tray while the 12-seat rotating carousel is empty. The Vial Loader moves on the first sample vial of the sequence and transfers it into the position **1** of the 12-seat rotating carousel.
2. At this point, the carousel rotates up to reach the incubation door, then lift the sample vial into the incubation oven.

   The **incubation** phase starts. This phase retains the same configuration as the **Standby** phase or **Standby with Purge** phase. See Figure 71 and Figure 72.

   The incubation oven equilibrates the vial at a temperature from room temperature to 300 °C. The sample vial remains into the incubation oven for a certain time defined in the method.

   In addition, the incubation oven can shake the sample vial at three different levels defined in the method.
3. At the end of the **Incubation** phase the sampler lifts the sample vial in the injection position. The sample needle penetrates into the sample vial and the system starts the **Pressurization** phase. See Figure 73.

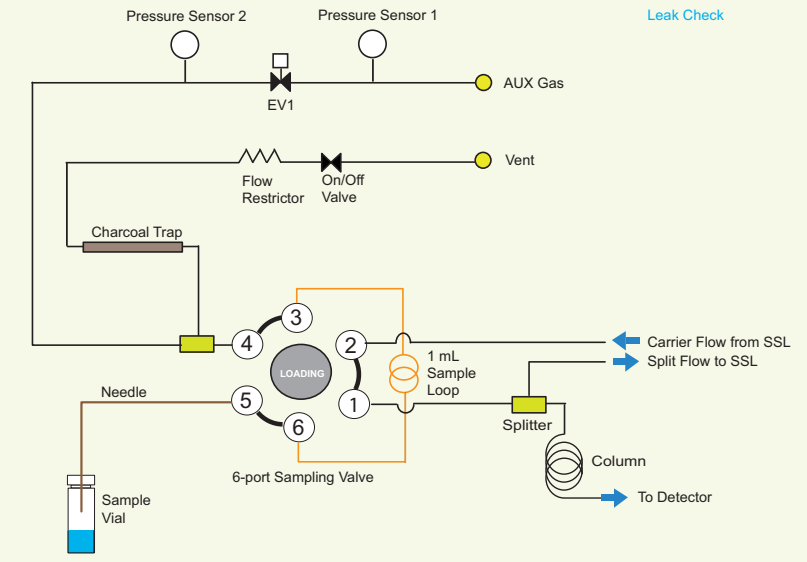
Figure 73. Pressurization Phase



The user can choose among three modes for pressurizing the sample vial. When the time for equilibrating the pressure of the sample vial is elapsed, the pressurization phase ends.

4. At this point the system closes both EV1 and On/Off valves for performing a **Leak check** on the sample vial. See Figure 74.

Figure 74. Leak Check



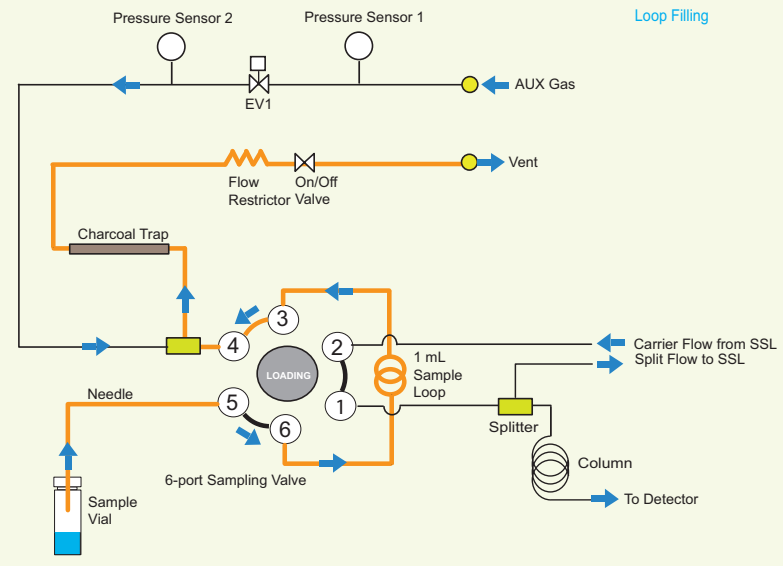
The duration of the Leak check is **12 seconds**.

– If a leak is detected, the instrument behaves according to the **Error handling** configured (for example **Ignore and Inject**, **Fake Injection**, or **Abort**).

– If the Leak check passes, the sampler re-opens the both EV1 and On/Off valves to perform the filling of the loop.

5. The pressurized sample goes to **Vent** through the sample loop. See Figure 75.

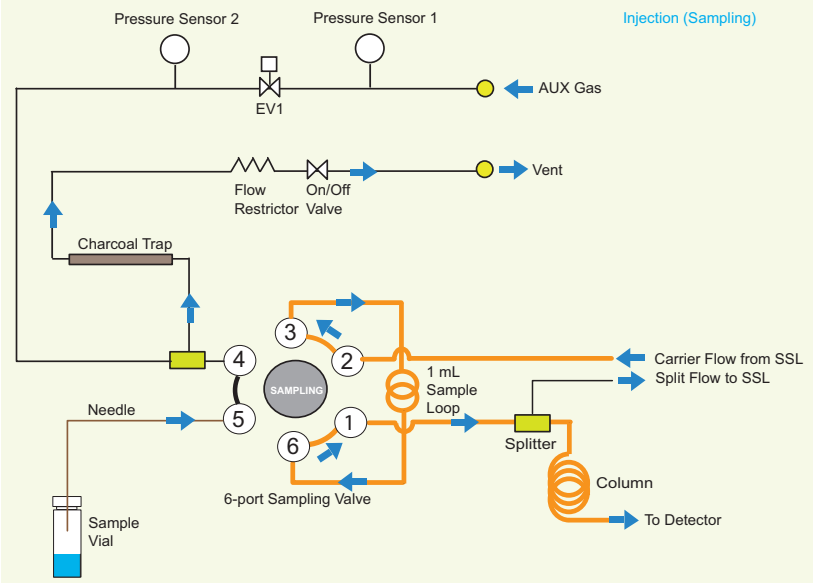
Figure 75. Loop Filling



The system pressurizes the loop. After the specified conditions are met the loop is considered filled.

6. After loop pressure is achieved and the equilibration time of the sample loop elapses, the system injects the sample. See Figure 76.

Figure 76. Injection (Sampling) Phase



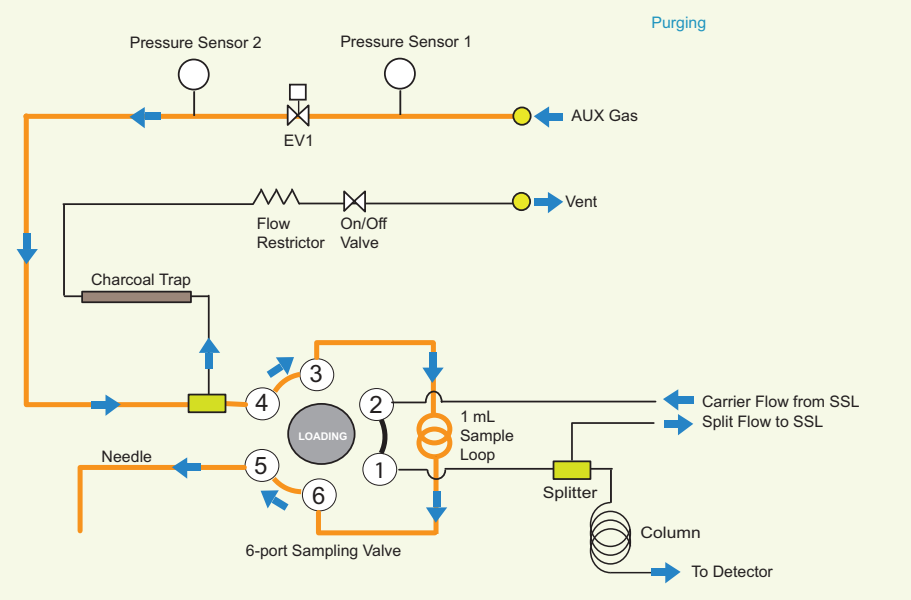
The sampling valve switches on the **sampling** position allowing the transfer of the sample from the sample loop to the analytical column for an injection time defined in the method.

According to the injection mode selected and the relevant parameters, the sampler performs a **Standard**, **Enrichment**, or **MHE** injection.

7. At the end of the sampling phase the sampling valve switch on the **loading** position, the On/Off valve is open and the **vial venting** is performed. The sample vial is removed from the sampling needle and it is transferred back to its original position.

8. The **Purging** phase starts to reduce the carryover. During this phase a purge flow is maintained for a defined period of time at a selected level. See Figure 77.

Figure 77. Purging Phase

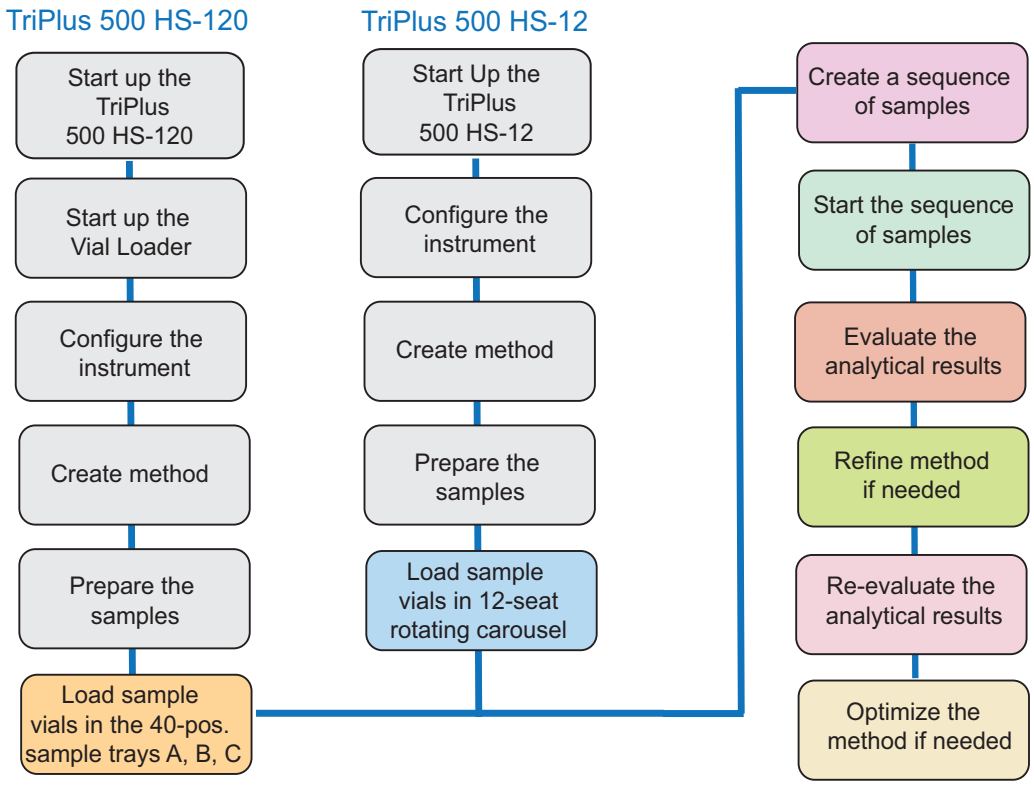


9. The Purging phase ends the analytical sequence.

Method Developing Workflow

Figure 78 shows the typical workflow for developing a method with the TriPlus 500 HS.

Figure 78. Method Developing Workflow



Note It is assumed that TRACE 1300/1600 Series GC and TriPlus 500 HS are properly installed. The Chromeleon or TraceFinder Chromatography Data System (CDS) is installed on your PC. For details refer to Chapter 2 and Chapter 3 of the *TriPlus 500 Headspace Sampler Hardware Manual*.

The user performs the configuration of the sampler, setting up the method parameters and the sequence of samples through the CDS. See Chapter 4 or Chapter 5 in this guide for details.

The user can perform the configuration of the sampler through the user interface (HMI) on the touch screen of the TRACE 1310 or TRACE 1610 GC; or through the CDS for the TRACE 1300 GC or TRACE 1600 GC.

The HMI reflects the parameters set on the CDS. Nevertheless, the user can use the HMI to control the instrument parameters and monitoring the status of GC and HS sampler when an analysis is running. The execution of a sequence of samples is possible in particular cases but without data acquisition (e.g. to check the system when the CDS is not available).

1. Start up the system.

   TriPlus 500 HS-12

   a. Open the gas supplies for GC and TriPlus 500 HS-12.

   b. Power on the GC by placing the power switch in the On (up) position marked I.

   c. Power on the TriPlus 500 HS-12 by placing the power switch in the On (up) position marked I.

   TriPlus 500 HS-120

   a. Open the gas supplies for GC and TriPlus 500 HS-12.

   b. Power on the GC by placing the power switch in the On (up) position marked I.

   c. Power on the TriPlus 500 HS-120 by placing the power switch in the On (up) position marked I.

   d. Power on the Vial Loader by placing the power switch in the On (up) position marked I.

2. Configure the instrument.

   Note The TriPlus 500 HS is shipped from the factory with Dynamic Host Configuration Protocol (DHCP) enabled. If the DHCP is unable to acquire an IP address from the server it will use the following default settings:

   - The default static IP address is 169.254.250.4.
   - The default netmask is 255.255.255.0.
   - The default gateway is 169.254.250.1.
   - The port is a number given by the network administrator - for example 2551.

   a. Run the CDS installed on your computer, then open the **Configuration** dialog window for the TriPlus 500 HS.

   b. Define the **Name** of the device and insert the **Network Address** of the HS sampler.

   c. The system recognizes the Hardware configuration of the HS sampler.

   d. Define the **GC Ready IN** and **GC Start Run OUT** handshake signals. It is suggested to leave the default values.

   e. Define the type of **sampling valve** mounted on the HS sampler and the **volume** of the loop.

   f. Define the type of **auxiliary gas** and the **pressure unit**.

   g. In the area **Error handling** configure how to treat vial missing errors.

   h. Click **Get Configuration** (Chromeleon CDS) or **Connect** (TraceFinder CDS); the configuration is automatically read by the TriPlus 500 HS.

      In case of HMI press **Apply** to confirm the selections.

3. Create Method.
    a. Open the **Setting** page of the CDS in use.
    b. Select the type of **vial** in use (10 mL, 20/22 mL).
    c. Define the **incubation temperature**, the **incubation time** and the **shacking** mode if required.
    d. Define the **pressurization mode** (Pressure, Time or Rate) and the time for equilibrating the pressure of the vial.

> **Note** According to the pressurization mode selected the associated parameters are enabled to be set.

    e. Define the temperature of the **loop sample path**, the target **pressure** that the loop must achieve and define the **time** for equilibrating the loop after the pressurization phase.

CAUTION Thermo Fisher Scientific recommends setting the incubation temperature and the loop sample path temperature to the same value.

ATTENTION Thermo Fisher recommande de régler la température d'incubation et la température du trajet d'échantillonnage de la boucle sur la même valeur.

    f. Select the **injection** mode required (Standard, Enrichment, or MHE)

> **Note** According to the injection mode selected the associated parameters are enabled to be set.

    g. Enable or disable **Vial venting**. When enabled, it eliminates residual pressure in the vial.
    h. Select **Needle purge flow level** (2 is the default value) and the duration time of the purging.
    i. Enable/disable **Standby purge**. When enabled, the purge gas flows continuously in the system.

4. Prepare the sample.
    a. According to the nature of the sample prepare it in such a manner as to maximize the concentration of the volatile components in the headspace.
    b. Quickly pour the sample into a pre-cleaned vial without shaking it. Fill the vial paying attention that the maximum filling limit is 6 mL for 10 mL vial and 15 mL for 20/22 vial.
    c. Quickly close the vial using the proper septa and metallic cap, then crimps the vial correctly to avoid leaks (or screws according to the vial type).

d. Place the vial into the 12-seat rotating carousel or into a 40-position sample tray according to version of your TriPlus 500 HS-12 or HS-120.

e. Repeat the previous steps for all the required sample vials to analyze.

5. Load the sample vials.

   a. In the case of HS-12 version, load the sample vials into the 12-seat rotating carousel.

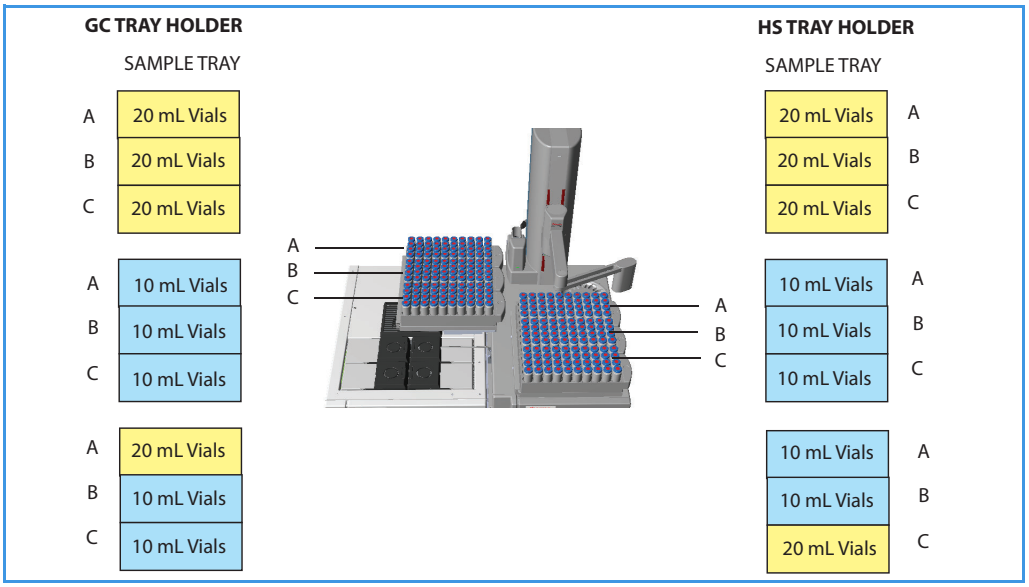
   b. In the case of HS-120 version, load the sample vials into the sample tray while the 12-seat rotating carousel is empty.

CAUTION DO NOT place 10 mL and 20/22 mL vials into the same sample tray. If 10 mL and 20/22 mL vials are used simultaneously, place them on Sample Tray as shown in Figure 79.

ATTENTION Ne placez PAS les flacons de 10 ml et 20/22 ml dans le même plateau d'échantillons.

Si des flacons de 10 ml et 20/22 ml sont utilisés simultanément, placez-les sur le plateau d'échantillons, comme illustré à la Figure 79.

Figure 79. How to Place the Vials into the Sample Trays

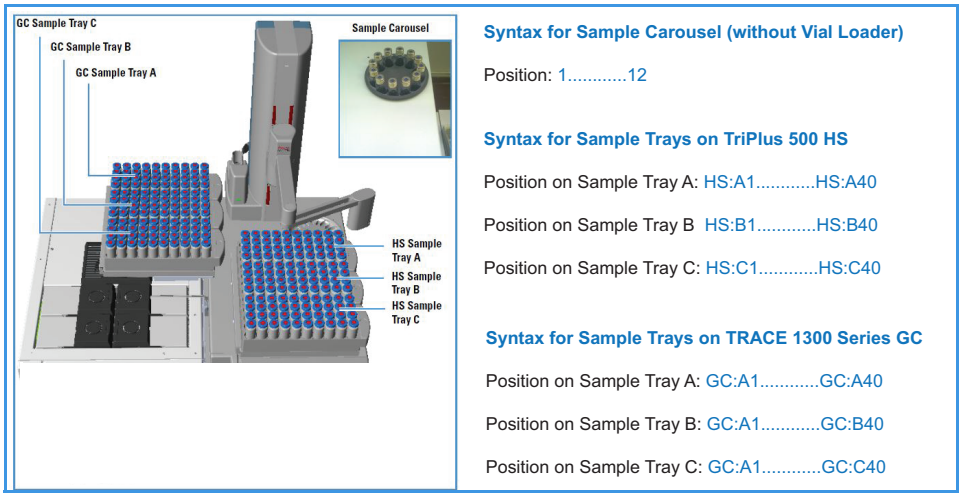


6. Create a sequence of samples.

   a. Open the **Sample Table** of the CDS in use.

   b. Compile the Sample Table using the **correct syntax** for identifying the position of the vials into the 12-seat rotating carousel or into the sample trays. See Figure 80.

Figure 80. Vial Sequence Syntax



7. Start the sequence of samples.
8. At the end of the sequence evaluate the analytical results.
9. If needed refine the method and repeat the sequence of samples.
10. At the end of the sequence re-evaluate the analytical results.
11. If necessary optimize the method until the results are satisfied, then save the method for future applications.

Analytical Troubleshooting General Guidelines

This sections provides general troubleshooting guidelines regarding the Headspace technique. See Table 4.

Table 4. Headspace Technique Points Regarding GC Effects - General Troubleshooting Guideline (Sheet 1 of 2)

Symptom or Error Message	Possible Cause	Action
No peaks	Needle bending or clogging	Check the integrity of the needle. Contact local service.
	Possible leaks in the sampling system	Check for leaks in the system including sample vials, sampling needle, sampling valve and GC column interface
Sample peaks or responses are not reproducible	Possible leaks in the sampling system	Check for leaks in the system including sample vials, sampling needle, sampling valve and GC column interface
	Possible issue with sample preparation	Verify the consistency of the sample preparation
	SSL module split flow unstable	Check the split flow stability
	Improperly crimped vials	Check vial cap by attempting to rotate manually. Loose caps might cause selective loss of more volatile components from sample. Adjust crimping tool correctly.
	Inadequate loop filling	Increase the pressure delta between the vial and the loop
	Poor choice of equilibration temperature or time	Increase the equilibration time Try shaking the sample to improve sample equilibration
	None or too low purge gas flow rate	Check the needle purge level
	Method parameters	Check recommended Method parameters
Ghost peaks	Sample carryover from previous analysis	Purge adequately the sampling valve and the loop Increase loop/sample path temperature
	Vial contaminated	Use pre-cleaned vials and proper septa

Table 4. Headspace Technique Points Regarding GC Effects - General Troubleshooting Guideline (Sheet 2 of 2)

Symptom or Error Message	Possible Cause	Action
Excessive carry-over between samples	None or too low purge gas flow rate	Check if the vent line filter is clogged
	Inadequate purging	Check **Needle purge level** value Set the function **Constant purge** to **On**
	Sample concentration too high	Reduce sample concentration Increase loop/sample path temperature
Low response	Inadequate incubation temperature	Increase sample incubation temperature
Peak distortion or tailing	Method parameters	Check recommended Method parameters
	Solvent not suitable for application	Check the boiling point of the solvent and try to change the solvent (when e.g. a high boiling point organic solvent is used as matrix)
	GC related parameters	Any parameter such as the injector, oven or detector temperature can contribute to bad peak shape Check the column type and connections
Unexplained chromatographic peaks	Dirty purge gas	Check the purge gas supply for impurities

7

Using the TriPlus 500 Web Interface

This chapter provides the instructions for using the TriPlus 500 Web Interface.

Contents

- Introduction
- Menu Bar
- Logging Out of the TriPlus 500 Web Interface

Introduction

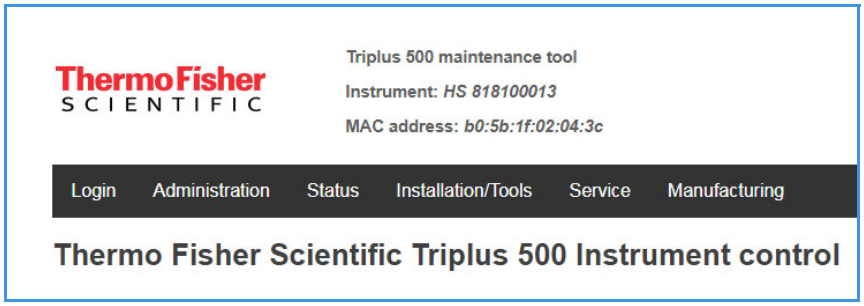
The TriPlus 500 Web Interface is a web based application used to check the network configuration, the status and the instrument control of the TriPlus 500 HS. Besides, the instructions for calibrating the tray holder, for performing the leak check and for testing the reading of the barcode label on the vial are also included.

❖ To access the TriPlus 500 Web Interface

1. Take note of the **Actual IP** address of the TriPlus 500 HS to control.
2. Open your Internet browser and type the actual IP address of the TriPlus 500 HS to check:

   http://actual IP address

   for example: http://169.254.250.4
3. Press **Enter** and wait for the Web Interface Main Page to display.



4. This page has three access levels:

- **User**
- **Service**
- **Manufacturing**.

  Each level is accessible with a specific **User name** and **Password**.

  The operator can ONLY use the **User** level.

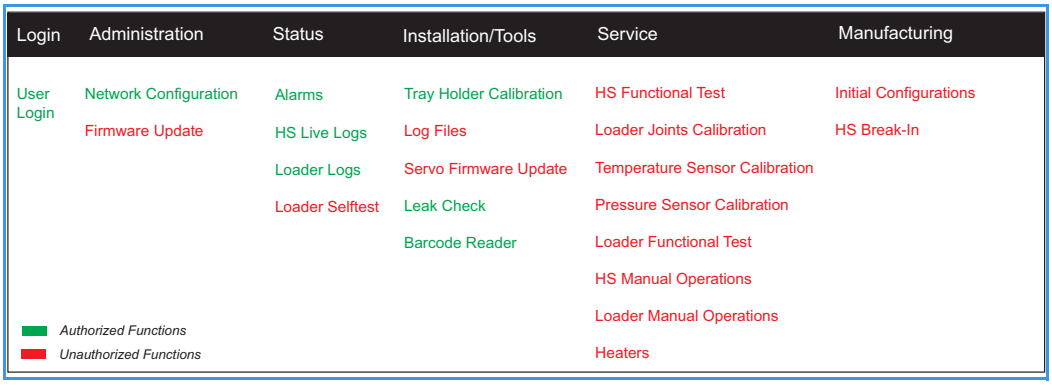
  Service level and **Manufacturing** level are accessible respectively by an authorized and trained Thermo Fisher Scientific Field Service Engineer (FSE) or by the Manufacturing Team previous the login with the relevant dedicated user name and password.

Menu Bar

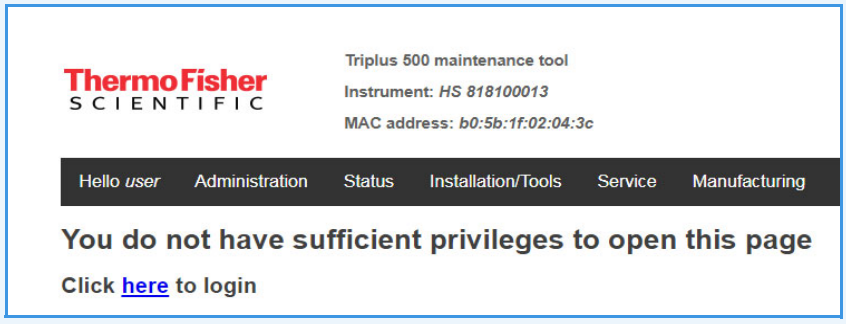
The menu bar of TriPlus 500 Web Interface includes the following six menus.

Each menu lists a series of functions available. All the functions are always visualized but the access is allowed only to a limited number, according to the level access.

The functions authorized for the **User** level are highlighted in green, the unauthorized functions in red.



Note When the operator selects an unauthorized function, the following message is visualized:



For details see the following sections:

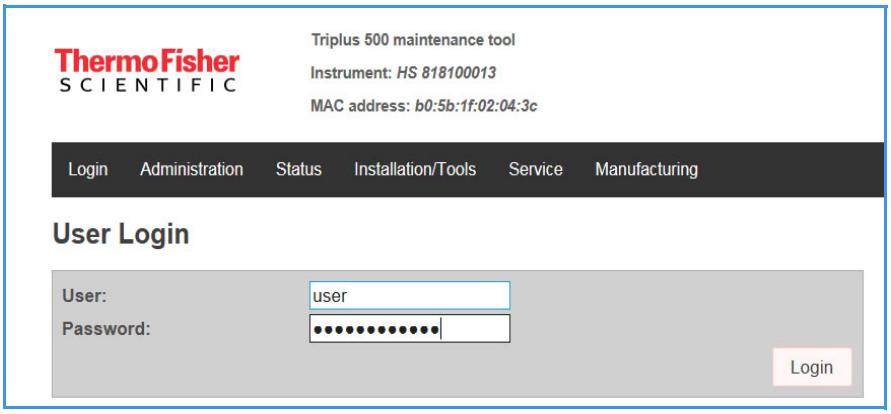
- "Login Menu" on page 114
- "Administration Menu" on page 114
- "Status Menu" on page 115
- "Installation/Tools Menu" on page 118
- "Service Menu" on page 122
- "Manufacturing Menu" on page 122

Login Menu

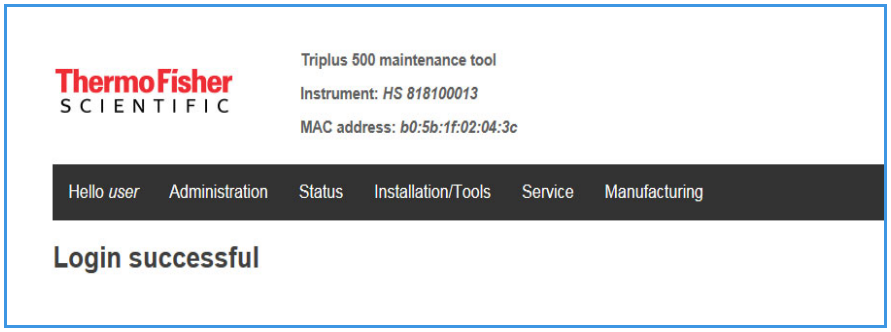
In the menu bar select **Login** and perform the **User Login**.

The operator must type the following **User name** and **Password**:

- User name = **user**
- Password = **ThermoFisher**

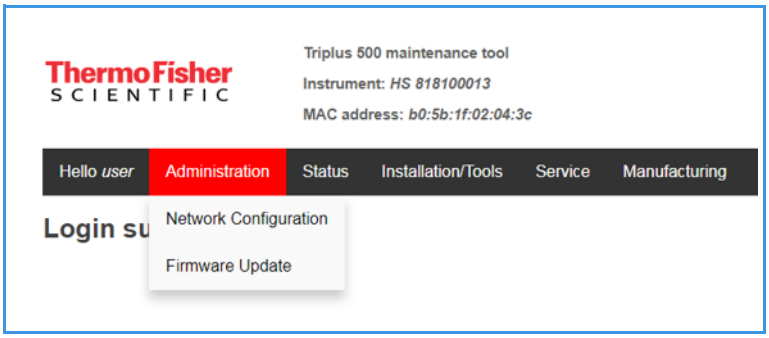


Press **Login**. If the login is correct the following page is visualized. The operator is now able to select the authorized functions.

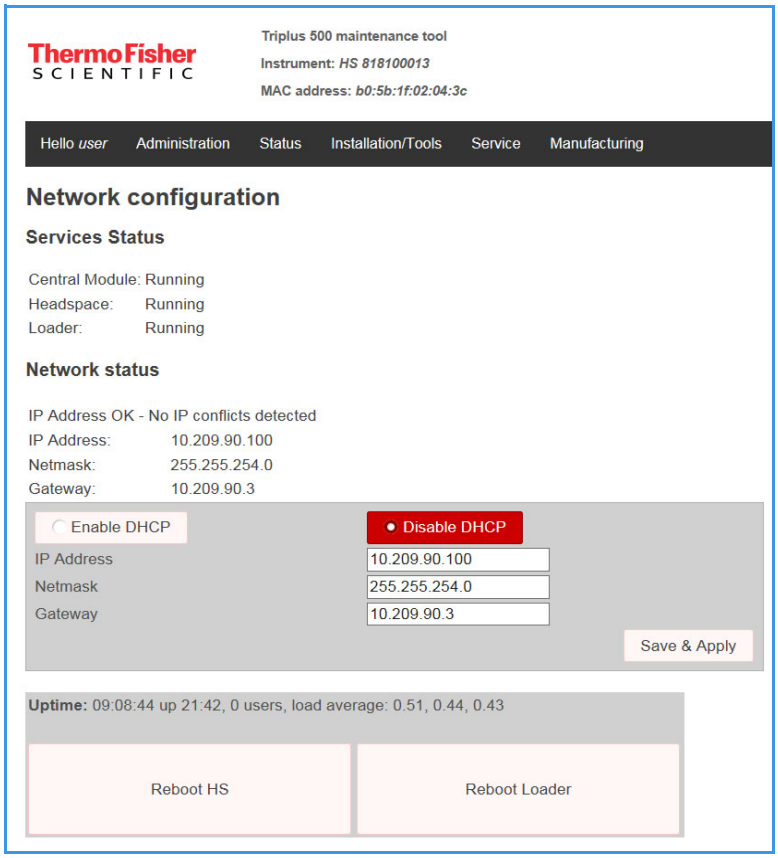


Administration Menu

Includes the following functions:



- **Network Configuration** — Check and /or reconfigures the network parameters of the TriPlus 500 HS.

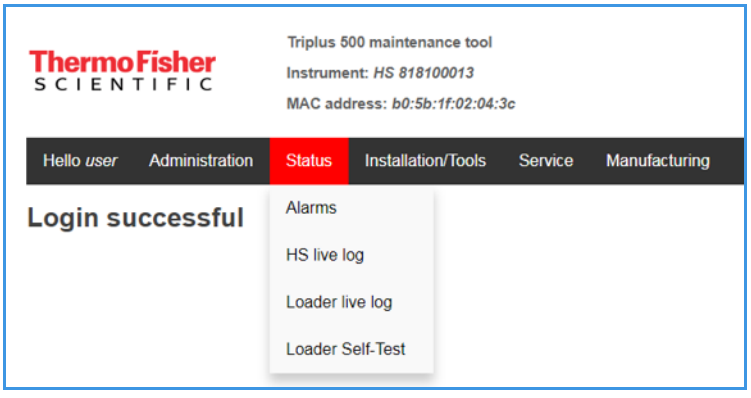


In case of re-configuration of the network parameters, save and apply the changes selecting **Save&Apply**, then **Reboot HS** and **Reboot Loader** when the Vial Loader is present.

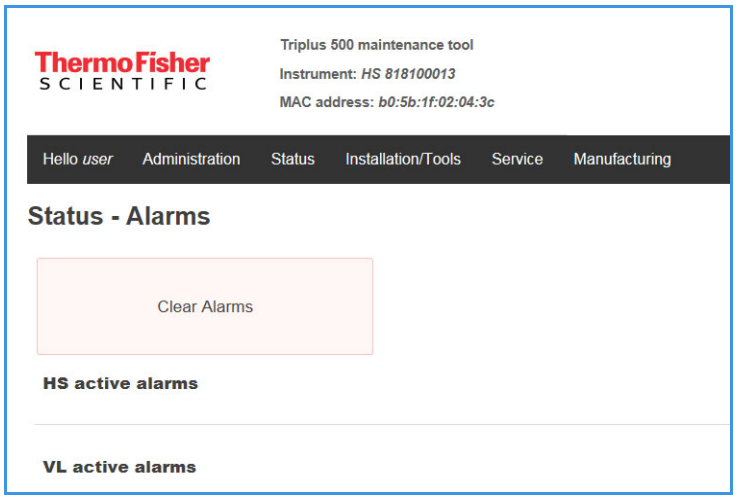
- **Firmware Update** — This function is unauthorized for the operator.

Status Menu

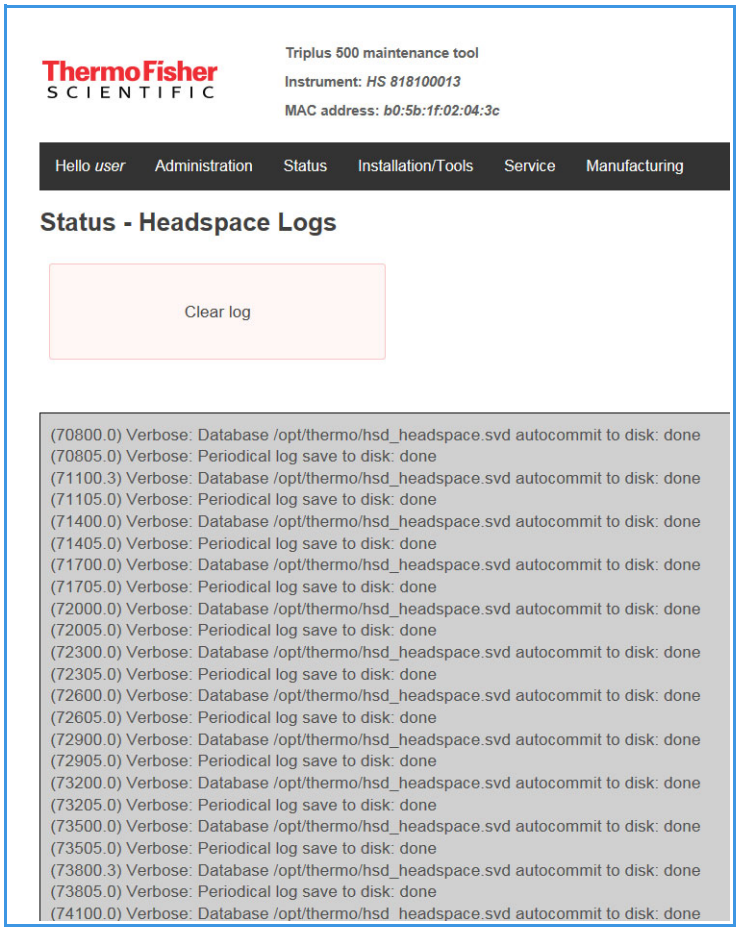
Includes the following functions:



- **Alarms** — Lists the HS sampler and Vial Loader active alarms that happened in the system. The operator can clear the list selecting **Clear Alarms**.

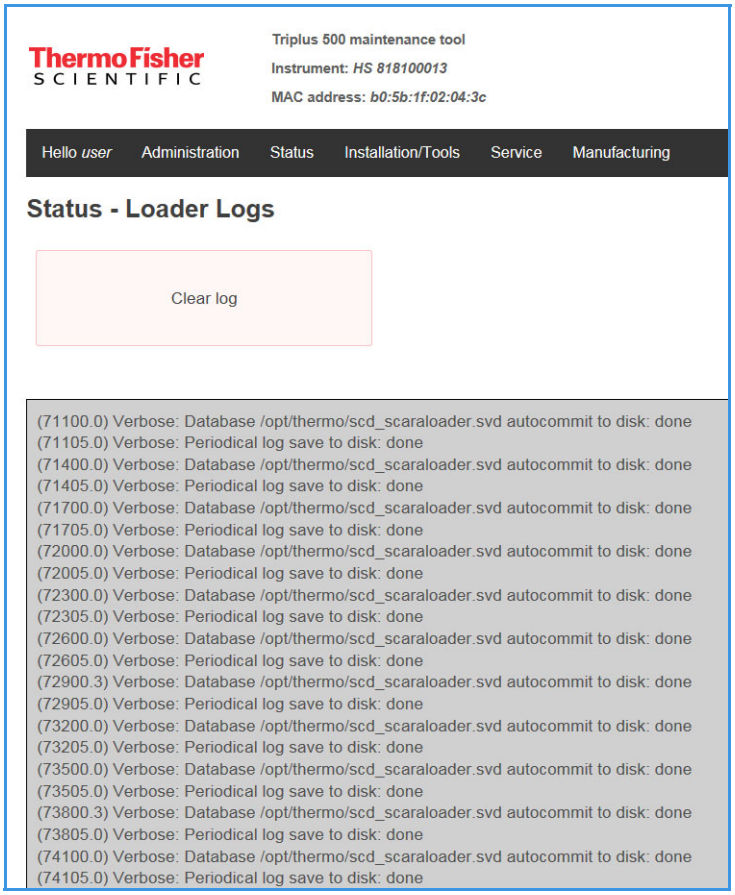


- **HS Live Logs** — Shows the operations the TriPlus 500 HS is performing.



To clear the list select **Clear log**.

- **Loader Logs** — Shows the operations the Vial Loader is performing.

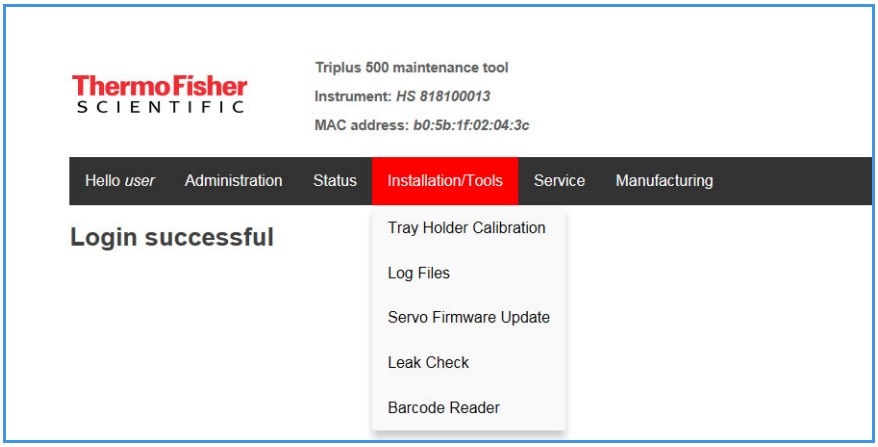


To clear the list select **Clear log**.

- **Loader Selftest** — This function is unauthorized for the operator.

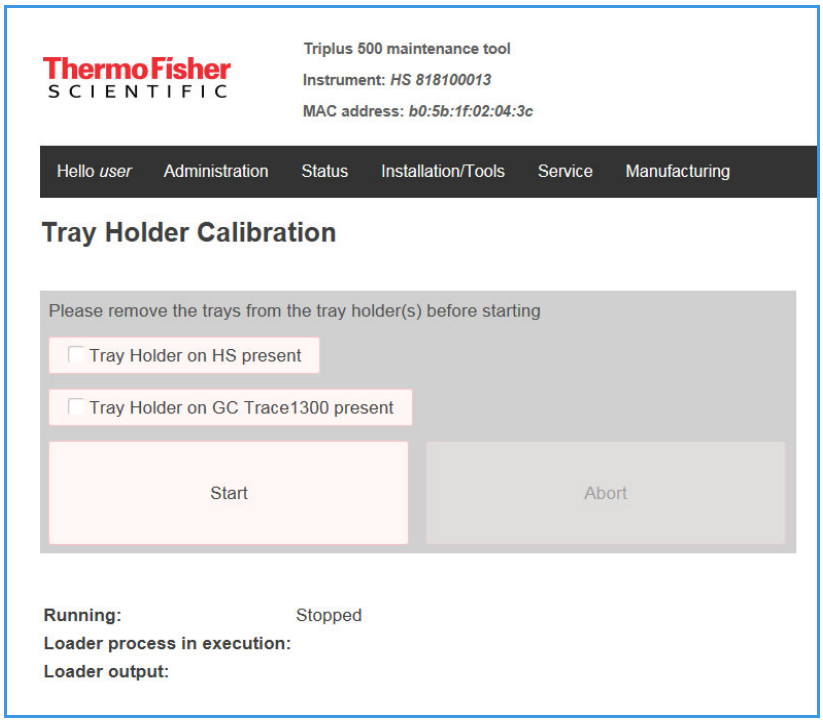
Installation/Tools Menu

This menu includes the following functions:

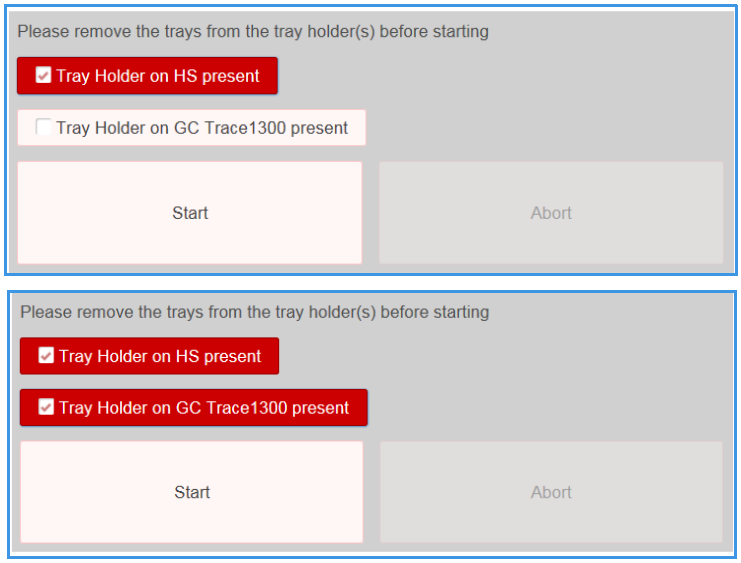


- **Tray Holder Calibration** —The purpose of this operation is to calibrate the motion of the Vial Loader on the holder plate without the sample trays installed.

  This operation is mainly required during the installation of the Vial Loader on the TriPlus 500 HS.



  Before starting, the system asks users to remove all the vial trays from the tray holder plate. When ready, select where the tray holders are present (HS sampler, GC, or both) by selecting the corresponding check box.

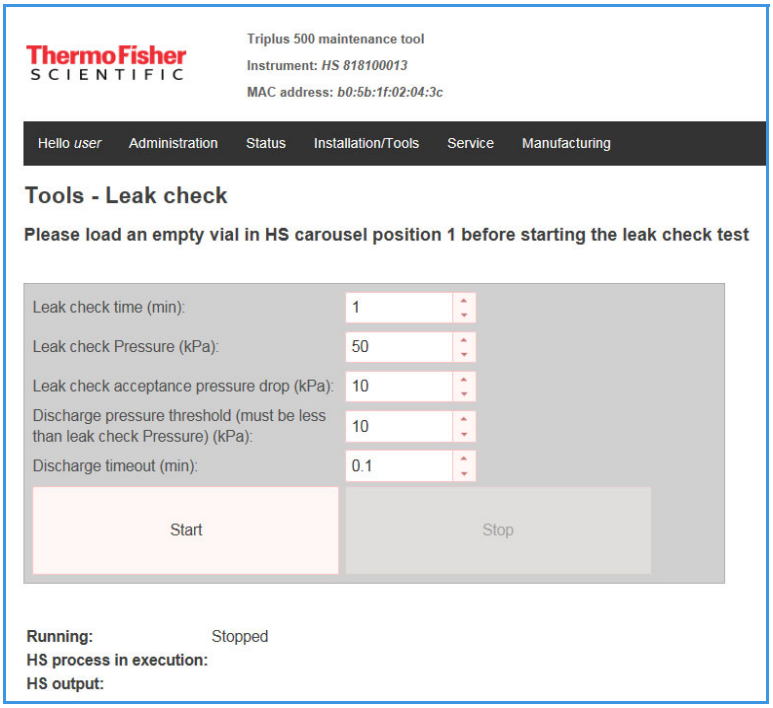


or

When ready select **Start**. The Vial Loader performs the automated calibration routine at the end of which the Vial Loader goes back in home position and the routine is stopped.

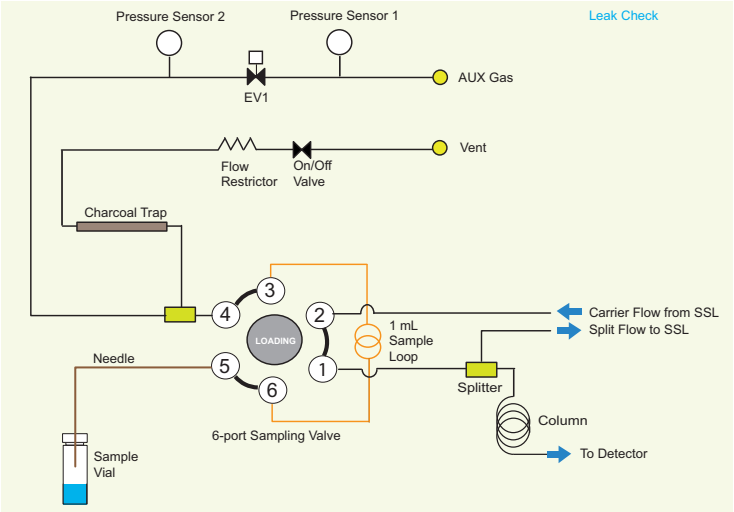
Note See also the section **Calibrating Tray Holder** in the *TriPlus 500 Headspace Sampler Hardware Manual*.

- **Log Files** — This function is unauthorized for the operator.
- **Servo Firmware Update** — This function is unauthorized for the operator.
- **Leak Check** — Allows the leak check of the system.



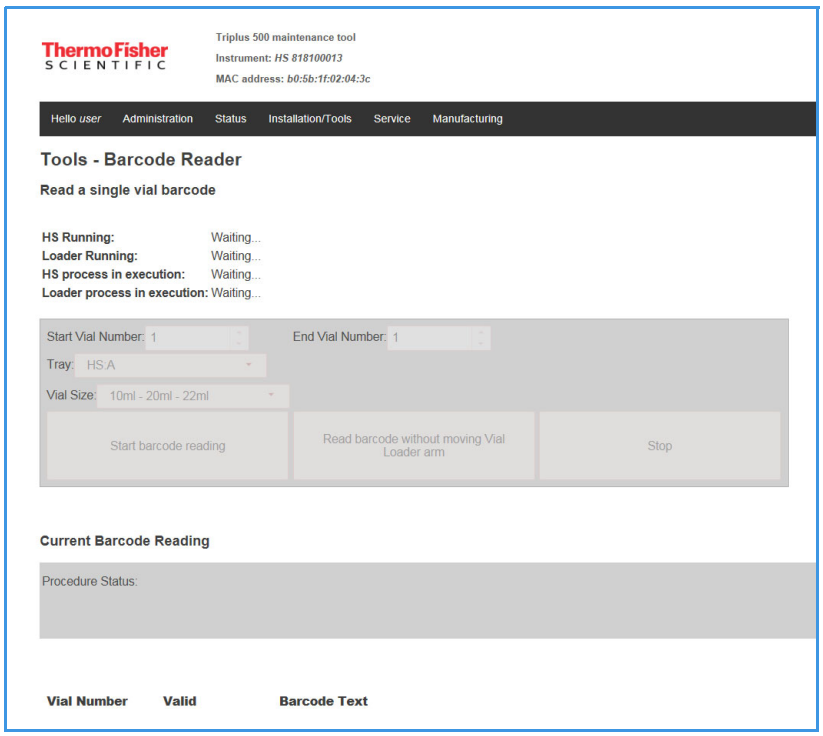
Before starting the leak check, the system asks users to load an empty vial into the position 1 of the HS carousel.

The operator can use the default parameters visualized the first time this function is selected, or modify them. To begin the leak check routine select **Start**.



EV1 and On/Off valve are closed. The system monitors the P2 decay, calculates the decay rate and compares with a set value to assess if there is a leak. At the end of the routine select **Stop**.

- **Barcode Reader** — Tests the reading of the barcode label on the vial. It is suggested to perform this test to verify the correct position of the barcode label on each sample vial before starting the analytical sequence of the samples.

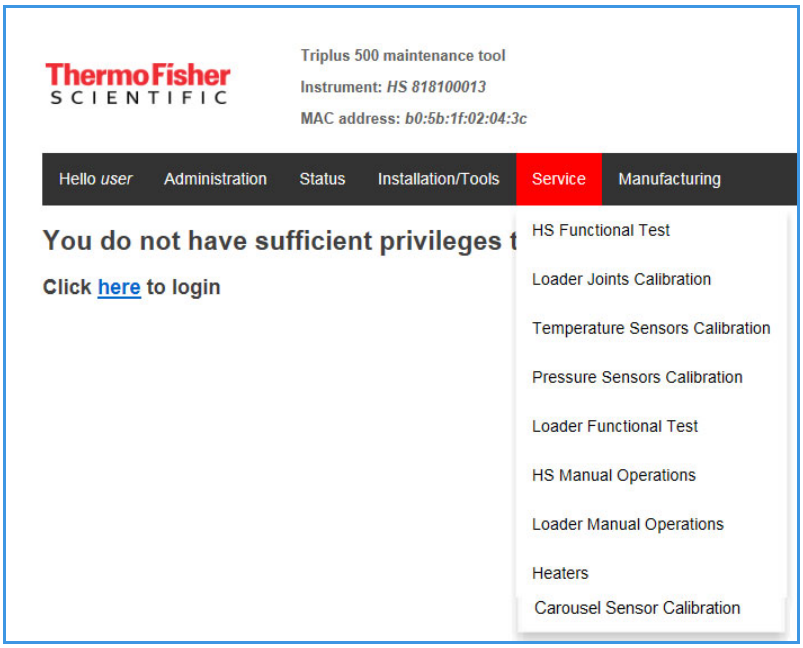


The operator can perform the test in **sequential** or **manual** mode.

- **Sequential mode** — Performs automatically the reading of the barcode labels on the vials placed into a sample tray.
  - Set the **Start Vial Number** and the **End Vial Number**. For example 1 and 30.
  - Select the **Tray** into which the vials are placed. For example Tray **A**.
  - Select the **Vial Size**. For example 20 mL.
  - Select **Start barcode reading**. The Vial Loader moves sequentially each vial on the barcode reader for the reading test. The result of each test is visualized in the bottom area of the menu page.
- **Manual mode** — Manual reads the barcode label on the vial in object.
  - The vial is manually placed on the barcode reader by the operator.
  - Select **Read Barcode without moving Vial Loader arm**. The result of the test is visualized in the bottom area of the menu page.

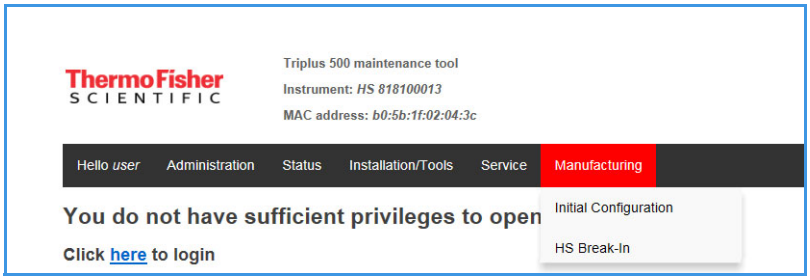
Service Menu

Shows the menu page but the functions listed are unauthorized for the operator.



Manufacturing Menu

Shows the menu page but the functions listed are unauthorized for the operator.



Logging Out of the TriPlus 500 Web Interface

To log out from the TriPlus 500 Web Interface, select **Hello *user*** from the bar menu then select **Logout**.

8

Ordering Parts

To order parts for the TriPlus 500 HS instrument, refer to the *TriPlus 500 Headspace Spare Parts Guide*.

Glossary

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

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

A

A ampere

ac alternating current

ADC analog-to-digital converter

B

b bit

B byte (8 b)

baud rate data transmission speed in events per second

C

C Carbon

°C Celsius

CDS Chromatography Data System

CIP Carriage and Insurance Paid To

cm centimeter

CPU central processing unit (of a computer)

<Ctrl> control key of the keyboard

D

d depth

DAC digital-to-analog converter

dc direct current

DS data system

E

EMC electromagnetic compatibility

ESD electrostatic discharge

F

f femto

°F Fahrenheit

FOB Free on board

FSE Field Service Engineer

ft foot

G

g gram

GC gas chromatography- gas chromatograph

GND electrical ground

H

h height

h hour

H Hydrogen

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

He Helium

HS Headspace

HV high voltage

Hz hertz (cycles per second)

I

ID inside diameter

IEC International Electrotechnical Commission

Impulse See *transient*

in. inch

I/O input/output

K

k kilo (10^3 or 1024)

K Kelvin

kg kilogram

kPa kilopascal

L

l length

L liter

LAN Local Area Network

lb pound

LED light-emitting diode

M

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

M mega (10^6)

µ micro (10^{-6})

min minute

mL or ml milliliter

mm millimeter

MS mass spectrometry-mass spectrometer

m/z mass-to- charge ratio

N

n nano (10^{-9})

N Nitrogen

negative polarity The inverse of a detector signal polarity.

nm nanometer

O

OD outside diameter

Ω ohm

P

p pico (10^{-12})

Pa pascal

PCB printed circuit board

PN part number

psi pounds per square inch

R

RAM random access memory

<Return> <Return> key on the keyboard

RF radio frequency

ROM read-only memory

RS-232 industry standard for serial communication

S

s second

sag See *surge*

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

SOP Standard Operating Procedures

SSL split/splitless injector

source current The current needed to ignite a source, such as a detector lamp.

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

T

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

V

V volt

Vac volts, alternating current

Vdc volts, direct current

VGA Video Graphics Array

VL Vial Loader

W

w width

W Watt

When a unit of measure has a quotient (e.g. Celsius degrees per minute or grams per liter) this can be written as negative exponent instead of the denominator:

For example:
$°C\ min^{-1}$ instead of °C/min
$g\ L^{-1}$ instead of g/L