TurboMatrix
Headspace Sampler and
HS 40/110 Trap
User's Guide

Release History

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Notices

Copyright Information

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Introduction

In headspace sampling the sample (either a gas, a liquid, or a solid) is placed into the headspace vial, which is closed immediately and equilibrated and pressurized. An aliquot is then withdrawn from the closed vial and transferred directly to the gas chromatographic system or pre concentrated on a trap for focusing (in headspace trap only).

The HS is an automatic sampler for headspace analysis. Three models of the HS are available, as are various accessories that enhance the operation of your system.

TurboMatrix Headspace

The HS-16 provides automated headspace analysis of up to 16 vials (not available in the trap functionality). Standard PerkinElmer vials can be loaded into the vial magazine. A single vial oven allows you to thermostat your sample before injection onto the GC via the heated transfer line. You can upgrade the HS-16 to an HS-40 or an HS-110.

Up to 40 vials can be loaded into the magazine of the HS-40. The oven can accommodate up to 12 vials and overlapping thermostatting allows the instrument to obtain maximum sample throughput. An optional shaker device is available to reduce equilibration time. You can upgrade the HS-40 to an HS-110.

NOTE: The HS-40 and HS-110 have exactly the same function except for the number of vials.

The HS-110 can accommodate up to 110 vials. The 12-vial oven is standard and overlapping thermostatting allows the instrument to obtain maximum sample throughput. The advanced automation system can be programmed to run series of vials according to preset methods. The HS-110 also comes standard with BCD output capability, which is optional on the other models.

NOTE: The 12-vial oven is in reality a 15-vial oven but the software only allows up to 12-vial simultaneous loading and overlapping thermostatting.

Programmed pneumatic control (PPC) provides the electronic control of pressures and flows for inlet, and auxiliary gases. This option can be installed on any one of the above models to automate

gas handling and delivery in the HS. PPC comes standard on the HS 40/110 trap. High pressure sampling is supplied as part of the PPC option.

HS 40/110 Trap

NOTE: The Trap model is offered with two vial options for either 40 or 110 but for the purposes of this Users Guide it will be referred to as the HS 40/110 Trap.

The HS 40/110 Trap includes the ability to prioritize sample vials containing rush samples during an active sequence and a time-saving, pre-programmed shut-down and wake-up mode that allows for complete unattended operation.

The HS 40/110 trap improves headspace detection limits. It allows for the extraction of the full vapor content of the sample vial into the trap to the GC and subsequent transfer.

European Union Industrial Environment

230V / 50Hz. TurboMatrix headspace instruments manufactured for use in the European Union are intended for the industrial environment. The instrument is to be connected to a mains power network supplied from a high or medium-voltage transformer dedicated for the supply of an installation feeding a manufacturing or similar plant.

Industrial environments are characterized by the existence of one or more of the following conditions:

- industrial, scientific and medical (ISM) apparatus are present
- heavy inductive or capacitive loads are frequently switched
- currents and associated magnetic fields are high

These are the major contributors to the industrial electromagnetic environment and as such distinguish the industrial from other environments. The instrument is not intended for connection to a public mains network supplying residential, commercial and light-industrial locations.

About this Manual

This manual is an integral part of your automatic headspace sampler product. It begins with unpacking and general safety information in Chapter 1. Installation and setup are described in Chapter 2. Operation of the instrument is covered in Chapter 3. Optional accessories are described in Chapter 4 and method development is offered in Chapter 5. A routine maintenance schedule and the related procedures are discussed in Chapter 6. Chapter 7 contains lists of possible fault and error messages and some basic troubleshooting procedures.

If you find an error in this manual or have any comments or suggestions, please let us know so that we can correct the mistake or improve the manual. Contact information is provided in the Appendix A.

Conventions in this Manual—In the text you will also find various warnings and notes.

Warnings, other cautionary information and notes are denoted in the text as follows:

WARNING *This indicates a warning. We use the term warning to inform you about situations that could result in personal injury to yourself or other persons.*

CAUTION *This indicates a caution note. We use the term caution to inform you about situations that could result in serious damage to the instrument or other equipment.*

NOTE: The term Note indicates any significant information that can help you avoid false analytical results or deterioration in instrument performance.

Other Manuals and Reference Material—Before you install or use your automatic headspace sampler, and in order to get the best results, you should be familiar with all the instruments in the system and know how to operate them. You must also be aware of the safety procedures that are in effect in your laboratory.

Details of gas chromatography are not covered in this manual, but a working knowledge of your gas chromatograph and the theory of gas chromatography are required to operate this instrument. Refer to the documentation supplied with your GC.

Some accessories are supplied with separate installation guides and user manuals. If you have any of these accessories installed, please refer to the associated manual for detailed information on operation, troubleshooting and maintenance of the accessory.

For information on the TurboMatrix Control Software refer to the TurboMatrix HS Control Software User's Guide.

A list of reference materials covering the theory of headspace chromatography and various applications is provided in this manual. Refer to Appendix C.

Unpacking the Instrument

Keep the original packing materials for possible future storage or reshipment.

Observe the following information when unpacking the instrument:

1. Open the carton at the top, folding the flaps outward. On top of the instrument is a foam insert containing various accessories.
2. Lift out the insert and accessories using the hand grips at the side.
3. Lift out the instrument.
4. Check that all ordered parts have been received undamaged (see Tables 1 and 2).

The following items should be included with your HS instrument:

Part No.	Description	Qty.
00090652	Septum Kit, Fairprene (50/Pkg.)	1
09903157	Nut 1/16"	1
L1003026	Graphite Vespel Ferrule, 1.6 mm ID	3
04966624	Wrench Open End	1
04972433	Graphite Ferrule, 0.5 mm ID	3
B0131410	Tool O-Ring Assembly	1
B0147449	O-Ring Extractor Tool	1
B0151737	Vial Gauge	1
B0500843	Spigot Key	1
B0503956	Glass Lined Tube Adapter	1
B0505266	HS Injector Adapter	1
B0510403	Transfer Line Cover	1
09907233	Wrench Open End (1/4 in x 5/16 in)	1
M0415330	Top Seal Changing Tool	1
M0417030	Terminal Block Plug, 14 Poles	1
N1011206	HS Start/Ready Cable	2
N9301357	Fused Silica Transfer Line, 0.32 ID	1
N9301376	Wafer-Ceramic Cutter (10/Pkg)	1
09904956	Rheodyne Nut, 1/16 in	1
M0417002	Fuse 5 x 20 MM 10A 250V, TD	2
M0417038	Fuse 5 x 20 MM 5@ 250V, TD	2
M0413401	Users Manual	1

Table 1 TurboMatrix 16, 40, 110 Headspace Shipping Kit (Part No. M041-3403)

The following items should be included with TurboMatrix Trap.

Part No.	Description	Qty.
00090652	Septum Kit	1
04966624	Double Ended Wrench	2
L1003026	Graphite Vespel Ferrule, 1.6 mm ID	3
M0413628	Air Monitor Trap	1
09903392	Nut Union 1/16	1
09900105	Graphite Vesp Ferrules 1/16 x 0.5 mm	3
09926067	O-Ring 0.0145 ID x 0.070 WD	5
B0131410	O-Ring Tool Assembly	1
N6701077	Trap Removal Tool, Tygon	
B014-7449	O-ring Extract Tool	1
B1511737	Vial Gauge	1
B0500843	Spigot Key for All Systems	1
B0505266	HS Adapter Injector	1
B0510403	HS 40/110 Transfer Line Cover	1
L4271302	Ferrule Trap Tube, PTFE	5
M0413401	Users Manual	1
M0415010	Locking Nut	1
M0415330	Top Seal Changing Tool	1
M0417002	Fuse 5 x 20mm 10.0A 250V, TD	2
M0417030	Terminal Block Plug	1
M0417038	Fuse 5 x 20mm 5.0A 250V, T	2
N1011206	Cable Assembly for All Systems	2
N6701053	Washer, non-trap	1
09200061	Wrench, 5/8 in	1

Part No	Description	Qty
N6701079	Valco Ferrule Removal Tool Kit	1
N9301357	Empty Fused Silica Tubing 0.32	1
N9301376	Wafer Ceramic Cutter	1

Table 2 HS 40/110 Trap Shipping Kit (Part No. N670-0116)

If any of these items are missing or are damaged, please contact PerkinElmer immediately. In the event of damage, file an immediate claim with the carrier and report the matter to your PerkinElmer office.

Symbols Used on the Instrument

Indicates alternating current.

Indicates the off position of the main power switch

Indicates the on position of the main power switch

Indicates the Protective Conductor Terminal

Indicates Hot Surface

Indicates Risk of Electric Shock

Indicates an earth ground terminal

Caution, risk of danger
Documentation must be consulted to determine the nature of the potential hazard and any actions which have been taken.

Safety Information

WARNING

*Do **not** attempt to make adjustments, replacements or repairs to this instrument except as described in the accompanying user documentation.*

NOTE: This equipment requires no specified inspection or preventive maintenance to ensure the continuous functioning of its safety features.

Please read this section carefully before beginning operation of the HS and pay particular attention to any advice it contains concerning potential hazards that may arise from the use of the instrument.

This manual contains information and warnings that must be followed by the user to ensure safe operation.

Possible hazards that could harm the user or result in damage to the instrument are clearly stated throughout this manual.

The advice is intended to supplement, not supersede, the normal safety code of behavior prevailing in your country.

WARNING

Warning: Toxic Fumes-Fume Ventilation System
Without adequate ventilation, potential toxic vapors can build up in the laboratory. your laboratory must have a reliable fume ventilation system before you use this instrument.

WARNING

Warning: Explosive Atmosphere
This instrument is not designed for operation in an explosive atmosphere.

WARNING

*This equipment **must** be used in a manner specified by this manual. Otherwise the protection provided by the equipment may be impaired.*

Electrical Safety

Electrical Protection—The following precautions have been taken to provide electrical protection:

Pollution Degree	This equipment will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 as defined in IEC 61010-1.

Table 3 Electrical Protection

If you suspect for any reason that the instrument is not electrically safe for use, do not operate it and secure it against any unauthorized operation. Have the equipment inspected by a PerkinElmer Service Engineer or similarly qualified person.

The instrument is likely to be electrically unsafe when it:

- Has been subjected to severe transportation stresses.
- Shows visible damage.
- It has been subjected to prolonged storage under unfavorable conditions.

WARNING

- *This instrument must be grounded for maximum protection against electric shock. Intentional interruption or disconnection of the protective conductor (earth ground) inside or outside the instrument is prohibited.*
- *Hazardous voltages are present in the instrument. To prevent the risk of electrical shock, the line cord must be unplugged from the AC outlet before removing any instrument covers or panels requiring the use of a tool.*
- *Hazardous voltages remain present in the instrument even when it is switched off, but not disconnected from the AC outlet.*
- *Wait at least one minute before opening the instrument after disconnecting it from the AC outlet. Internal capacitors may remain charged for several seconds after the instrument has been switched off.*
- *Do not operate the instrument with any covers or parts removed.*
- *Servicing must be carried out only by a PerkinElmer Service Engineer or similarly qualified person.*
- *Only persons trained and qualified in the use of the HS are authorized to carry out adjustments and maintenance described in this manual.*

Line Cord—Use only approved line cords with a protective ground conductor (green or green/yellow) to ensure safe operation. The line cord must be connected to a correctly installed AC power outlet with a protective ground/earth terminal that conforms to the local safety code.

If the line cord plug has been installed by someone other than a PerkinElmer service representative, ensure that it is wired correctly.

Terminal	Cord Lead Colors	
	International	**USA**
Live	Brown	Black
Neutral	Blue	White
Protective Conductor (earth/ground)	Green/Yellow	Green

Table 4 AC Line Cord Connections

Servicing of incoming AC power line components in your laboratory must be performed by a licensed electrician.

Fuses—Use only fuses with the required current rating and of the specified type for replacement

.

WARNING

For protection against fire hazard, replace only with the same type and rating of fuse.

Voltage @ 50/60 Hz	Fuse (250V rated)	Part Number
100	T 10A	M041-7002
120	T 10A	M041-7002
220	T 5A	M041-7038
230-240V	T 5A	M041-7038

Table 5 HS Replacement Fuses

Electromagnetic Compatibility (EMC)

Europe

All information concerning EMC standards is in the Declaration of Conformity, and these standards may change as the European Union adds new requirements.

This autosampler has been designed and manufactured, having regard to the state of the art, to ensure that:

a. the electromagnetic disturbance generated does not exceed the level above which radio and telecommunications equipment or other equipment cannot operate as intended;

b. it has a level of immunity to the electromagnetic disturbance to be expected in its intended use which allows it to operate without unacceptable degradation of its intended use.

United States (FCC)

This product is classified as a digital device used exclusively as industrial, commercial or medical test equipment. It is exempt from the technical standards specified in Part 15 of the FCC rules and Regulations based on Section 15.103 (c).

Use of this product in residential areas may cause interference and such use should be avoided unless special measures are taken by the user to restrict emissions to a level that allows the reception of broadcast emissions.

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CAUTION	*The TurboMatrix contains protective circuitry. Contact PerkinElmer Service before performing AC line tests.*

Mechanical Hazard

When working with the instrument, please observe the following:

- Keep your hands, clothing and other objects away from the moving parts of the instrument.
- Do not operate the instrument without the safety covers.

- Do not touch any moving parts.
- Do not load or unload the magazine while it is in motion.

Chemicals

Some chemicals used with this instrument may be hazardous or may become hazardous after completion of an analysis. Do not store, handle, or work with any chemicals or hazardous materials unless you have received appropriate safety training and have read and understood all related Material Safety Data Sheets (MSDS). Use, store, and dispose of chemicals that you require for your analyses in accordance with the manufacturer's recommendations and local safety regulations. You must comply with all federal, state, and local laws related to chemical storage, handling, and disposal.

MSDS sheets provide information on physical characteristics, precautions, first aid, spill clean up and disposal procedures. It is good practice to familiarize yourself with the information on the MSDS sheets before using any chemical substances.

You must work under a suitable hood when handling and mixing certain chemicals. The room in which you work must have proper ventilation and a waste collection system. Always wear appropriate safety attire (full-length laboratory coat, protective glasses, gloves, etc.), as indicated on Material Safety Data Sheets.

Do not sample carbon disulfide or other compounds with a self-ignition point of 100 °C or less. Refer to the MSDS sheets for compound-specific information.

These types of compounds should not be sampled due to the risk of self-ignition of the CS_2 in the thermostatting oven or at other heated parts of the system.

WARNING

The responsible body (e.g., Laboratory Manager) must take the necessary precautions to ensure that the surrounding workplace is safe and that instrument operators are not exposed to hazardous levels of toxic substances (chemical or biological) as defined in the applicable Material Safety Data Sheets (MSDS) or OSHA, ACGIH, or COSHH documents.

OSHA: Occupational Safety and Health Administration (United States)

ACGIH: American Conference of Government Industrial Hygienists (United States)

COSHH: Control of Substances Hazardous to Health (United Kingdom)

If you are working with volatile solvents, toxic substances, etc., you must provide an efficient laboratory ventilation system to remove vapors that may be produced when you are performing analyses.

WARNING

You must vent hazardous fumes and vapors. Disposal of wastes must be in accordance with all national, state and local health and safety regulations and laws.

Your laboratory should have all of the equipment ordinarily required for the safety of individuals working with chemicals: fire extinguishers, first-aid equipment, safety shower and eyewash fountain, spill cleanup equipment, etc.

WARNING

*Due to the explosive nature of hydrogen, **NEVER** use hydrogen gas as a carrier gas in any GC headspace system.*

Compressed Gases

WARNING

High pressure gas cylinders can be dangerous if mishandled or misused. Always handle gas cylinders with caution and observe your local regulations for the safe handling of gas cylinders.

Handling Compressed Gas—Handle cylinders of compressed gas with care, in accordance with local regulations. We recommend that gas cylinders be located outside the laboratory and the gases fed to

the laboratory through approved gas supply lines. Use only approved tubing, connectors, and regulators for gas supply lines.

Cylinders of compressed gas, such as the carrier gas and calibration gas, must be handled with care.

Please observe the following handling procedures:

- Ensure each cylinder is clearly labeled.
- Do not store cylinders in hazardous locations. Store cylinders in an upright position away from possible sources of heat or sparks.
- Do not heat the cylinders or expose them to direct sunlight. The cylinders may rupture at high temperatures.
- Do not mutilate cylinders.
- Do not drag or roll cylinders. Large cylinders should only be moved on carts designed for compressed gas cylinders. Do not move cylinders without the valve protection cap in place.
- Always secure cylinders before removing the cylinder valve protection cap and before connecting the regulator and adapter tubing.
- Use only the specified regulator for the carrier gas cylinders.
- Never plug, obstruct or tamper with safety relief devices.
- Wear safety glasses and ear protection when working with compressed gases. When using hydrogen or methane, special care must be taken to avoid the build-up of explosive gas mixtures in the GC oven or interior of HS system.
- Ensure that all hydrogen line couplings are leak-free and do not allow hydrogen to be vented inside the oven.

WARNING

*Do **not** use H_2 with HS systems.pressure venting makes this very dangerous.*

Regulators for Compressed Gases—Use only approved regulators for gas supply lines and observe the following:

- Use only the specified regulator for carrier and calibration gas.

Confirm regulator type and material with your specialty gas supplier.

- Mark each regulator with its intended service and never use a regulator for more than one service. Do not change gas service, or adapt equipment without consulting your gas supplier.
- Ensure regulator construction materials are compatible with the gas, and that the cylinder pressure gauge will withstand the cylinder pressure.
- Never use the regulator as a shut-off valve. Close the cylinder valve when it is not in use.
- Do not subject the regulator to an inlet pressure greater than recommended.
- Do not move or detach the regulator when it is pressurized or when it is in use.
- Before connection, ensure the cylinder valve and the regulator CGA connection are clean.
- When connecting a regulator to a large gas cylinder turn the valve on the cylinder clockwise to close the cylinder. Turn the regulator off. Open the cylinder valve slowly and check for leaks. Adjust the delivery pressure and then open the regulator outlet valve.

Adequate Ventilation—If compound threshold limit values (TLVs) are exceeded, you should use a fumehood to prepare samples and vials and provide adequate ventilation for the vial venting option.

You must also vent the GC detector and split vent ports. Use flexible tubing fitted with adapters to vent these ports to a fumehood or outdoors. The pressure set by the method will determine the outlet flow rates at these ports.

Extreme Temperatures

High Temperatures—The heated zones (oven, needle, transfer line in trap) of the headspace sampler can generate temperatures up to 210 °C (to 400 °C in trap). Do not touch any part of the oven, or recently unloaded vials until they have cooled down to room temperature.

Low Temperatures—Liquid nitrogen is used as a cooling medium in conjunction with the cryofocusing accessory (TurboMatrix 40/110

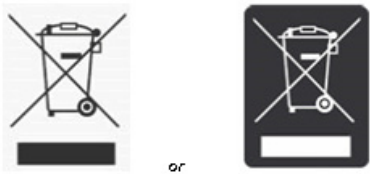
Headspace only). Make sure the following safety measures are observed when dealing with liquefied gases:

- Do not touch the liquefied gases.
- Always wear protective clothing (gloves, face mask, safety glasses) when handling liquefied gas.
- Secure the Dewar vessel so that it cannot tip over.
- Protect the Dewar vessel from any damage and from sources of heat. The Dewar vessel must be fitted with a pressure relief valve.
- Cold, vaporized nitrogen is heavier than air and can thus collect in low lying parts of the laboratory, representing a suffocation risk.

Waste Disposal

If the materials being sampled are hazardous in any way, you must treat the collected samples, and the vials that contained them, as hazardous waste. Used vials and seals may contain small amounts of the substances that were analyzed and may thus constitute a chemical or biological hazard. Refer to your local safety regulations for proper disposal procedures.

WEEE Instructions for PerkinElmer Products



A label with a crossed-out wheeled bin symbol and a rectangular bar indicates that the product is covered by the Waste Electrical and Electronic Equipment (WEEE) Directive and is not to be disposed of as unsorted municipal waste. Any products marked with this symbol must be collected separately, according to the regulatory guidelines in your area.

The objectives of this program are to preserve, protect and improve the quality of the environment, protect human health, and utilize natural resources prudently and rationally. Specific treatment of WEEE is indispensable in order to avoid the dispersion of pollutants into the recycled material or waste stream. Such treatment is the most effective means of protecting the customer's environment.

Requirements for waste collection, reuse, recycling, and recovery programs vary by regulatory authority at your location. Contact your local responsible body (e.g., your laboratory manager) or authorized representative for information regarding applicable disposal regulations. Contact PerkinElmer at the web site listed below for information specific to PerkinElmer products.

Web address:

http://las.perkinelmer.com/OneSource/Environmental-directives.htm

For Customer Care telephone numbers select "Contact us" on the web page.

Products from other manufacturers may also form a part of your PerkinElmer system. These other producers are directly responsible for the collection and processing of their own waste products under the terms of the WEEE Directive. Please contact these producers directly before discarding any of their products.

Consult the PerkinElmer web site (above) for producer names and web addresses.

Sample Vials

CAUTION *Using sample vials, caps and septa other than those supplied by PerkinElmer may result in improper operation of the TurboMatrix Headspace or Trap Headspace Sampler. Damage to the instrument and/or loss of sample materials or data resulting from the use of sample vials, caps and septa* ***not*** *supplied by PerkinElmer may occur.* ***The subsequent service visit to remedy the situation, caused by the choice to use these non-PerkinElmer sample vials, caps and septa is not included under your warranty or service contract agreement.*** *Your Service Engineer can discuss the benefits of using only PerkinElmer sample vials, caps and septa.*

PerkinElmer sample vials and patented safety closures are carefully selected. They are under permanent quality control. If you use vials and closures from other manufacturers the instrument may not function correctly; if a vial should rupture, you risk injury from glass splinters and possible damage to the instrument.

WARNING *Using sample vials, caps and septa other than those supplied by PerkinElmer can result in damage to the instrument and/or injury to the user if they attempt to remove the broken glass vials.*

- It is possible that a few vials in a batch are not within tolerance. If in doubt, we recommend that you check the sample vials using the vial gauge (Part No. B015-1737) provided with the instrument.
- Observe the maximum filling volume of 15 mL for liquid samples when using 22 mL sample vials.
- Check the safety closure for reliable tightness after sealing the sample vial.
- Use only felt tip pens to mark sample vials. Adhesive labels may jam in the oven.

- Sample vials just unloaded from the thermostatted oven into the magazine can be very hot and may still be under pressure. Cool and vent the sample vials before you open or dispose of them. Use the PerkinElmer venting tool (P/N B009-9590). TurboMatrix units provide optional automatic venting.

- Use only the cap removing tool to open the sample vials (P/N B003-8135) under the fumehood.

- Carefully check the sample vials after cleaning for hairline cracks and damage before reuse. Do not use unsuitable vials. Replace the reused sample vials regularly. PerkinElmer vials are guaranteed for single use only.

Cleaning and Decontamination

Before using any cleaning or decontamination methods except those specified by PerkinElmer, users should check with PerkinElmer that the proposed method will not damage the equipment.

Decontamination

Customers wishing to return instrumentation and/or associated materials to PerkinElmer for repair, maintenance, warranty or trade-in purposes are advised that all returned goods must be certified as clean and free from contamination.

The customer's responsible body is required to follow the "Equipment Decontamination Procedure" and complete the "Certificate of Decontamination". These documents are available on the PerkinElmer public website:

http://las.perkinelmer.com/OneSource/decontamination.htm

If you do not have access to the internet and are located in the U.S., call toll free at 1-800-762-4000 or (+1) 203-925-4602, 8:30 a.m. - 7 p.m. EST and speak to Customer Support.

In Canada, call toll free 800-561-4646 and speak to Customer Support.

If you are located outside of the United States or Canada, please call your local PerkinElmer sales office for more information.

Cleaning the Instrument

Exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution. Do not use abrasive cleaners or solvents.

Installation 2

Introduction

Before the instrument arrives, you may have received a pre-installation checklist to ensure that your lab is setup correctly. If you did not receive the checklist, refer to the *Pre-Installation Checklist* below to ensure that you have enough space and that electrical and gas supplies meet the requirements of the HS, the GC and all of the related support equipment.

Normally, the instrument is installed and set up by a PerkinElmer service engineer.

Installation consists of seven steps:

1. Unpack the HS and place it correctly on the lab bench.
2. Connect gas and electrical supplies to the HS.
3. Connect to the GC
4. If you are using the optional Remote Control Software, you must connect and configure a suitable PC.
5. Connect any optional accessories.
6. Leak test the system
7. Perform a test run to ensure all system components are operational.

Installation and setup of the HS are relatively straightforward. The instructions provided here are for the experienced user.

You will require the following tools and accessories:

- Flathead screwdriver
- Phillips screwdriver
- 1/4 and 1/8 inch wrench
- 1/16 inch and 3/8 inch wrench
- 2 mm Allen Key
- Pneumatic leak detection device or concentrated ethanol and water solution. Do **not** use soap solutions to detect leaks.- **Warning** Concentrated ethanol is flammable.
- Multimeter

- Bubble flow meter

Pre-Installation Checklist

Contact your safety engineer, industrial hygienist, environmental engineer, or safety manager before you install or use this instrument to determine if there are any other considerations in addition to the information listed below.

Before the instrument arrives:

1. Check your laboratory environment and ensure that the temperature and humidity settings are within the acceptable operating range for the HS. See *Operating Environment* later in this chapter.
2. Read *Safety Information* later in this chapter and ensure that you have complied with the requirements for each section.
3. If you are using the PC software, you must have a computer capable of running the software. If you are running TotalChrom, the HS control software can be run from the same computer.
4. Obtain the required gas supplies. See the section on *Gas Supply Specifications* later in this chapter. Ensure that you have the required carrier gas filters.
5. Ensure that you have the specified AC power supply. See *Electrical Connections* later in this chapter.

When the instrument arrives:

6. Check the instrument and any other equipment for any visible signs of damage. If you find that something is damaged, file a claim with the authorized carrier immediately, and inform your nearest PerkinElmer office.
7. Ensure that all ordered accessories have been delivered. Some accessories such as the PPC and the vial shaker will be installed at the factory.
8. Check that nothing is missing; a list of the equipment delivered is provided in *Table 1*. If you find that

something is missing, inform your PerkinElmer representative.

9. Contact your local PerkinElmer office to arrange for the installation. The service engineer will go over the pre-installation checklist to ensure all required pneumatic and electrical requirements have been met. A PerkinElmer service engineer will install the instrument and put it into operation for the first time.
10. All packing materials should be retained at the time of installation to ensure that nothing is mistakenly discarded. The customer should then retain the packing material for recycling, storage or further shipment.
11. Ensure that you have an adequate supply of sample vials, septa and caps. You will also need a crimper to seal the vials and a decapper to empty the vials.
12. If you have a GC other than an AutoSystem XL or a Clarus 400, 500 or 600, ensure that you have the proper adapter kit and start/ready cable to connect the HS to your GC and contact your PerkinElmer Service Representative for advice.
13. If you are using the cryofocusing accessory you need to obtain a supply of liquid nitrogen and a suitable Dewar vessel.
14. Review this manual and write down any questions that arise. Contact your local PerkinElmer office to answer your questions or provide an alternate source of information.

Laboratory Requirements

Operating Environment

WARNING

This instrument is not designed for operation in an explosive atmosphere.

General	The site for the instrument must be: • Indoors, as the HS is designed for indoor use only. • Free of dust, smoke and corrosive fumes. • Smooth, level and free from vibration.
Temperature	Ambient temperature between 10 and 35 ºC (50 and 95 ºF). Safe operation: 5 to 40 ºC. In addition, the site for the instrument must be: • Free of drafts • Out of direct sunlight. • Away from radiators and heaters. • Away from heating and air conditioning ducts as they will affect thermal stability.
Humidity	20 to 80% relative humidity (non-condensing).
Altitude	Sea level (0 metres) to 2000 m
Storage	You can store the instrument safely under the following conditions: • Ambient temperature –20 to 60 ºC (-4 to 140 °F) • Ambient relative humidity from 20 to 80%, (non-condensing) • Altitude in the range 0 m to 12000 m When you remove the instrument from storage, allow it to stand for at least a day under the approved operating conditions before putting it into operation.

Other	The laboratory should be free of flammable, explosive, toxic or corrosive vapors. Always provide adequate ventilation. When analyzing hazardous compounds it may be necessary to arrange for venting the detector effluent into a fumehood. Gas cylinders should be located outside of the laboratory whenever possible. All gas cylinders should always be stored and operated in the vertical position and should be firmly clamped to a suitable surface. Care must be taken not to kink or stress the gas delivery lines. **Installation Category:** The HS is able to withstand transient over-voltage according to Installation Category II as defined in IEC 1010-1. **Pollution Degree:** HS will operate safely in environments that contain non-conducting foreign matter up to Pollution Degree 2 in IEC 1010-1.

Table 6 Required Environmental Conditions

Space Requirements for the Instrument

You should leave sufficient room around the instruments to access all of the connections at the rear, and reach the electrical supply points. See *Table 7* for the space requirements.

Dimensions:	HS-16	43 cm (17”) wide x 61 cm (24”) high x 58 (23”) deep
	HS-40	43 cm (17”) wide x 61 cm (24”) high x 61 (24”) deep
	HS-110	43 cm (17”) wide x 61 cm (24”) high x 61 (24”) deep
	HS-40/110 Trap	43 cm (17”) wide x 61 cm (24”) high x 61 (24”) deep

	Clarus 400/ 500	66 cm (26”) wide x 40 cm (16”) high x 72 cm (28.5”) deep
	Clarus 600	53 cm (21”) wide x 99 cm (26”) high x 82 cm (32”) deep
	AutoSystem XL	66 cm (26”) wide x 40 cm (16”) high x 64 cm (25”) deep
Weight:	HS-16	32 kg (70 lb.)
	HS-40	33 kg (73 lb.)
	HS-110	35 kg (77 lb.)
	HS-40/110 Trap	35 kg (77 lb.)
	AutoSystem XL	49 kg (108 lb.)
	Clarus 400/ 500	49 kg (108 lb.)
	Clarus 600	64.5 kg (142 lb.)

Table 7 Space Requirements

Allow a minimum of 15 cm (6”) of clearance at the rear of the HS and 126 cm (4.1 ft.) of clearance at the top of the HS/GC system for ventilation. If this is not possible, install the HS/GC system on a bench that has wheels.

Allow at least 90 cm (36”) on either side of the GC/HS to accommodate additional equipment (for example, the computer).

The laboratory bench should be sturdy enough to support the full weight of the HS and GC as well as any additional support equipment (i.e., computer, data interface and printer). Expect the total weight of the HS/GC system and accessory equipment to weigh at least 136 kg (300 lb.).

The HS is typically positioned to the right of the GC to allow connection of the transfer line.

If the HS must be located on the left side of the GC, the HS should be fitted with the optional long transfer line (P/N M0413532). In these cases, approximately 180 cm (72”) of bench space will be required for the HS/GC system. This does not include the space required for the computer and related hardware. The spacing requirements will increase if you are using the TurboMass Mass Spectrometer or an AutoSampler.

If the cryofocusing accessory (M0413411) is installed, a 35 liter Dewar flask (B0500924) is required for the liquid nitrogen and should be placed under the lab bench. The Dewar flask is approximately 50 cm (20 inches) high x 25 cm (10 inches) wide.

For maximum performance and minimum maintenance, the site for the instrument and any accessories must:

- Be located close to the required electrical supply and gas supplies.
- Have sufficient room to work comfortably with the instruments, and allow you to reach connections at the rear of the instruments.
- Have space to place the gas chromatograph near the HS in a convenient position.
- Have space to place the computer and any hardware associated with the data handling system near the instrument in a convenient position. If you are connecting a printer, make sure that there is enough space for the printer and associated paper supply.

Electrical Connections

AC Line Connections

WARNING

To prevent potential injury to yourself and damage to the instrument, switch off all instruments in the system and disconnect them from the line power supply before you alter, or make any new electrical connections.

Installation Category	This instrument is able to withstand transient over-voltage according to Installation Category II as defined in IEC 1010-1.	
Power Consumption:	Approximately 1000 watts for the HS, the AutoSystem XL/Clarus 500 GC will consume 2400 watts. Add 250 W for the computer and 100 W for a printer.	
Power Specifications:	All electrical supplies must be smooth and free of transients greater than 40 volts peak-to-peak and must meet and remain within the following tolerances:	
	HS	100V±10% @ 50/60 Hz ±1%, 8 A 120V±10% @ 50/60 Hz ±1%, 8 A 220V±10% @ 50/60 Hz ±1%, 4 A 230-240V±10% @ 50/60 Hz ±1%, 4A
	Auto System	120 VAC ±10% @ 50/60 Hz ±1% 230 VAC ±10% @ 50/60 Hz ±1%
Power Outlets:	HS	A minimum of one dedicated 120 VAC outlet at 15 amps or a 230 V outlet at 6 amps
	Auto System or Clarus 400, 500 or 600	A minimum requirement of one dedicated 120 VAC outlet at 20 A or one 230 VAC outlet at 10 A or greater.

Table 8 Power Requirements

Additional equipment, such as computers and printers, should be connected as per their specifications.

Instruments and peripherals should not be connected to or near circuits with large inductive or large and variable loads (i.e. large motors, discharge lamps, photocopy systems, radio transmitters, etc.). All instruments of the system should be connected to a common phase.

The line power supply should conform to local safety regulations and must include a correctly wired protective earth/ground terminal. It should be checked by a qualified electrician before you connect the instrument.

To avoid interference caused by ground loops, always connect the HS and any accessory to the same phase of the line power supply and insure that they share a common earth ground. Observe outlet power limits.

Refer to the individual accessory manuals for details on installing various accessories and their power requirements.

Electrical Surge

The toroidal power transformer and the switched-mode power supply unit (SMPSU) of the HS provide a compact, high efficiency unit. A feature of these components, however, is that they can draw high “inrush” current from the external electricity supply during power up. In some circumstances, the current can be sufficient to cause external excess-current devices, particularly magnetically operated circuit breakers, to open.

Although the SMPSU incorporates thermistors to limit the “inrush” current, these are only effective when they are close to room temperature. During normal operation these components run at an elevated temperature and, as a result of their reduced electrical resistance, are unable to prevent the passage of high surge currents associated with brief interruptions of the external supply.

To avoid nuisance tripping of excess-current protection devices, do not supply power to the HS through fast acting circuit breakers. In addition, it is recommended that when you switch off the power to reset the HS that you wait at least 30 seconds before restoring the power. This enables the thermistors to recover.

This information refers only to excess-current protection devices. Any tripping of devices that detect current running to ground, for example earth leakage circuit breakers (ELCBs), residual current devices (RCDs), etc., is due to a different mechanism. Such occurrences must be investigated by a qualified person with expertise in electronics.

Connecting the HS to a Gas Chromatograph

Connecting the HS to a gas chromatograph involves the following procedures:

1. Connecting the gas supplies to the HS
2. Making the electrical connections
3. Installing the heated transfer line
4. Connecting optional accessories
5. Connect AC line cords
6. Leak testing the system
7. Performing a test run to ensure all system components are operational.

The HS–Gas Chromatograph configuration may vary slightly depending on the type of GC that you are using and on your application.

The HS is controlled from a computer (PC) or from its own touch screen keypad. Control lines, ready and start signals are provided to synchronize the HS, the GC and the data acquisition system.

Gas Supply System

WARNING

*Due to the explosive nature of hydrogen, **NEVER** use hydrogen gas as a carrier gas in any GC headspace system.*

Filtered helium or nitrogen with a minimum purity of 99.995% or better will be required. Use only approved gas lines to install the gas supply system for the HS. You must use the same carrier gas for the HS that is required for the GC analysis.

Carrier gas for an HS/GC system performing trace analysis or using either a mass spectrometer or an electrolytic conductivity detector

(ELCD) must have a minimum purity of 99.999% and must be properly filtered.

Always use either copper or stainless steel tubing which is free of grease, oil and organic material for all gases delivered to the HS/GC system. The carrier gas connections require 1/8” tubing with 1/8” connectors.

CAUTION	*Never clean carrier gas tubing with organic solvents. Any remaining traces of solvent will contaminate your system.*

Use compression fittings to make any joints in the tubing. Do not use soldered joints. The flux used in solder may contain a strongly electrophilic compound.

If the HS is fitted with the cryofocusing accessory, a supply of an inert gas, i.e. nitrogen and a Dewar vessel containing liquid nitrogen is required.

 **WARNING**	*Observe proper handling procedures for compressed gas cylinders. See Compressed Gases on page 27.* *Check the gas lines and connections regularly for leaks.*

Gas purity improves when filters are included in the supply lines. Oxygen filters (N930-1179), moisture filters (N930-1193) and charcoal filters (N930-1192) are recommended for carrier gas lines. When these filters are used together, they should be installed in the gas line in the following order: gas cylinder, hydrocarbon filter, moisture filter, oxygen filter and HS.

If you use filters from other suppliers, refer to the installation notes that are supplied by the manufacturers.

Gas Supply Specifications

Carrier	HS carrier gases require a minimum purity of 99.995%. Carrier gas with a purity of 99.999% is required if you are using a TurboMass or ECD detector on your GC. Pressure differences are minimized when helium is used as a carrier gas.

Dry Air	Dry air or nitrogen is used to drive the pneumatic automation systems on the HS 110. Use medical grade air. The purity of the dry air and dry air delivery system is not an issue, as this gas does not come into contact with the sample.
Liquid Nitrogen	If the HS is fitted with a cryofocusing accessory, it requires a supply of liquid nitrogen. The liquid nitrogen does not come into contact with the sample.
Nitrogen	An inert gas, such as nitrogen, is required as the coolant for the cryofocusing accessory. Do not use compressed air as a coolant gas; at low temperatures condensation can cause a potentially dangerous enrichment of oxygen. The purity of the coolant gas and associated delivery system are not an issue, as this gas does not come into contact with the sample.
Hydrogen	If you are using hydrogen for GC detectors only, ensure that all hydrogen lines and connections are leak-free. When using a hydrogen tank, install an in-line hydrogen flame arrestor (P/N 0009-0038) between the tank regulator and the delivery tubing.

Table 9 Gas Supply Specifications

Electrical Connections

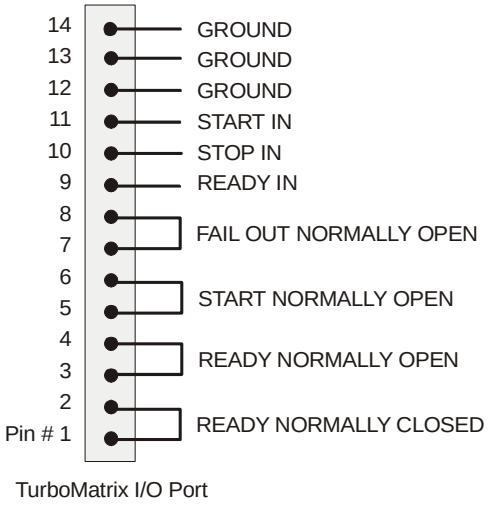
Electrical connections between the HS and the GC are made at the HS input/output port, shown in *Figure 1*. This port is located on the back of the instrument.

There are the following six options for connecting the HS to a GC:

1. HS to AutoSystem XL or Clarus 500 GC with TotalChrom LINK Interface
2. HS to AutoSystem XL GC or Clarus 500 and PE Nelson 900 Series Intelligent Interface
3. HS to AutoSystem XL GC or Clarus 500 and Network Chromatographic Interface (NCI)
4. HS to a GC other than an AutoSystem XL or Clarus 500 and the HP 6890
5. HS to the HP 6890
6. If the HS is PC controlled, the PC must be connected to the HS.

The HS Input/Output Port

Communication consists of a number of relay contact signals provided by the HS, which can be read by the external device, and a relay contact signal provided by the external device, which is read by the HS. *Figure 1* shows the signal connections to the Input/Output Port.



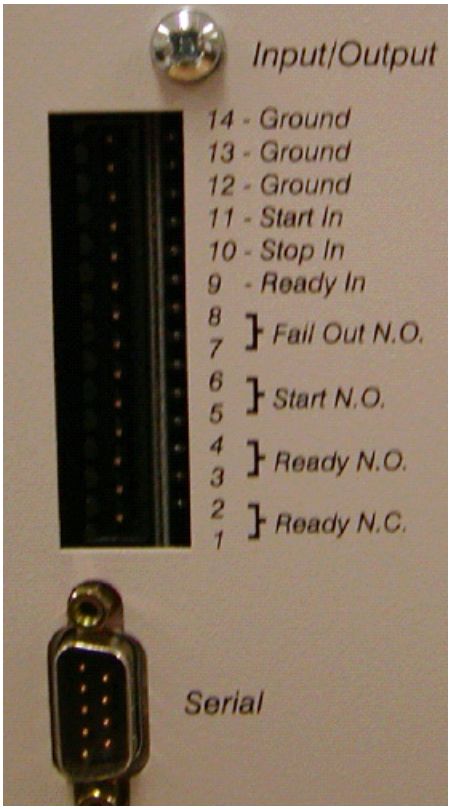


Figure 1 The HS Input/Output (I/O) Port (Diagram and Photo)

HS Output Signals

The Input/Output Port of the HS enables communication with other instrumentation, for example GCs and data handling systems.

NOTE: The relay contacts are rated to switch 10 W (maximum). Do not exceed 50V or 0.5A.

Ready Out—These signals, which indicate the HS ready status, are provided by a normally open relay. A contact closure between pins 1 and 2 (Ready Out) signals that the HS is Ready, or a contact break between pins 3 and 4 signals that the HS is “Ready”.

Normally, the HS acts as the controller, and neither of the above signals are connected.

Start—The Start (Inject) signal is a relay contact closure between pin 5 and 6 (Start Out) and coincides with the start of an injection onto the GC column. The contacts remain closed for 20 seconds. The Start signal is usually used to start a GC run by connecting it to the GC Start terminals.

NOTE: Normally the GC is used to start the data handling system or Remote Control Software or Bridge Application.

Fail Output Signal—The HS can be used with instrumentation that is unable to read the BCD sample vial number and therefore unable to report it with the chromatographic results. In this situation, care is required when a chromatogram is assigned to a particular sample vial. This is due to the fact that synchronization between the HS and such instrumentation is usually provided by the Inject signal from the HS, which starts the GC. See *BCD Interface* on page 165.

If processing of a vial is started by the HS, but is stopped, for example, by a missing vial: a GC run is not performed for this vial because an Inject signal is not generated. The next GC run is produced only when the sample from the following vial has been injected onto the GC. There will be gaps in the GC runs corresponding to samples that were not successfully transferred to the GC.

Although the position of these gaps can be determined from the Log tab, some data handling systems automatically increment the sample number, reported with the chromatogram. This can be misleading.

The fail output signal consists of a relay contact closure between pins 7 and 8 (Fail Out). See *Figure 1*. The Fail Out signal is generated instead if the Start signal in the event of a missing vial. This signals to an external device that the expected sample will not be transferred and that there will be a gap in the chromatographic data. Under these circumstances, the Fail signal can be connected to the external device in parallel with the Inject signal so that either signal can start the run. This results in blank runs being generated for tubes that fail, but synchronization of the HS and the other instrumentation is maintained.

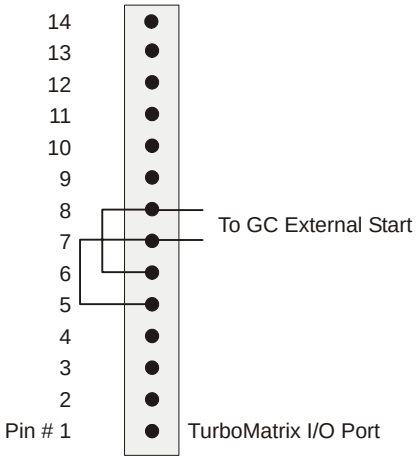


Figure 2 Connecting the Fail Output Signal in Parallel with the Start Signal

NOTE: Depending on the configuration of the external instrumentation, it may be more appropriate to connect the Fail signal elsewhere, e.g., to a data handling system. Many variations are possible.

Use of the Fail signal to start an external device and maintain synchronization depends on the device having completed its previous run and being in a state from which a blank run can be started.

BCD—The number of the vial in the analysis position (the rear-most position on the carousel) is available in BCD form. Each digit is signaled by relay contact closures between the appropriate pins and Common Out. See *BCD Interface* on page 165.

NOTE: The options board is required in order to use BCD signals This board is standard on HS 110 and Trap instruments.

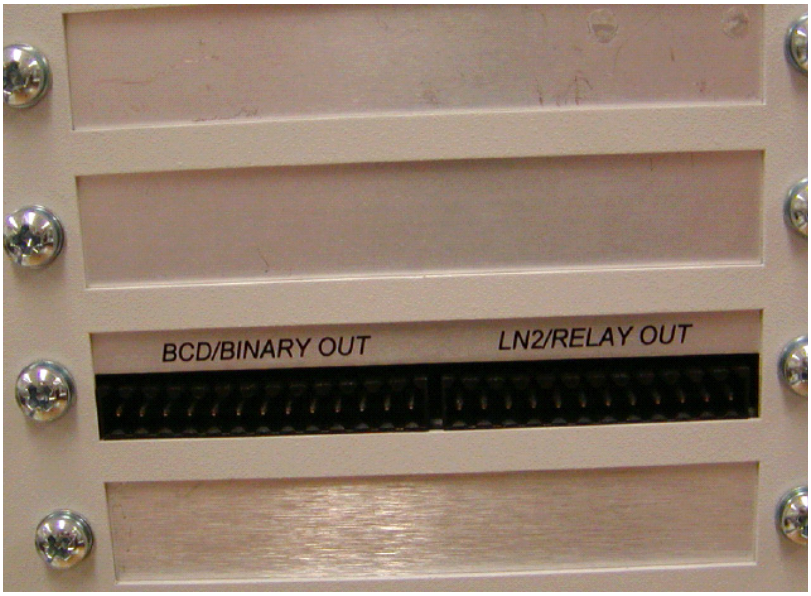


Figure 3 BCD located at the back of the instrument

HS Input Signals

Ready In—The HS can determine the status of external instrumentation using the Ready In signal. A relay contact closure, or equivalent, within the external device, connected between pin 9 (Ready In) and Ground (pins 12, 13 or 14), indicates to the HS that the GC is ready to start. See *Figure 1*.

The HS examines the status of the external device at several times during a sequence including before a sample vial is loaded and before injection. If a Ready signal is not received at these times, the HS status message “Ready To Inject” appears and the HS waits until the Ready signal is received.

If overlapping thermostating is selected GC ready signal is examined only before sample injection.

Start In and Stop In—The inputs are handshake signals, that are currently not used by the instrument.

RS-232C Port—This port operates at 9600 baud, and is intended for computer communications. It can be configured through the Preferences tab. See *Preferences Tab (Headspace and HS 40/110 Trap)* on page 136. Refer to the HS Control Software manual for details on connecting your computer to the HS.

Timed Event Relays

If you will be controlling external devices through the relays on the options board and timed events, you must first connect the external devices to the HS and then program the events.

The relay contacts are rated to switch 10 W (maximum). Do not exceed 50 VDC or 0.5A. External devices include external switching valves, audible alarms, very small motors, etc.

WARNING

Take care when you connect or disconnect the voltage outputs.

To connect external devices to the relay contacts:

1. Turn off the HS and disconnect it from the line supply.
2. Disconnect the I/O connector.
3. Connect your external devices to the appropriate connector contacts. Insert the wire leads into the connector and tighten the screws.
4. Connect all of your devices.
5. Re-insert the connector.
6. Connect the instrument to AC power and turn it on.

NOTE: Relays 1-4 are contact closures and Relay 5 and 6 are switched 24-volts.

Once all the devices are connected, you will power them up and program the timing for each device through the HS touch screen or the control software. Refer to *Timed Events* on page 163 for important details.

Connecting the HS to the AutoSystem XL or Clarus 500 GC

To connect the HS to the AutoSytem XL GC or Clarus 500 GC:

1. Connect the generic Ready/Start Cable Assembly (P/N N1011206) between the Input/Output socket on the HS and make the appropriate connections on the GC, as shown in *Figure 5*.
2. The HS requires a contact closure, or equivalent, to be applied between pin 9 (Ready In) and Ground (pins 12, 13 or 14) to receive a GC ready signal. The HS provides a contact closure between pin 5 and pin 6 (Start Out) to start a GC run.

NOTE: In the following illustration Pin # 9, 12, 13 or 14 are Ready In and Pin #5 and 6 are Start N.O. out

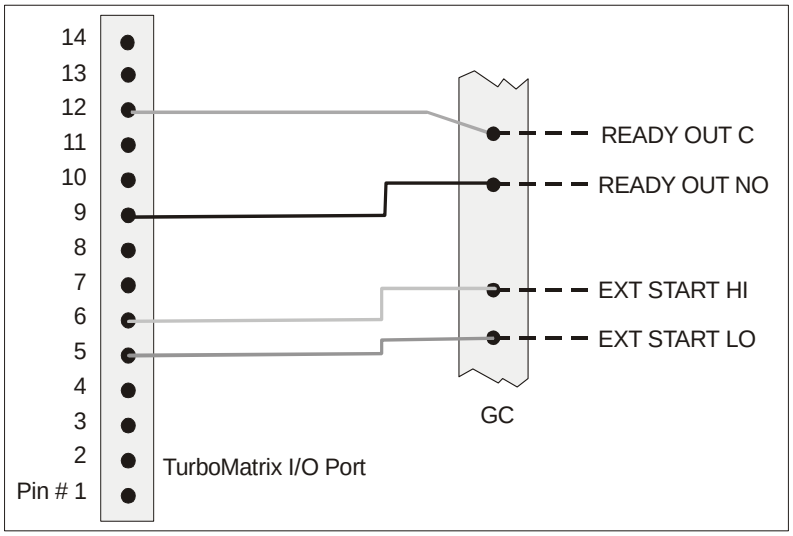


Figure 4 Connecting the HS to the AutoSystem XL GC using the Ready/Start Cable

Connecting the HS to other GCs except the HP 5890, HP 6890 and HP 7890

To connect the HS to other GCs (except the HP or 5890 or 6890):

1. Connect the generic Ready/Start Cable Assembly (P/N N1011206) between the Input/Output socket on the HS and make the appropriate connections on the GC, as shown in *Figure 5*.
2. The HS requires a contact closure, or equivalent, to be applied between pin 9 (Ready In) and Ground (pins 12, 13 or 14) to receive a GC ready signal. The HS provides a contact closure between pin 5 and pin 6 (Start Out) to start a GC run.

NOTE: In the following illustration Pin # 9, 12, 13 or 14 are Ready In and Pin #5, 6 and 7 are Start N.O.

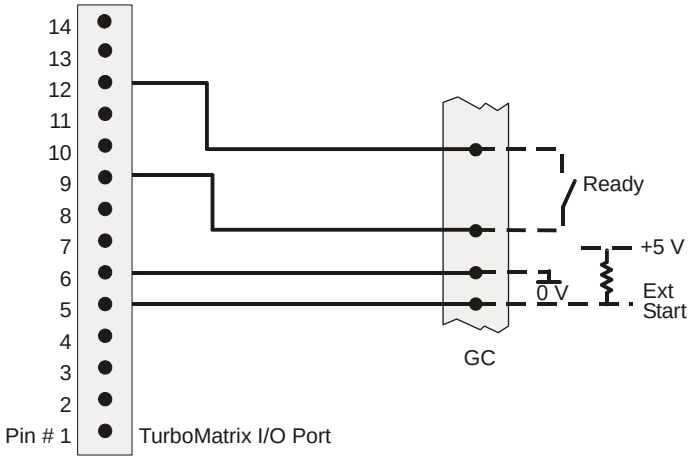


Figure 5 Connecting the HS to Another GC Using the Ready/Start Cable

Connecting the HS to the HP 5890 and HP 6890

The HP 6890 or 5890 GC uses one TTL connection for both Start In and Start Out and uses a second TTL connection for Ready In and Ready Out.

The HP 6890 GC does not have a Ready Output relay or a Ready Start relay and, therefore, it needs a special cable kit to communicate with the HS (P/N N6100410). This kit contains the parts necessary to connect a PerkinElmer sampler to a HP 6890 GC.

To connect the HS to the HP 6890 or 5890:

NOTE: This cable contains a resistor, diode, and transistor contained within the 9-pin connector shell.

1. Connect the 9-pin connector on the cable (P/N N6100402) to the Remote Start - Stop connector (port #3) on the rear panel of the HP 6890 or 5890.

NOTE: You can use either port #3 or port #6 since they are wired in parallel with each other.

2. If the HP6890 or HP 5890 is prepared and ready to start a run, placing a contact closure (shorting) across the white wire and either one of the black wires will start the run.
3. The open collector output from the red wire to either one of the Black wires simulates the GC Ready Output relay's Normally Open position with respect to Common at the black wire. Use the red wire as the Ready Output relay's Normally Open position and use the black wire as the Ready Output relay's Common position.
4. When the HP6890 or HP 5890 is Ready to begin the next run, the red wire will be effectively shorted to the black wire (ground) through the transistor's collector-emitter junction.

The HP 6890 GC cannot accept BCD signals from any external samplers including PerkinElmer samplers. There is no cable or procedure available to accomplish this.

1. You must ensure that the HP 6890 GC or HP 5890 GC data file and the appropriate HS vial number correspond to each other.
2. Locate the I/O port on the rear panel of the HS.
3. Connect the white wire of the cable assembly (P/N N610-0402) to the Start - Normally Open (Pin #5).
4. Connect one of the black wires to the Ground (Pin #12).
5. Connect the red wire to the Ready In (Pin #9).
6. Connect the remaining black wire to the Start - Normally Open. (Pin #6)

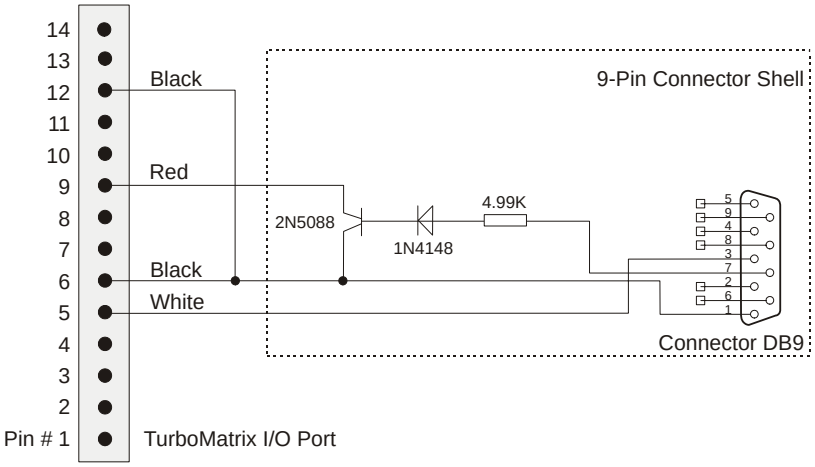


Figure 6 Schematic of the HP Sampler Cable

Installing the Heated Transfer Line

Installing the heated transfer line is an important procedure for the GC. The headspace version and the HS 40/110 trap have different requirements for this procedure.

*NOTE: For the headspace setup the best and most typical configuration is **Installing the Heated Transfer Line at the GC Injector for Split Operation**.*

For the headspace **only** configuration (split), the heated transfer line connects the sampling head to the GC. The sample moves through

the sample line from the vial to the GC column. Normally, at the GC end, you will connect the fused silica tubing to the injector. If your GC injector supports it, it is best to split the sample. The carrier gas is supplied and controlled by both the Headspace and GC. See the procedure, *Installing the Heated Transfer Line at the GC Injector for Split Operation,* later in this chapter.

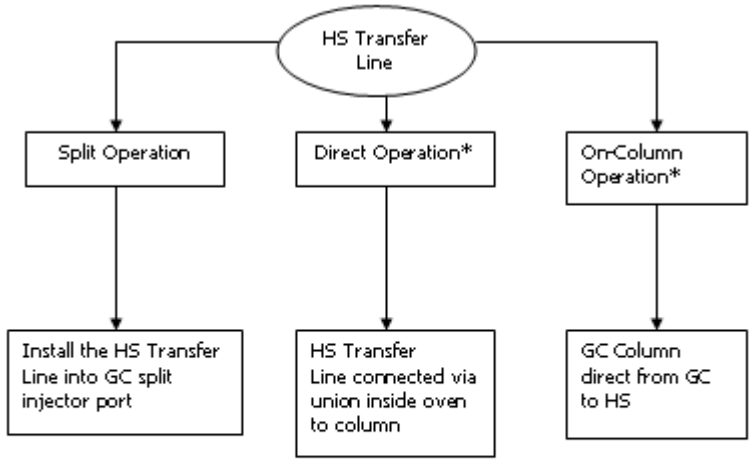
NOTE: For optimum performance the HS 40/110 trap requires a ***splitless*** *sampling setup.*

For the headspace and the HS 40/110 trap configurations, you can make a direct (splitless) connection by installing the fused silica transfer line so that it bypasses the GC carrier gas inlet. You can make this connection by using the universal column connection (P/N N930-2149) inside the oven. In this configuration the carrier gas is supplied and controlled by the HS. All vapor entering the transfer line enters the column. See the procedure, *Installing the On-Column Connection,* later in this chapter.

For the headspace and the HS 40/110 trap configurations you can make a direct connection from the GC capillary column to the sampling head. Sample separation begins immediately and again the vapor entering the transfer line enters the GC column. The first meter of the column is under isothermal control and will not be affected by temperature program. See the procedure, *Installing the Heated Transfer Line at the GC for Direct Connection,* later in this chapter.

NOTE: For the "on column connection" ensure that the transfer line temperature is high enough to elute your last component of interest.

Lastly, for the headspace and the HS 40/110 trap to improve performance the procedure, *Composite Zero Dilution Split Injector Liner for Headspace Interfacing,* later in this chapter

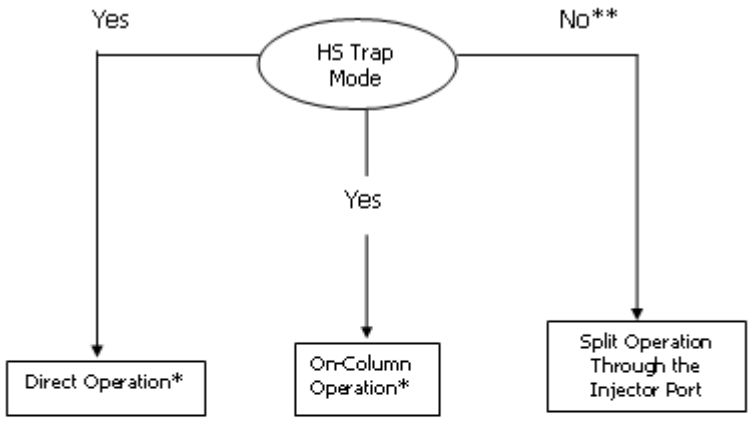


NOTE: If liquid autosampler is installed, the transfer line must be installed outside of the autosampler path.

*These options can be connected:

- side panel mounting kit
- rear panel of GC
- injector port

Figure 7 Headspace Setup



NOTE: If liquid autosampler is installed the transfer line must be installed outside of the autosampler path

*These options can be connected:

- side panel mounting kit
- rear panel of GC
- injector port

**This connection is typically not recommended for the HS 40/110 Trap setup. An advanced understanding of chromatography pressures and flows are required if you use this setup. The ZDL (Zero Dilution Lines) is desirable.

Figure 8 HS40/110 Trap Setup

Installing the Heated Transfer Line at the HS Needle Unit (TurboMatrix Headspace Only)

To install the heated transfer line at the HS needle unit:

1. Cut the white PTFE sleeve that extends from the transfer line, so that it is nearly flush with the outer shell of the transfer line. Ensure that the sliding insulation tube is in the transfer line header.
2. Place the electrical connector through the retaining collar.

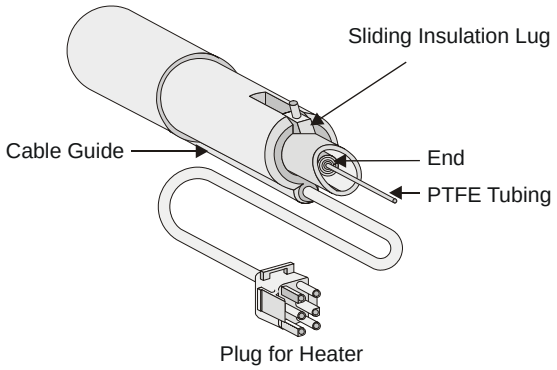


Figure 9 The HS End of the Transfer Line

3. Slide the transfer line into the retaining collar so that the top of the tube is flush with the collar. Do not tighten the mounting screws as you will need room to install the fused silica tubing. The transfer line and the glass-lined tube (GLT) adapter should be in a straight line.
4. Remove the cap from the outlet of the GLT connector tubing.
5. Insert the GLT connector tubing into the nut and ferrule and install these into the fitting of the needle unit.
6. Tighten the nut slightly, allowing some back and forth movement of the GLT connector tubing.
7. Slide the GLT connector tubing into the needle unit until it stops.
8. Tighten the nut to hold the GLT connector tubing in place. Do not over tighten the nut as you may damage

the ferrule or crack the glass in the GLT connector tubing. See Figure 9.

9. Unravel approximately 1 meter of the fused silica tubing. Insert the fused silica tubing, from the GC end, into the transfer line until approximately 5 cm of the line extends from the HS end of the transfer tubing.
10. Slide the 1/16” sleeve nut and the graphite ferrule onto the end of the fused silica tubing between the transfer line and the GLT connector tubing.

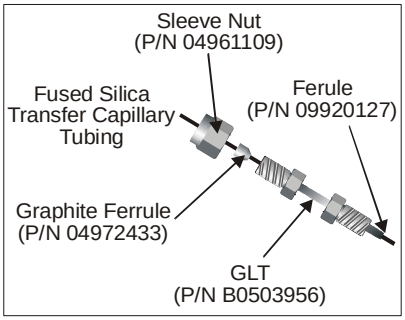


Figure 10 Assembling the Fittings Ahead of the GLT Connector

11. Score and break the fused silica tubing to remove any graphite residue, which may block the carrier gas flow.
12. Push the fused silica tubing into the GLT connector tubing until it reaches the needle. Slide the ferrule and nut into position and tighten finger tight. Mark the position on the fused silica at the end of the nut. Withdraw the fused silica tubing 1 to 1.5 cm back from the needle. Tighten the nut.
13. Plug the other end of the fused silica tubing by forcing an injector septa on it. Leak test the system before you finish assembling the transfer line. See the *Leak Testing* procedure later in this chapter for details on performing a leak test.
14. Slide the transfer tubing down so that the PTFE sleeve is within 1-2 mm of the sleeve nut and then tighten the mounting screws on the collar.

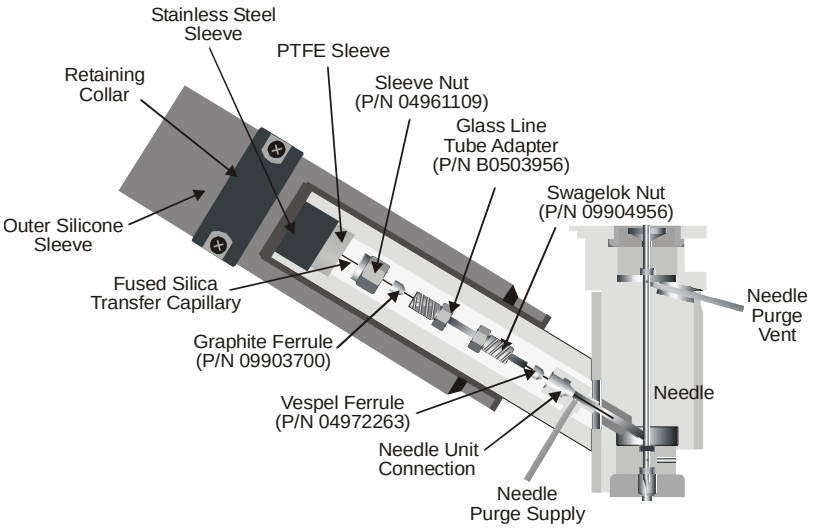


Figure 11 Installation of Transfer Line at the Needle Unit

15. Pull the sliding insulation tube from the transfer line all the way down so that it touches the needle unit in order to protect the fused silica tube connections and to keep the area heated.

16. Plug the electrical connector into the receptacle above the retaining collar. The connector is polarized so that it will only fit one way. Do not force the connection.

Installing the Heated Transfer Line at the HS Needle Unit (HS 40/110 Trap Only)

To install the heated transfer line at the HS needle unit:

1. Cut the white PTFE sleeve that extends from the transfer line, so that it is nearly flush with the outer shell of the transfer line. Ensure that the sliding insulation tube is in the transfer line.
2. Place the electrical connector through the retaining collar.

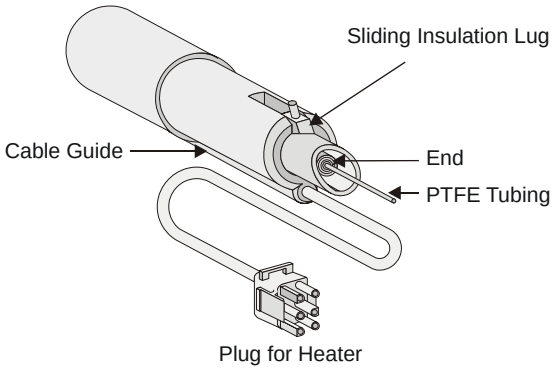


Figure 12 The HS End of the Transfer Line

3. Slide the transfer line into the retaining collar so that the top of the tube is flush with the collar. Do not tighten the mounting screws as you will need room to install the fused silica tubing. The transfer line and the deactivated tube adapter should be in a straight line.
4. Remove the cap from the outlet of the deactivated tube adapter.
5. Insert the deactivated tube adapter into the nut and ferrule and install these into the fitting of the needle unit.
6. Tighten the nut slightly, allowing some back and forth movement of the deactivated tube adapter.
7. Slide the deactivated tube adapter into the needle unit until it stops.
8. Tighten the nut to hold the deactivated tube adapter in place. Do not over tighten the nut as you may damage

the ferrule or crack the glass in the deactivated tube adapter. See Figure 9.

9. Unravel approximately 1 meter of the fused silica tubing. Insert the fused silica tubing, from the GC end, into the transfer line until approximately 5 cm of the line extends from the HS end of the transfer tubing.

10. Slide the 1/16” sleeve nut and the graphite/vespel ferrule onto the end of the fused silica tubing between the transfer line and the deactivated tube adapter.

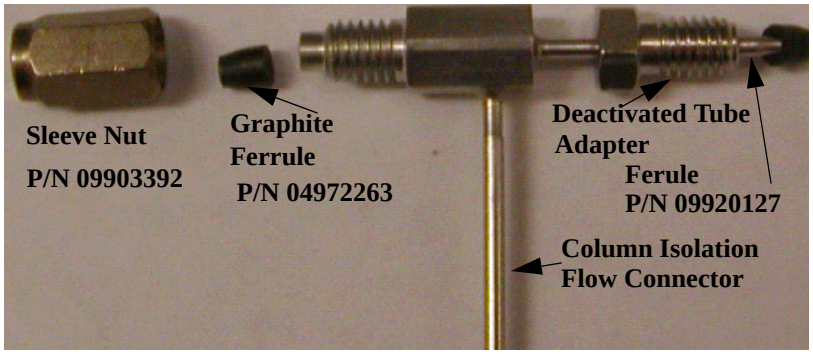


Figure 13 Assembling the Fittings Ahead of the Deactivated Tube Adapter

11. Score and break the fused silica tubing to remove any graphite residue, which may block the carrier gas flow.

CAUTION	*Do **not** break the column isolation flow connection. This connection is very delicate take care not to break it.*

12. Push the fused silica tubing into the deactivated tube until it reaches the end. Slide the ferrule and nut into position and tighten finger tight at the needle entry. Tighten both nuts.

13. Plug the other end of the fused silica tubing or column by forcing an injector septa on it. Leak test the system before you finish assembling the transfer line. See the *Leak Testing* procedure later in this chapter for details on performing a leak test.

14. Slide the transfer tubing down so that the PTFE sleeve is within 1-2 mm of the sleeve nut and then tighten the mounting screws on the collar.

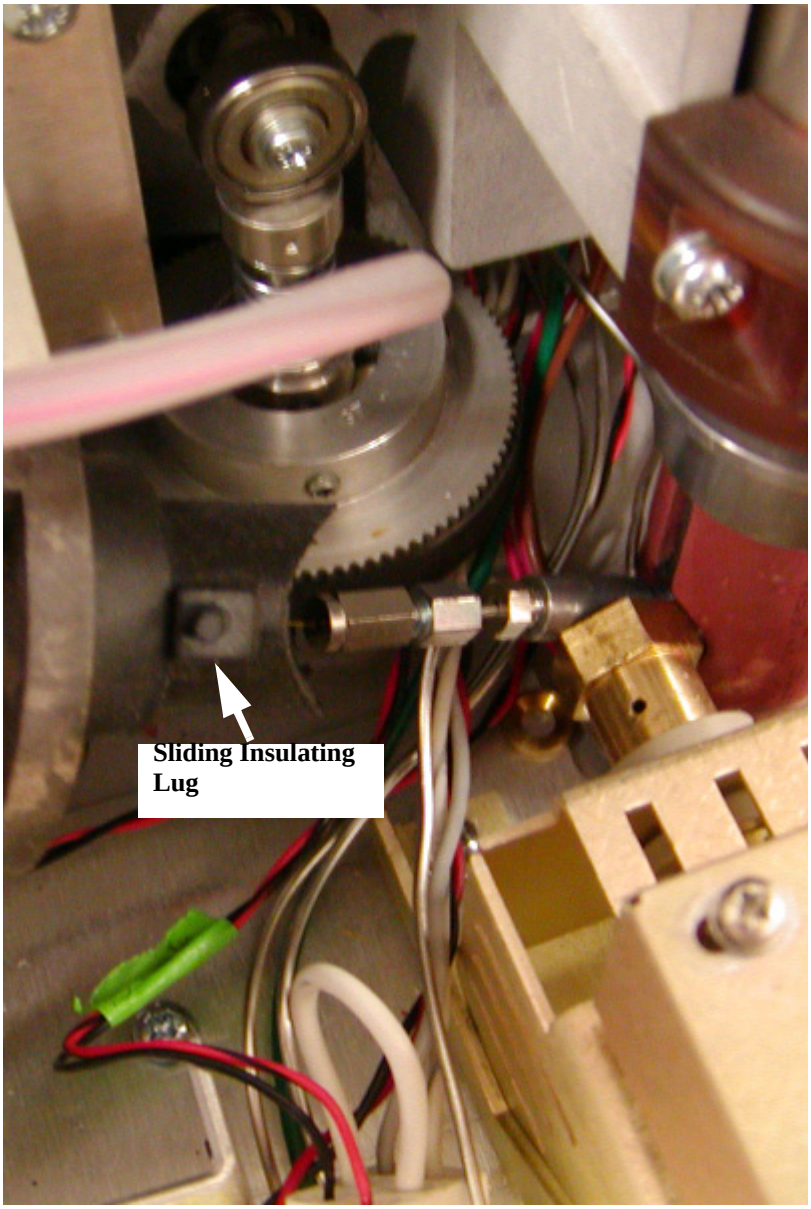


Figure 14 Installation of Transfer Line at the Needle Unit

15. Pull the sliding insulation tube from the transfer line all the way down so that it touches the needle unit in order to protect the fused silica tube connections and to keep the area heated.
16. Plug the electrical connector into the receptacle above the retaining collar. The connector is polarized so that it will only fit one way. Do not force the connection.

Installing the Heated Transfer Line at the GC Injector for Split Operation (TurboMatrix Headspace Only)

Once the transfer line has been connected to the HS and the connections have been leak tested, you can then connect the transfer line to the GC. An AutoSystem XL or Clarus 500 GC adapter is supplied with your instrument. If you have another type of GC, contact your PerkinElmer service engineer.

WARNING

The threads on the adapter are sharp and can cause cuts to your fingers or hand. Use the proper protection (a paper towel or gloves) before tightening the adapter.

Liner Recommendations

We recommend using certain liners when installing different injectors on an Autosystem XL or Clarus 500 GC.

Packed Injector	**Standard** Liner	No Packing
Capillary Injector	2 mm or 4 mm Liner	No Packing
PSS Injector	2 mm or 1 mm Liner	No Packing

To install the heated transfer line at the GC injector for split operation:

1. Turn off the GC and allow the injector to cool.
2. Use a wafer scribe (P/N N9301376) or other column cutting tool to score and break the fused silica transfer

tubing leaving a length of fused silica tubing extending 10 to 15 cm past the end of the transfer line.break.

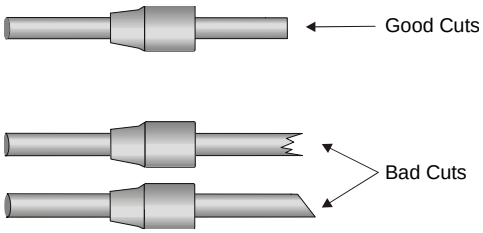


Figure 15 Cutting the Fused Silica Transfer Line

3. The transfer line is connected to the gas chromatograph using a stainless steel adapter and a septum.
4. Place the septum into the adapter.
5. Carefully push the fused silica tubing through the adapter, piercing the septum.

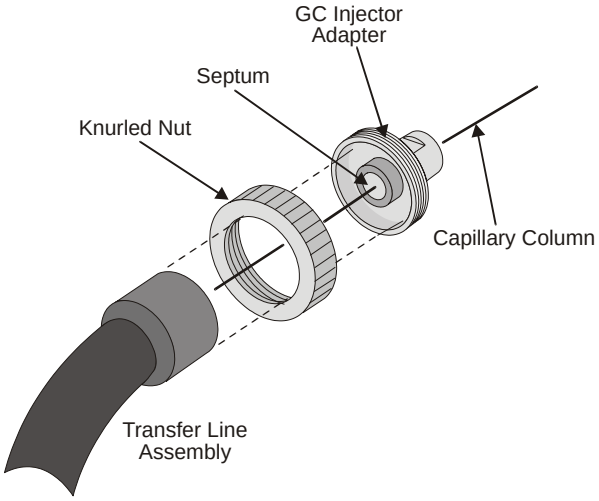


Figure 16

6. Connect the adapter to the end of the transfer line.
7. The fused silica tubing must be cut so that 5 cm extends from the end of the stainless steel adapter not from the

end of the insulated transfer line. See the following figure.

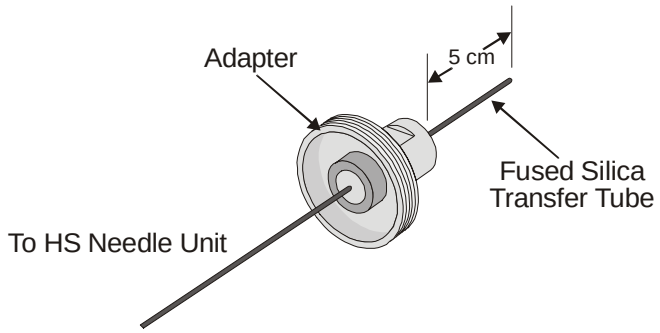


Figure 17 Length of Transfer Line that Must Extend Past the Adapter for Installation at GC

8. Remove the septum retaining nut from the GC injector port. Store this nut in a safe location for later use.

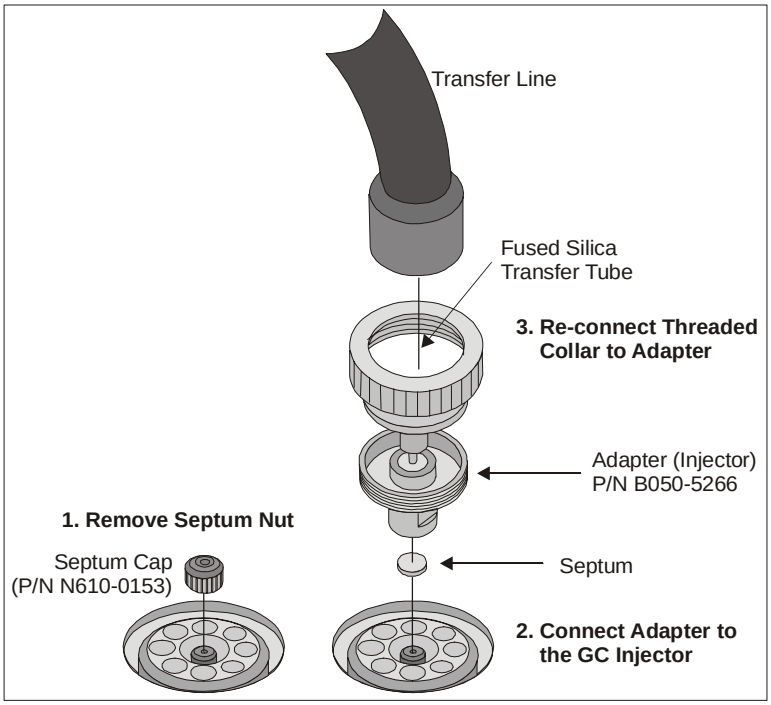


Figure 18 Installing the Heated Transfer Line at the PerkinElmer GC Injector

WARNING

The threads on the adapter are sharp and can cause cuts to your fingers or hand. Use the proper protection (a paper towel or gloves) before tightening the adapter.

9. Loosen the threaded collar from the stainless steel adapter but do not remove the adapter, carefully **(see warning above)** use the threads on the adapter, to screw the adapter into the injector port and tighten.
10. Replace the threaded collar of the transfer line onto the stainless steel adapter and tighten.

Leak test the system as outlined in the *Leak Testing* procedure later in this chapter.

Installing the Heated Transfer Line at the GC for Direct Connection

Recommended for the HS 40/110 trap

You can install the transfer line so that it bypasses the inlet split of the GC injector. By installing the fused silica tubing in this manner the GC carrier gas is now supplied by the HS and the incoming headspace sample is not split or diluted in any way.

To install the heated transfer line at the GC for direct connection:

1. Turn off the GC and allow the injector to cool.
2. Inside the GC oven disconnect the GC column from the injector.
3. Score and break the fused silica tubing extending out of the end of the HS transfer line. Be sure to have plenty of transfer line to work with (20 to 50 cm).
4. The transfer line is connected to the GC injector using a stainless steel adapter (Part No. B0505266) and a septum.
5. Place the septum into the adapter (See Figure 18).

6. Carefully push the fused silica column through the adapter, piercing the septum.
7. Connect the adapter to the end of the transfer line.
8. Remove the septum nut from the GC injector port.

NOTE: Store the septum nut in a safe location for later use.

9. Feed the fused silica tubing through the GC injector. Loosen the threaded collar from the stainless steel adapter and using the threads on the adapter, as a handle, screw the adapter into the injector port.
10. Replace the threaded collar of the transfer line onto the stainless steel adapter (use the universal connector).
11. Inside the GC oven, connect the fused silica of the headspace transfer line to the other end of the column connecting union (P/N N9302149).
12. Leak test the system. See the procedure “Leak Testing” later in this chapter.

On-Column Connection

Recommended for the HS 40/110 trap

In the on-column configuration, you will take the capillary column out through the injector and connect it to the sampling head.

1. Turn off the GC and allow the injector to cool.
2. Inside the GC oven disconnect the GC column from the injector. Unravel approximately 1 meter of the capillary column.
3. Remove the nut and fitting. Score and break the fused silica column if necessary to remove the ferrules.
4. Feed the column up through the injector until it clears the top of the injector.
5. Place the septum into the adapter.
6. Carefully push the fused silica column through the adapter from the bottom side, piercing the septum.

7. Loosen the threaded collar from the stainless steel adapter and using the threads on the adapter, as a handle, screw the adapter into the injector port.
8. Insert the capillary, from the GC end, into the transfer line until approximately 5 cm of the line extends from the HS end of the transfer line.
9. Slide a 1/16” sleeve nut and graphite ferrule onto the end of the fused silica column between the transfer line and the deactivated tube adapter.
10. Score and break the fused silica column to remove any graphite residue, which may block the carrier gas flow.
11. Push the fused silica column into the deactivated tube until it reaches the needle. Slide the ferrule and nut into position and hand tighten. Mark the position on the fused silica at the end of the nut. For the TurboMatrix Headspace, withdraw the fused silica column 1 to 1.5 cm back from the needle and tighten the nut. **For the HS 40/110 trap do not withdraw the column at all.** See Figure 13.
12. Plug the other end of the column and leak test the system before you finish assembling the transfer line. See “Leak Testing” later in this chapter for details on performing a leak test.
13. Slide the transfer line down so that the PTFE sleeve is within 1-2 mm of the sleeve nut and then tighten the mounting screws on the collar.
14. Pull the sliding insulation tube from the transfer line all the way down so that it touches the needle unit in order to protect the fused silica tube connections and to keep the area heated.
15. Plug the electrical connector into the receptacle below the retaining collar. The connector is polarized so that it will only fit one way. Do not force the connection.

Composite Zero-Dilution Split Injector Liner for Headspace Interfacing

Installation Instructions

1. Cool the oven and injector. Remove the column and the existing injector liner.
2. Check the outer and inner liner components of the composite liner for contamination or damage and replace as necessary.
3. Insert the inner liner into the outer liner.
4. Install the composite liner into the injector using a standard O-ring. The inner liner should be upper most. The outer liner should be pushed fully into the injector.
5. Replace the injector head and secure with the spanner.
6. Thread a length of 0.32 mm i.d. deactivated fused silica tubing through the transfer line and attach to the HS sampling head.
7. The other end of the tubing should be thread through the septum in the HS septum nut. Cut the tubing so that 62 mm is left protruding from the septum (57 mm from the edge of the septum nut). this length can be shorter (down to 20 mm) if 0.32 mm or 0.53 mm i.d. columns are being used.
8. Insert the fused silica tubing into the injector and secure the HS injector adapter.
9. Insert the column into the base of the injector and push it through the liner until it just reaches a stop (about 8 cm). **Do not push the column too hard.** At this point, the fused silica transfer line and the column should be at either side of the restriction in the inner liner. Withdraw the column about 2 mm and tighten the column ferrule and nut.

Operation

1. The liner seems to function satisfactory at pressure drops across the transfer line at 0.5 psi or above -2.5 psi is recommended (approximately 20ml/min).

2. The GC split flow should be set to 10 ml/min or more although higher flow rates will just waste gas.

Connecting to a Packed Column System

If you are using a packed column in your GC, you must use a packed column injector.

1. Install the packed column as outlined in *Installing the Heated Transfer Line at the GC Injector for Split Operation (TurboMatrix Headspace Only)* on page 68.
2. Set the HS pressure accordingly. You may need to use the a high pressure injection to eliminate the possibility of pre-injections. See the section “High Pressure Sampling” in the Operation chapter.

Gas Connections

For information on the *Gas Supply System* see the section earlier in this chapter.

Always use clean tubing preferably copper or stainless steel, with the minimum possible number of joints for carrier gas lines. If necessary, pass a stream of clean, inert gas through the tubing, while baking it in an oven at a temperature high enough to remove any trace organic solvents. **Never clean the tubing or fittings with organic solvents.**

Use compression fittings to make tubing connections. Do not use soldered joints.

Carrier Gas

Carrier gas can be a major source of contamination. Contamination can originate from the gas itself or from the tubing used to deliver the gas. Use only carrier gases with a purity of 99.995% or better. Only top quality gases are suitable; typical laboratory supplies are usually not pure enough. Gas purity can be improved when filters are included in supply lines. Hydrocarbon, oxygen and moisture filters are recommended for carrier gas lines.

When oxygen filters and moisture filters are used together, install them in the gas line in the following order: gas cylinder, hydrocarbon

filter, moisture filter, oxygen filter, and HS. This sequence prevents any hydrocarbons present in the gas stream from reaching the oxygen filter. To minimize carrier-gas impurities and reduce instrument contamination we recommended installing a charcoal filter in the carrier gas line, close to the inlet of the HS.

Most filters are disposable. Replace them when a new cylinder is installed or as soon as contamination is suspected.

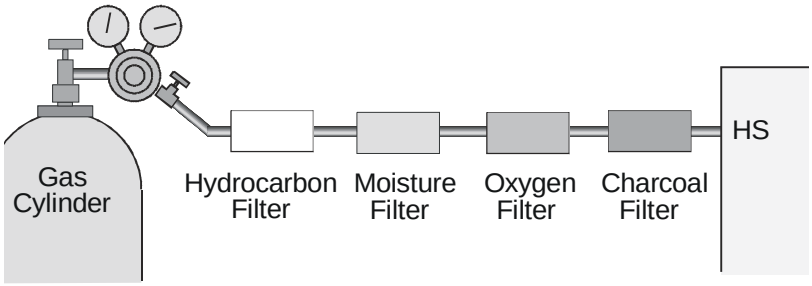


Figure 19 Connecting Filters to the Gas Supply System

WARNING

Ensure that gas lines containing filters are protected from excessive pressure.

To connect carrier gas to your HS:

1. Locate the carrier gas tanks in a secure location. See the section on *Compressed Gases* earlier in this chapter.
2. Connect a clean, high purity, two-stage regulator to the carrier gas tank. The regulator should also have a 1/8”, parallel, compression fitting with which to connect the regulator to the HS. The delivery pressure must be adjustable to 90 psig (620 kPa).

NOTE: The regulator must be absolutely clean and free from any oil or other contamination.

3. Connect any filters that are required as close as possible to the HS.

4. Connect the regulator to the HS Carrier In port. Use copper or stainless steel tubing only. Securely tighten all fittings.
5. Open the tank and set the delivery pressure to 90 psig (620 kPa). The carrier gas delivery pressure must not exceed 100 psig (690 kPa), and it must be sufficient to maintain the required column head pressure at the gas chromatograph.

WARNING

Do not set the delivery pressure higher than 100 psig (690 kPa).

Dry Air

If you are running an HS 110 or HS Trap with an internal standard accessory, you will also require a supply of dry air. The dry air is used to drive the pneumatics required for the automation system. The purity of the dry air and delivery lines is not an issue, as this gas does not come into contact with the sample.

A high purity stainless steel regulator is not required for the dry air, as this gas does not come into contact with the sample. A brass regulator will be suitable. You can use Teflon or copper tubing to connect the regulator to the HS.

NOTE: If you are running more than one instrument from a single supply of dry air, you must ensure that your supply system can deliver a minimum of 3 liters per minute at 70 psi for each instrument under all sampling conditions.

To connect the dry air supply to your instrument:

1. Connect a clean, two-stage regulator to the dry air tank. The regulator should also have a 1/8”, parallel, compression fitting with which to connect the regulator to the HS. The delivery pressure must be adjustable to 90 psig (620 kPa).
2. Connect the regulator to the HS Dry Air in port. The purge connection at the rear of the HS is a 1/8” brass fitting.

3. You can use Teflon® tubing for the dry air supply lines. If you are using Teflon tubing, ensure the lines can withstand pressures of 100 psig.
4. Securely tighten all fittings.
5. Open the tank and set the delivery pressure to 90 psig (620 kPa).

WARNING

Do not set the delivery pressure higher than 100 psig (690 kPa).

Connector	Dimension	Function
Carr In	1/8” stainless steel compression fitting	Carrier gas moves the sample through the system. The carrier gas connected to the HS also supplies the GC. Maximum delivery pressure is 100 psig (690 kPa). 90 (620 kPa) psig is recommended.
Dry Air	1/8” brass compression fitting	Dry air is to drive the automated vial handling components on the HS 110 and HS 40/110 trap. Maximum delivery pressure is 100 psig (690 kPa). 90 (620 kPa) psig is recommended.
Cryo In		Cryofocusing Inlet Maximum delivery pressure is 100 psig (690 kPa). 90 (620 kPa) psig is recommended.
Cryo Out		Cryofocusing Outlet
Water Trap		Carrier Gas for the Water Trap accessory

Table 10 HS Gas Connections

HS 40/110 Trap Connections

The HS 40/110 trap may require three gas connections; attach the carrier gas, any gas internal standard, and the dry air supply. The following table shows the required gases for the HS 40/110 trap. See the following photograph of the back of the instrument to find the location of the separate gas hook-ups.

TRAP Model and Options	**Gases**	**Remarks**
TurboMatrix 40 Trap	He or N2	Carrier Gas
TurboMatrix 40 Trap and Int. Std (IS)	1.He or N2 2.Dry Air	1.Carrier Gas 2.for the air Actuator of IS valve
TurboMatrix 110 Trap w/ or w/o Int. Std (IS)	1.He or N2 2.Dry Air	1.Carrier Gas 2.and/or crane IS.

NOTE: It is assumed that if a cylinder containing an Internal Standard gas is used, its pressure regulator outlet is connected to the respective inlet at the back of the trap unit.

Figure 20 Connections on back of the instrument

Installing the Trap in the HS 40/110 Trap

The HS 40/110 trap is shipped with the trap housing already installed. You must install the trap.

NOTE: When installing the trap in the instrument for the first time or you must take the trap assembly apart, you must do an alignment procedure. If you are just removing an old trap and replacing it with a new trap you do ***not*** *need to do an alignment procedure.*

WARNING *Wear gloves when you are handling the trap. Handle the trap with great care since it is made from glass and can easily break.*

NOTE: Before proceeding always have one or two spare traps on hand in case of breakage.

NOTE: Turn off the unit and Column Isolation will be automatically applied.

CAUTION ***Do not over tighten****. Only finger tighten the large thumbscrew, otherwise you will break the trap.*

1. Remove the looks cover by opening it and taking the cover off the hinges by lifting it straight up. Turn off the HS40/110 trap or start the column isolation flow. This will enable gas flow to continue to the GC but the trap will be blocked off.
2. Remove the large thumb screw and put it in a secure location.

3. Pull out the dry purge assembly (P/N N6700112). See the following photo.

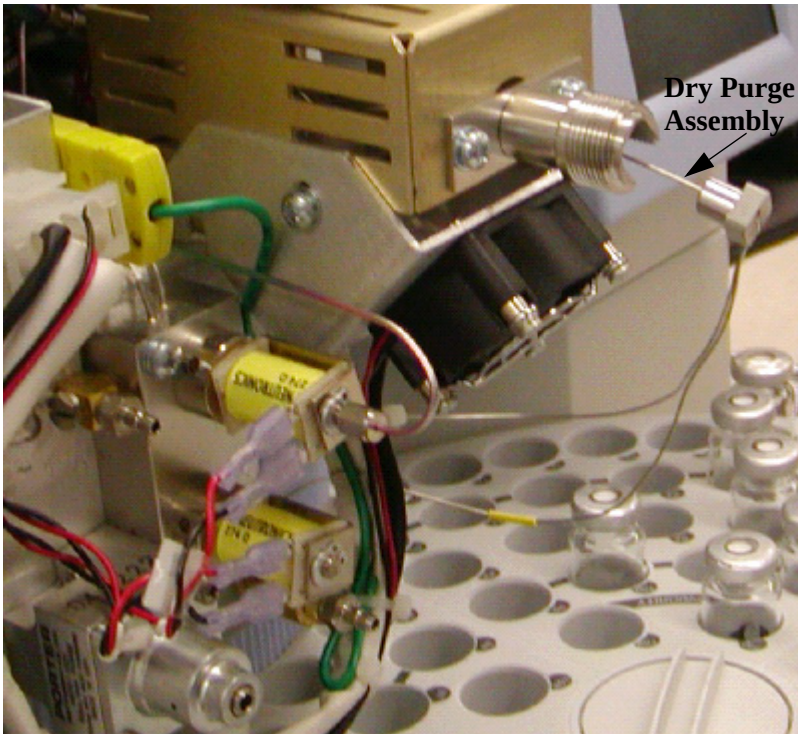


Figure 21

4. Carefully loosen the nut in the back of the trap assembly.Turn it 1/4 to 1/2 turn only since if you loosen it too much the O-ring inside it will be out of alignment and difficult to reinstall. See the following photo.

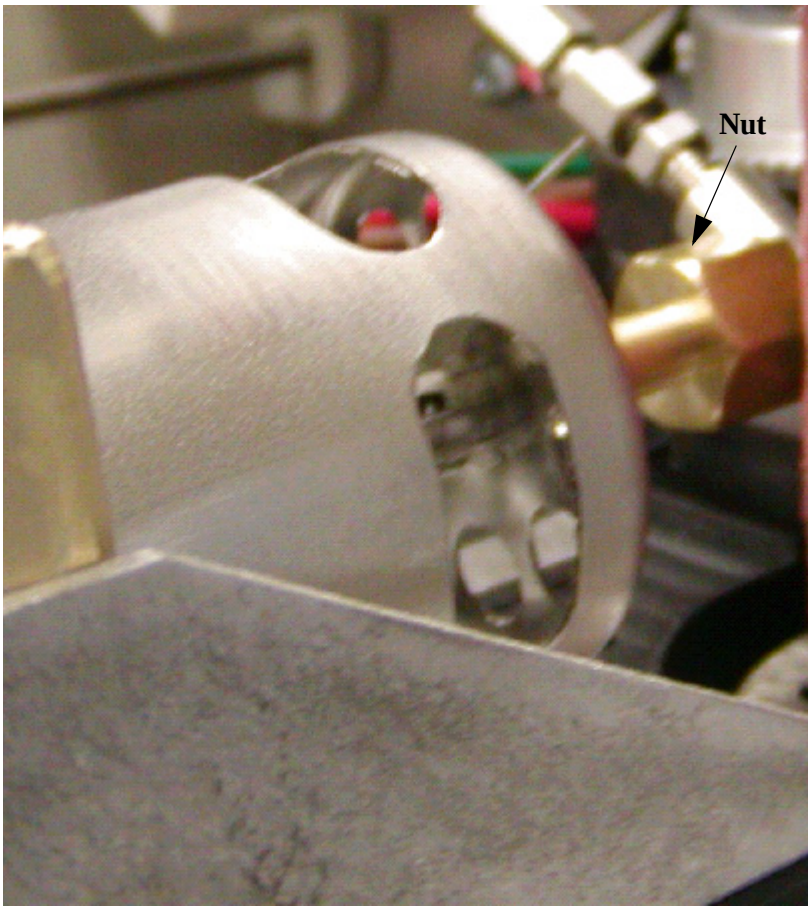


Figure 22

5. Use the trap removal tool (P/N N670-1077) to carefully remove the trap. See the following figure.

6. Wearing gloves, carefully remove the new trap from the box and insert it into the trap housing (hollow tube end first, wire end last).

Figure 23

7. Put the ferrule on the trap (the tapered edge must face towards the front). See the next photo.

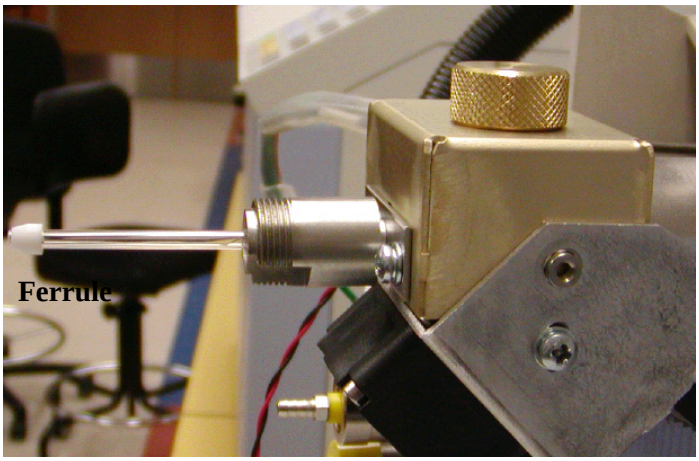


Figure 24

8. By hand gently push the new trap in as far as you can.
9. Use the Alignment Rod (P/N N6700122) to gently push the trap into the O-ring into the proper position. You will feel a small pop as the trap goes through the O-ring and is seated properly in the trap housing.
10. Retighten the back nut until it stops.
11. Inspect the dry purge assembly to see that it is not damaged. Feed the dry purge assembly into the trap until it stops.

NOTE: If the dry purge assembly is damaged see the Routine Maintenance chapter for information on replacing this assembly.

12. Reinstall the trap housing. Do not use any tools (only fingertighten) to tighten the thumb screw since it will damage the ferrule.
13. The trap must be conditioned (see the Routine Maintenance chapter for this procedure) before analytical use by establishing carrier gas flows and heating the trap several times to remove any volatile impurities from the trap packing.

If you heat the trap to high temperatures take care that the analytes do not degrade at these high temperature. For example, if the halogenated hydrocarbons are present in the sample, the temperature should not exceed 325 °C. When the trap is heated to 325 °C, trimethyl benzenes are released quantitatively. For higher boiling species it may be necessary to use a higher trap temperature.

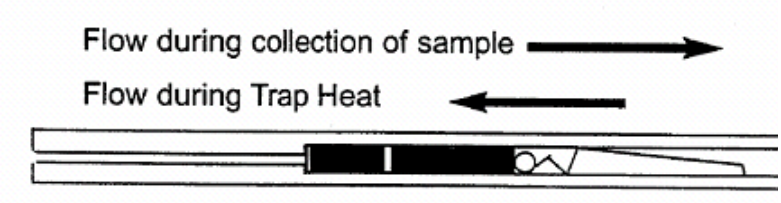


Figure 25 Air Monitoring Trap (M041-3628)

See the Routine Maintenance chapter in this guide for information on testing, conditioning and cleaning the trap.

Checking the Needle Purge Gas Flow

TurboMatrix Headspace and HS 40/110 Trap

The purge gas flow through the needle unit is set to 15 ± 3 mL/min. The flow is used to purge the needle of residual sample and to keep it clean between injections. The flow is fixed and you only need to initially measure the flow to ensure that it is within the recommended range.

To check the needle purge gas flow:

1. Ensure the carrier gas is connected. Connect a flowmeter to the Purge vent.
2. Switch on the instrument. Set the pressure for 5 psi.
3. If the flow rate is correct, then disconnect the flow meter. If the flow rate is less than 10 or greater than 15, contact your PerkinElmer service engineer.

Leak Testing the Headspace

TurboMatrix Headspace Only

If a leak is occurring and you cannot isolate the source, you may want to separate the HS (headspace) from the GC and leak test each instrument separately. Once each instrument is found to be leak tight you can then connect them and test them together.

The automated leak test will leak test the HS sampling system. You must plug the end of the fused silica tubing or if the fused silica tubing is directly connected to the GC column, then plug the end of the column.

To leak test the sample injection system:

1. Switch off the gas chromatograph and let the instrument cool down.
2. Use a blanking plug to seal the column outlet. Seal capillary columns with a new, clean septum.
3. Set the carrier pressure to 45 psi. See the section on *Carrier Gas* earlier in this chapter.
4. Open the Tools drop down menu and select **Maintenance** and then select **Leak Test**. If the leak test fails you will get the following screen. Go to step 8 for the procedure for a failed leak test.

NOTE: The first screen on the following page shows a failed test (red text indicates a failure) and the second screen shows a passed test (black text indicates a success).

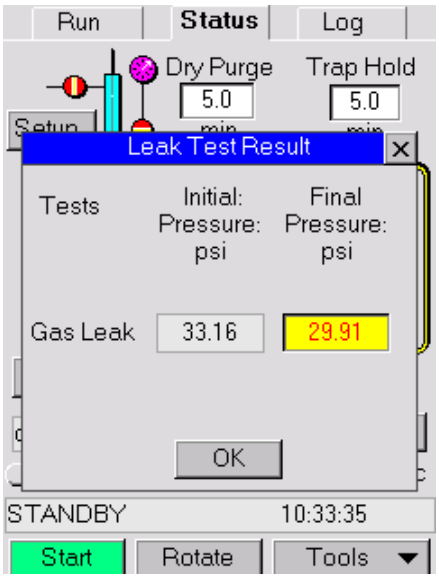


Figure 26 Failed Leak Test

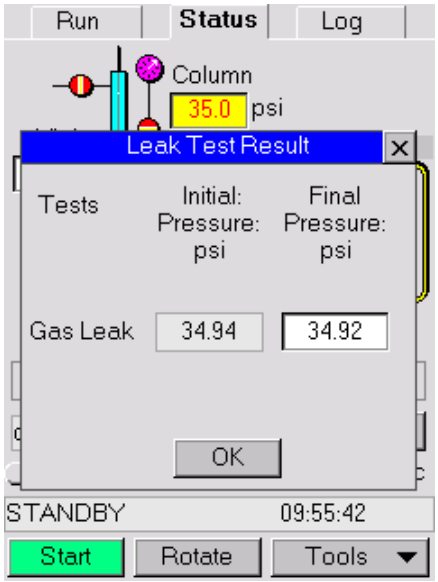


Figure 27 Passed Leak Test

5. The HS sampling system is now a closed, pressurized system. The pressure, displayed on the PPC tab, must not drop by more than 1 psi over a period of 30 seconds.

6. If a leak is detected, check all of the connections with a helium Leak Hunter or concentrated ethanol and water solution (a 50% ethanol and 50% water mix). Once you have checked and no leaks are detected then run the leak test again.
7. Reduce the carrier pressure to the original pressure.
8. Unplug the fused silica tubing and ensure that there is no septum material blocking the fused silica tubing.

If you are leak testing the HS sampling system and it has failed the leak test, you should check the following connections first:

- O-rings in the upper and lower needle sealing elements.
- Carrier gas connection at the rear panel.
- Carrier gas connections from the regulator, including all filters and unions.
- Transfer line connection to the needle unit.
- Column connection at the injector outlet (headspace only).
- Leak check all nuts around the trap.
- Be sure the trap is not cracked or broken.
- Check complete flow path and all valve connections.

If you are testing the whole chromatographic system, leakage may be occurring at the GC connections.

NOTE: Ensure the HS sampling system is leak tight before connecting the transfer line to the GC.

Refer to the GC manual to leak test the injector and detector connections. The following list provides further locations to test the GC connections:

- The connection of the heated transfer line with the GC injector (septum).
- Column connection at the injector outlet.

Leak Test the HS 40/110 Trap

You must leak test all the connections for the GC as outlined in the above procedure. You must also leak test the trap and connections (including the manifold, column isolation, flow connection valves and the connection from the needle to the transfer line nut area) for the HS 40/110 Trap to determine that there are no carrier gas leaks.

1. Switch off the gas chromatograph. Let the instrument cool down.
2. Undo and remove the chromatographic column at the detector inlet or inside the GC oven at the transfer line connection.
3. Use a blanking plug to seal the column outlet. Seal capillary columns with a new, clean septum.
4. Open the door and locate the desorb outlet (the brass fitting as shown in the next photo). Close the desorb flow by sliding the fitting in (to open the fitting slide the fitting out, when the desorb valve is in the valve is off. See the following photo).

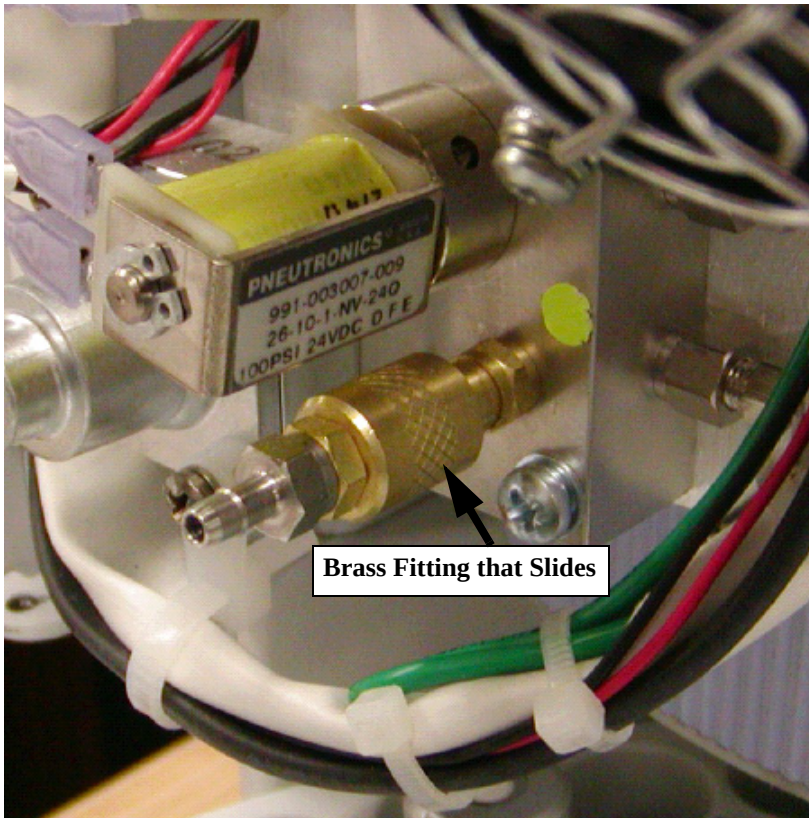


Figure 28

5. Set the carrier pressure to 45 psi. See *Carrier Gas* section earlier in this chapter.
6. Open the Tools drop down menu and select **Maintenance** and then select **Leak Test**. If the leak test fails you will get the following screen. Go to step 8 for the procedure of a failed leak test.

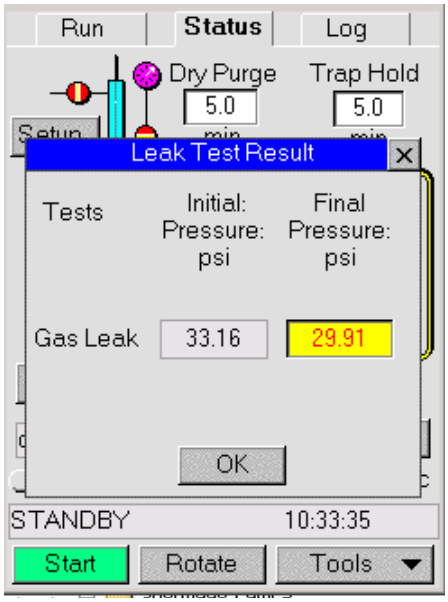


Figure 29 Failed Test (Red Text)

If the leak test passes you will get the following screen:

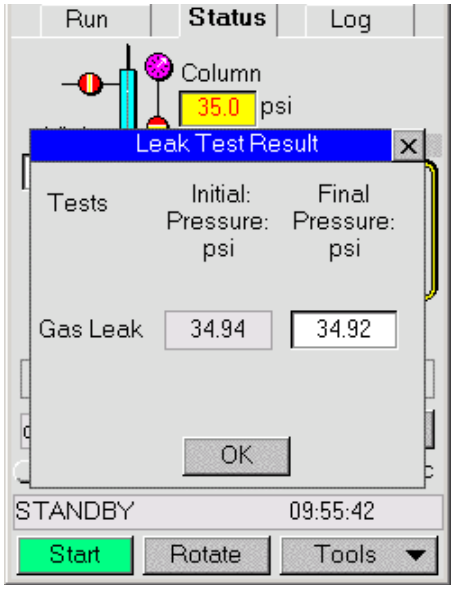


Figure 30 Passed Test (Black Text)

7. The HS sampling system is now a closed, pressurized system. The pressure, displayed on the PPC tab, must not drop by more than 1 psi over a period of 40 seconds.
8. If a leak is detected, check all of the connections with a helium Leak Hunter or concentrated ethanol and water solution (50% ethanol and 50% water solution). Once you have checked and no leaks are detected then run the leak test again. See the next page for more leak check details
9. Reduce the carrier pressure to the pressure specific to the method.
10. Because of the sensitivity of the HS trap make sure to trim the column where you plugged the end with a septum. Wear powderless gloves when you trim the column and reinstall.
11. Reopen the desorb flow path by **sliding the brass desorb fitting out** when finished.

WARNING

*Make sure, when you complete this procedure, to reopen the desorb flow path by sliding **out** the brass desorb fitting.*

If you are leak testing the TurboMatrix Headspace or the HS 40/110 trap sampling system and it has failed the leak test, you should check the following connections first (also check the gas leak schematic below):

- O-rings in the upper and lower needle sealing elements.
- Transfer line connection to the needle unit.
- Leak check all nuts around the trap.
- Be sure the trap is not cracked or broken.
- Check complete flow path and all valve connections.

NOTE: In the following figure no Internal Standard Accessory is

installed. If your system has an Internal Standard Accessory installed the leak test will be different.

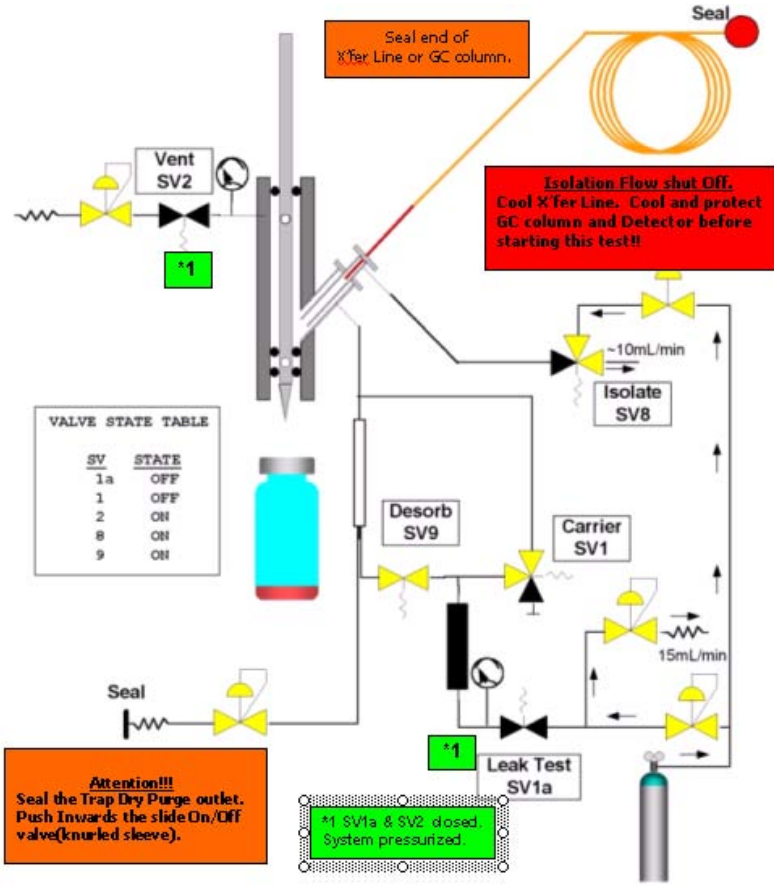


Figure 31

NOTE: *During shipping the valves may become loose and start to leak, if this happens, please go the* ***HS 40/110 Trap Valve Leak Test Procedure*** *in the Operation chapter.*

Operation 3

Introduction

TurboMatrix Headspace and the HS 40/110 Trap

Basic operation of the instrument is covered in this section. Method parameters are listed along with a brief description. A detailed discussion of HS method parameters and method development are provided in *Chapter 5*.

Safety Precautions—To protect yourself from harm and to avoid damaging the instrument, please observe the following notes:

- Before using this instrument, read and observe the safety information in *Chapter 1*.
- Do not attempt to analyze sample carbon disulfide or other solvents with a self-ignition point of 100 degC or less.
- Always follow the correct safety procedures and the manufacturer's recommendations when using any solvent. Refer to the MSDS sheets for compound-specific information.
- Do not touch moving parts of the instrument during operation.
- Do not operate the instrument with any covers or parts removed.

Supplies—The TurboMatrix and HS 40/110 trap is shipped without sample vials, septa, caps or a crimper tool. These parts are necessary to operate the HS and are available either individually or in the HS Starter Kit (B050-5601).

CAUTION	*Using sample vials, caps and septa other than those supplied by PerkinElmer may result in improper operation of the TurboMatrix Headspace or Trap Headspace Sampler. Damage to the instrument and/or loss of sample materials or data resulting from the use of sample vials, caps and septa* ***not*** *supplied by PerkinElmer may occur.* ***The subsequent service visit to remedy the situation, caused by the choice to use these non-PerkinElmer sample vials, caps and septa is not included under your warranty or service contract agreement.*** *Your Service Engineer can discuss the benefits of using only PerkinElmer sample vials, caps and septa.*

Before beginning operation ensure you have adequate supplies of vials, caps, septa and other sample handling materials. Ensure that the materials you are using are compatible both with your sample and your sampling method.

Powering up the Headspace Sampler

Before beginning operation of the instrument you may want to familiarize your self with the layout of the instrument and the basic concepts of headspace chromatography.

Before beginning operation you should:

1. Connect the electrical and gas supply lines; set the required flow rates and check that all gas connections are leak tight. Refer to *Chapter 2* for details.
2. Ensure the GC and any related support equipment and software are installed and are displaying a ready status. i.e., GC detectors, GC data acquisition systems, etc.
3. If you have any options installed they will be turned on with the unit, and allowed to warm-up or cool down as required. Refer to the documentation supplied with each installed accessory.

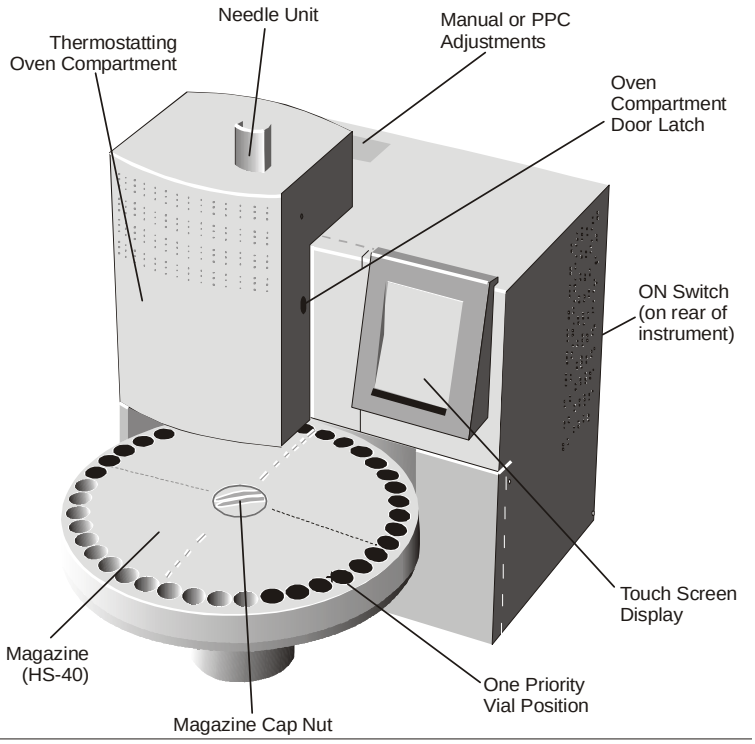


Figure 32 Layout of the TurboMatrix Headspace Sampler

4. Press the On switch. The power switch is located on the rear panel of the HS.
5. The HS splash screen is displayed.
6. Press anywhere on this screen.
7. The log in message displays. Enter your password and press OK. **hstd** is the default password.

   The local HS control interface displays.
8. If you are using the HS control software, connect the computer to the instrument as outlined in the control software manual. Start the software and install the HS on the Lab tab as outlined in the control software guide. Ensure the computer is communicating with the instrument.
9. When the HS is first powered up, it starts an

initialization process lasting approximately 10 seconds and resets the hardware to default status.

During initialization, vials, which are still in the thermostatted oven are unloaded. For this purpose the instrument looks for one empty magazine location in outside ring to unload the vials. If there is no empty location, the initialization process will be interrupted and the following error message is shown on the display: Magazine Full. Refer to *Loading the Magazine* on page 148 for details.

10. If the initialization process was interrupted you must remove sample vials from the magazine before you can restart the initialization process again.
11. Once the vials have been unloaded from the oven, reload your sample vials and then begin operation.

Setting the Carrier Gas Pressure

Once the gas has been connected at the rear of the instrument and you have established a flow of carrier gas through the system, you will need to set the carrier gas pressure through the transfer line. If you have connected the fused silica transfer line to the GC for splitless injection (direct connection), or installed the GC capillary column directly to the HS needle unit (on-column connection), then the HS will also supply the GC carrier gas. You must establish the column flow as well as set any pressure or flow programs that are required as part of the GC method.

Split Operation: In this configuration, you have connected the transfer line to the GC injector. The carrier gas pressure that you set at the HS moves the sample from the vial through the transfer line and onto the GC column.

To set the carrier gas flow for normal operation:

1. Connect the transfer line to the GC as outlined in the section *Installing the Heated Transfer Line* in the Installation chapter.
2. Determine the column head pressure. This information will be part of the GC method.

NOTE: Set the split vent flow to a typical setting of 25 to 30 ml/min.

3. Set the HS carrier pressure at 5 to 10 psi higher than the GC column head pressure. If the column head pressure is 10 psi, then set the HS carrier pressure to 15-20 psi.
4. If you have manual pressure control adjust the pressure regulator until the desired value is displayed on the PPC tab. Switch to the PPC tab and enable actual view so that you are viewing the current values rather than the set points. See *PPC Tab* on page 216.
5. If you have PPC control, enter the desired value on the PPC tab and allow the HS to reach the set point. See *PPC Tab* on page 216.

You must also ensure the needle purge flow has been established.

Splitless Operation: In splitless operation the fused silica transfer line is connected directly to GC column inside the GC oven (direct connection). Alternatively, the GC capillary column is brought through the injector and connected directly to the HS sampling head (on-column connection). In this configuration the HS supplies the total carrier gas flow for the HS-GC system. Set the pressure at the headspace to the correct head pressure your column requires (i.e. 30m, 0.25u, column = 16 psi).

Splitless operation is necessary for cryofocusing. Splitless sampling is less suitable for capillary columns that have a low pressure drop (i.e. 0.53 mm columns).

Direct Connection: In the direct connection configuration, the transfer line and the analytical column are directly connected together, using a butt connector, inside the GC oven. Two types of butt connectors are available; press fit connectors, which can only be used once, and standard low-dead volume unions, which can be reused.

To set the carrier gas flow for direct connection:

1. Before connecting the transfer line to the GC injector, ensure there is gas flow through the transfer line.
2. Connect the transfer line to the GC as outlined in the procedure *Installing the Heated Transfer Line at the GC for Direct Connection* previously in this chapter.

3. Determine the column head pressure. This information will be part of the GC method and should be shown on the GC display. Refer to the GC user's manual for further information.
4. Set the HS carrier pressure so that it is the same as the required column head pressure. If the column head pressure is 10 psi, then set the HS carrier pressure to 10 psi.
5. If you have manual pressure control adjust the pressure regulator until the desired value is displayed on the PPC tab. Switch to the PPC tab and enable actual so that you are viewing the current values rather than the set points. See *PPC Tab* on page 216.
6. If you have PPC control, enter the desired value on the PPC tab and allow the HS to reach the set point. See *PPC Tab* on page 216.
7. Turn off the supply of carrier gas at the GC.
8. You may want to check the column flow rate by connecting a flow meter to the outlet of the GC column.

On-Column Connection: To perform on-column sampling, the GC capillary column is taken through the transfer line sleeve and connected directly to the needle unit (*Installing the Heated Transfer Line On Column Connection* previously in this chapter). As with splitless operation, the HS supplies the total carrier gas flow for the HS-GC system.

To set the carrier gas flow for on-column sampling:

1. Connect the capillary column to the sample head as outlined in the section *Installing the Heated Transfer Line* or in the *HS 40/110 trap* in the Installation chapter.
2. Determine the column head pressure. This information will be part of the GC method.
3. Set the HS carrier pressure so that it is the same as the required column head pressure.
4. If you have manual pressure control adjust the pressure regulator until the desired value is displayed on the PPC tab. Switch to the PPC tab and enable actual so that you are viewing the current values rather than the set points. See *PPC Tab* on page 216.

5. If you have PPC control, enter the desired value on the PPC tab and allow the HS to reach the set point. See *PPC Tab* on page 216.
6. Turn off the supply of carrier gas at the GC. Monitor the system flow from the PPC tab.
7. Check the column flow rate by connecting a flow meter to the outlet of the GC column.

NOTE: It is assumed that if a cylinder containing an Internal Standard gas is used, its pressure regulator outlet is connected to the respective inlet at the back of the trap unit.

The Touch Screen Display

You will control the operation of the instrument through the touch screen display. With a stylus or your finger lightly touch the screen to enter data and issue commands.

NOTE: Do not use sharp objects such as pens and pencils to activate the touch screen as you may damage the display.

Using the three screen tabs, you can control the operation of the HS. The touch screen interface allows you to communicate directly with the HS so that you can:

- set and view current configuration parameters (Status tab)
- run analyses using one or more methods (Run tab)
- view instrument information (Log tab)

With the PPC option installed you can control all of HS functions directly from the touch screen. You can then monitor the carrier pressure from the touch screen. If PPC is not installed, you must use the pressure regulator to set the carrier pressure.

The interface consists of three tabs. The Status tab provides information on the current HS settings. You can also make changes to the HS parameters directly from the status screen. Changes you make on the Status tab will take effect immediately unless an analysis is in progress. Editing parameters will be disabled until the analysis is complete. You can use the Status tab to create and test new methods. Alternately, you can save or recall the methods using

the Tools button on the Run page and selecting the Method editor option.

The Run tab allows you to create a sequence of methods to be used on a series of samples. You create a sequence by selecting a range of vials and then specifying a method by which these vials will be analyzed.

The Log tab allows you to view the analysis history of the HS. An entry will be made whenever a vial is analyzed. Entries will also be made if an error occurs.

The Run tab, Status tab and Log tab have three buttons displayed on the bottom of the touch screen. These three buttons are **Start**, **Rotate/Load** and **Tools**.

The **Start** button will start a method or sequence.

If the system is not running the **Rotate** function will be visible. If you press this button the magazine will rotate so that you can load samples with the system off. You can press this button while the system is running and you will be able to add samples to the magazine while the analysis is in progress.

The **Tools** menu provides a drop down menu of options available such as Method Editor, Save As, Preferences, Calculator etc.

The Run Tab

Once you have created a method by which to run the samples, you will load the magazine and run the samples from the Run tab. See *Creating a New Method* in the *Method Development* chapter for details.

You can run samples using the archive or a saved method or a sequence of saved methods.

The options on the Run tab are determined by selections that have been made on the Method screen.

Single Method Operation

To facilitate operation for routine analyses, single method operation provides access to the vial range only. Single method operation and the desired method are selected by touching the method window and selecting the correct method from the choices. See *Single Method Operation on the HS* on page 149 for details

.

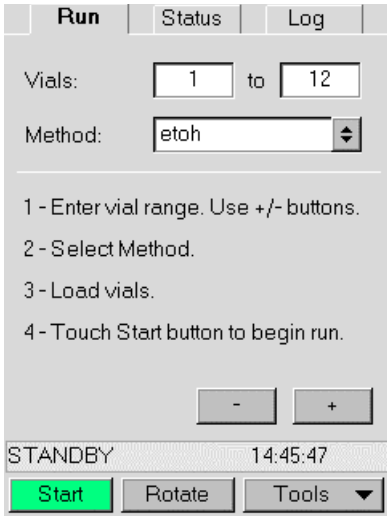


Figure 33 Run Tab - Single Method Operation

To start single method operation:

1. If a single method operation with a pre-selected method has been selected, then you only need to enter the vial range. The selected method is displayed.
2. Beside the **Vials** option, you enter the start and stop vials. Press the desired entry box to select the option.
3. Press the plus or minus button to select the desired start vial, then enter the desired end vial.
4. From the **Method** drop-down box, select the method to be used for the selected range of vials.
5. Press the **Start** button to begin the analysis.
6. A Start Run window will appear. Press the **OK** button.

NOTE: A message will appear if the instrument is not at equilibrium and what functions are not ready.

The HS will configure itself based on the method parameters. The instrument status will not start until the instrument reaches all of the set points. When the GC and the HS are ready a vial will be loaded into the oven. The vial will be thermostatted and the analyses will continue as determined by the method.

If you are using the HS 40 or HS 110 with the 15-vial oven, the first vial will be loaded (up to 12 vials at a time), and then vials are loaded as required based on the time established by overlapping thermostatting. For more detailed information on allowing access to various Run Modes see the *Preferences Tab* Section (page *135*) later in this chapter.

Creating a Sequence

You can use a sequence of methods to analyze a series of vials. The options on the Run tab allow you to select a range of vials and then specify a method to analyze the samples. You then select the next range of vials and select another method. You can also analyze the same range of vials using different methods. The sequence can be used on a daily basis or it can be edited as required.

You will not be able to edit any of these methods if method editing has been disabled from the Tools button.

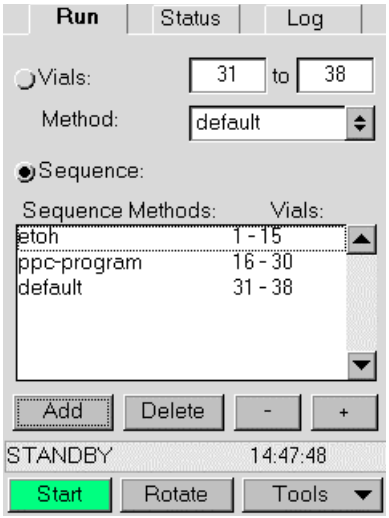


Figure 34 Run Tab - Sequence Operation

To create a new sequence refer to the Run Configuration in Preferences:

1. Select the **Run** tab.
2. Press the **Start vial entry** box and then press the plus or minus button to select the desired start vial. Next, enter the desired end vial.
3. From the method drop-down box, select the method to be used for the selected range of vials.
4. Press the **Add** button to add the entry to the sequence.
5. Repeat these steps until you have created the desired sequence. To delete an entry from the sequence, select the entry in the list and press the Delete button. To change the vial range, you must add a new entry with the revised vial range and then delete the old entry.
6. If you edit the methods called by your sequence, the sequence will use the revised methods. If you need to edit a method but do not want to affect your sequence, then save the revised method with a different name.

7. You can use up to 8 methods in your sequence. If you need to use more than 8 methods, you must use the PC control software (optional).

NOTE: You will not be able to edit any of the selected methods if method editing has been disabled from the Tools button.

8. Press the green Start button to run the sequence.

The HS will configure itself based on the method parameters. The instrument status will be Not Ready until the instrument reaches all of the set points, at which time it will become Ready. When the GC and the data handling system are ready, a vial will be loaded into the oven. The vial will be thermostatted and the analyses will continue as determined by the method.

If you are using the HS 40 or HS 110 with the 15-vial oven, the first vial will be loaded, and then vials are loaded as required based on the time established by overlapping thermostatting.

Status Tab (Headspace and HS 40/110 Trap)

The HS Status tab consists of four views: Temperature, Timing, Option and PPC. You select the desired view by selecting the corresponding radio button on the lower half of the touch screen. See the following figure. You can view the actual settings as they are on the HS or you can view the set points. When you are viewing the set points, the parameters are displayed in black. When you are viewing the actual values, the parameters are highlighted in yellow and displayed in red, until they have come into control.

The name of the method that is currently loaded on the Run tab is also displayed. You can change the settings and then save the revised method.

Normally, you will use the status display to monitor the instrument. You can also use the status tab to create a method. By using the status tab, rather than the method editor, you can enter parameters, run an analysis, change the settings based on the results and then perform another analysis. You can proceed in this manner until you have obtained the desired method parameters. Once the method is complete you can save it and use it on the Run tab.

NOTE: Additional Headspace trap features in the HS 40/110 Trap

section beginning on page 114.

Temperature Screen (Headspace and HS 40/ 110 Trap)

There are three standard temperature settings for the HS. The needle temperature, the transfer line temperature and the HS oven thermostatting temperature. If you have the cryofocusing accessory installed, you will also set the cryofocusing temperature from this tab.

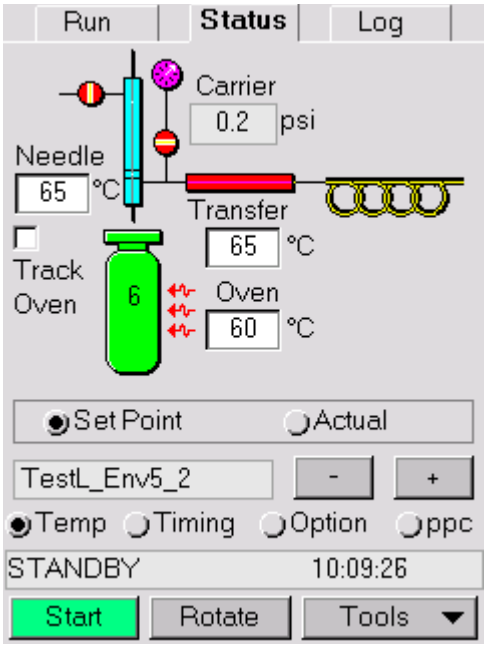


Figure 35 Temperature Tab

Entering a value of zero for any temperature parameter disables the related heater. For example, if you enter zero for the transfer line temperature.The transfer line heater will be turned off and the transfer line will be at ambient temperature.

To set the temperature:

1. Press the **Temp** radio button to switch to the Temperature screen.

2. Press the **Set-Point** option. The current temperature set-points will be displayed.
3. Press the **Needle** or **Transfer** entry box. A highlighted option indicates that it is active.
4. Press the **+** or **–** buttons to increase or decrease the temperature to the desired value.
5. The HS will cool or heat the needle or transfer line to the desired temperature. Allow some time for the instrument to reach the new value.
6. To save your settings as a new method or to update the existing method, press the **Tools** button and select **Save As**.
7. To save the method under the existing name press **OK.** To create a new method, enter a new name for the method by pressing the method name. An alphabetic keypad is displayed. To enter numeric characters, press the **Num** key. Press **Shift** to obtain uppercase characters. Press **Alpha** to return to the alphabetic keypad.
8. Press **OK** to enter the new method name and then press **OK** to save the method and return to the Status tab.

You can switch to the **Actual** screen to view the current needle temperature.

Needle Temperature—The needle is heated so that the headspace sample does not condense in the needle during injection. You must keep the needle warmer than the thermostatting temperature.

The needle temperature should be high enough to prevent condensation but not so high that the septum is burned with a needle that is too hot. For best reproducibility, set the needle and transfer temperatures to a value 5-10 ºC higher than the sample temperature. You must also consider the GC oven and injector temperatures. It is recommended to set the transfer line and inject needle temperature to a similar temperature. Typically the injector is a higher temperature.

The needle temperature can be set to any value between 35 and 210 ºC, in steps of 1 ºC. If you set the temperature to 0, the heaters are turned off.

Transfer Line Temperature—Set the temperature at or slightly above (5-10 ºC) the HS oven thermostatting temperature. You must also consider the GC oven and injector temperatures.Please refer to the section above, *Needle Temperature*, for recommendations.

However you need to remember that in the heated transfer line, the headspace gas is a mixture of air with trace concentrations of the analytes. Setting a high temperature may cause sample decomposition by oxidation.[1]

The transfer line can also be set to any value between 35 and 210 ºC, in steps of 1 ºC. If you set the temperature to 0, the heaters are turned off.

Oven Thermostatting Temperature—This is the temperature at which you will equilibrate your sample. The temperature must be set so that the maximum amount of analyte is moved into the headspace in the minimum amount of time. You must also consider the thermal stability of your sample when you set the thermostatting temperature.

WARNING

Always run the instrument with the front door panel closed to prevent injury. *Over heating or pressurization the vial may cause the vial to implode.*

The oven can be set to any value between 35 and 210 ºC, in increments of 1 ºC. If you set the temperature to 0, the heaters are turned off.

Temperature Mode (activating the track oven option)**—**These three temperatures can be combined so that when you raise or lower the combined temperature, all three settings are adjusted. If for example you enable the **combined** option and raise the temperature by 5 ºC, then the needle, the transfer line and the thermostatting temperature will all be raised by 5 ºC. If you choose the **separate** option, then each temperature can be set independently.

Cryofocusing Temperature (Headspace Only)—This option is only available if the cryofocusing accessory is installed. This

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 71

accessory allows you to concentrate your sample at the head of the GC column by cooling the head of the column. See *Cryofocusing Accessory* on page 172 for details. You can set the temperature to any value between -180 and -10 ºC in steps of 1 ºC.

Timing Tab (Headspace and HS 40/110 Trap)

You can view the timing values for the HS from the Timing tab. The exact values that you enter here will be based on your application. The HS timing parameters will also be affected by the GC method. Once all of the correct timing values have been entered, the period from injection to injection (PII) can be calculated.

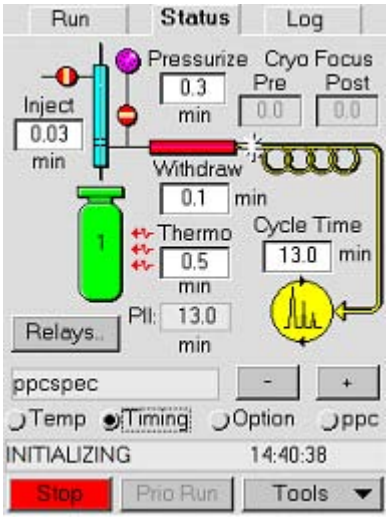


Figure 36 Timing Tab

Thermostatting Time—The thermostatting time should be the shortest time the sample has to be thermostatted. The analytical result will not change if the thermostatting time is longer than the time needed for equilibration. Excessively long thermostatting should be avoided, however, because some samples may be sensitive to prolonged heating. You can set the thermostatting time to any value between 0.1 and 999 minutes. The default value is 12 minutes.

Pressurization Time (Headspace only)—After equilibrium has been reached, the vial is pressurized by the carrier gas to a pressure equal to the sampling head pressure (P2). You can set the

pressurization time to any value between 0.1 and 999 minutes. The default value is 1 minute.

Injection Time (Headspace only)—At the end of the pressurization time, the carrier gas supply is interrupted by closing a valve in the carrier gas supply line and the pressurized gas in the vial expands onto the column, resulting in a flow of the headspace gas from the vial to the column. You can set the injection time to any value between 0 and 9.99 minutes. The default value is 0.04 minutes.

Injection Volume (Headspace only)—The injection volume (mL) is based on the column flow rate and the injection time. If you have opted to display a volume rather than a time on the Options tab, then you will enter an injection volume here and the corresponding injection time will be displayed. You can set the injection volume to any value between 0 and 10 mL.

Based on the injection volume that you enter, and the column flow rate an injection time is calculated. The injection time corresponds to the flow rate measured at the end of the GC column under normal atmospheric pressure and temperature conditions.

NOTE: The calculation of the injection time for the entered volume is based on the assumption that the vial pressure remains constant during the sampling time and no other gas is supplied to the column. This would be the case with a direct connection or splitless configuration.

GC Cycle Time—The GC cycle time is the minimum time between a sample injection and the time at which the GC is ready for the next injection. This time will be determined from the GC analysis time and the time required to return the GC to its ready state.

The Cycle Time value must be greater than or equal to the GC run time plus the GC oven equilibration time and the GC oven cool-down time. The HS will use this value to calculate when to begin the thermostatting time. If you are running an HS-40 or an HS-110, the cycle time will be used to determine at which time the vials should be loaded into the oven in order to run vials successively without wasted time in between injections.

For normal Headspace applications the cycle time is short but with Trap operation the cycle time is much longer. Make certain to enter a longer cycle time when using a trap otherwise you will see an apparent block on entering the GC cycle time.

The cycle time can be set to any value between 0.1 and 999 minutes. The default time is 5 minutes.

NOTE: Laboratory temperatures can strongly influence the GC oven cool-down time and can vary. The GC cycle time setting should take into account the longest oven cool-down time expected during the analytical sequence.

Withdrawal Time (Headspace only)—The length of time after the injection, before the sample needle is withdrawn from the sample vial or lowered into the vent position. During this time the needle remains in the vial. The withdrawal time can be set to any value between 0.1 and 99 minutes. The default time is 0.2 minutes.

Pre/Post Cryofocusing Time (Headspace only)—These optional parameters appear only if the cryofocusing accessory is installed. They define the cryofocusing time before and after sample injection. Pre and post-cryofocusing time can be used to optimize the cryofocusing duration before and after sample injection.

The pre-cryofocusing duration allows the head of the GC column to cool to the set cryofocusing temperature. The post-cryofocusing time maintains the head of the column at the low temp until the sample has been collected and unretained compounds have cleared the column. These values are determined when optimizing the cryofocusing. The total cryofocusing time comprises the pre- (before and during sample injection) and post- (after injection) cryofocusing times.

Both pre- and post cryofocusing time can be set to any value between 0.1 and 99 minutes.

Relays—This dialog box provides access to the timed events for the relays on the options board. See *Timed Events* on page 163 for details on connecting remote accessories and programming the on and off times.

Period from Injection to Injection (PII)—The HS will calculate the period from injection to injection (PII) from the timing values that you have entered. The PII value is shown on the Timing tab.

For optimum sample throughput (i.e. the greatest number of samples analyzed in the shortest time), it is essential that the PII value is only slightly longer than the cycle time. The PII value lets you see the effects of your analysis timing changes on vial throughput.

If you are running the 15-vial oven, it is possible to overlap the thermostatting times and reduce the PII.

The Status Tab Option Tab (Headspace)

The Option tab provides access to the headspace method options. Select the options that you have installed. If you have an option installed, but do not need to use it then de-select here. You can enable vial venting, the shaker, cryofocusing and high pressure injection from this tab. High pressure injection is only available if you have the PPC option installed.The cryofocusing option will only be available if the option has been installed on the HS and in the GC column oven.

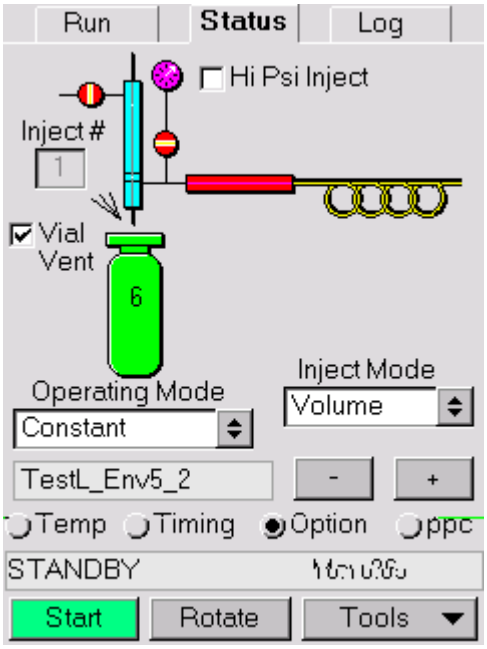


Figure 37 Option Tab

You will also select the operating mode and the injection mode from this tab.

Injection Mode—The volume of the injected sample can be entered as an injection **Time** or an injection **Volume**. The injection volume

(mL) is based on your entry for the column flow rate. The injection volume corresponds to the flow rate measured at the end of the column under normal atmospheric pressure and temperature conditions.

If you select injection **Volume** as your mode of injection, specify the column flow rate (Tools—>Preferences—>Config Tab) and the desired injection volume. The HS will automatically calculate the corresponding injection time. If you select **Time** as the injection mode, the HS will determine the injected volume from the time you have entered, on the timing tab, and the column flow rate.

Vial Venting—To enable automatic venting of the vial set to a venting time. This parameter enables vial venting of the sample vial. The vial is vented after the withdrawal time. If you have enabled vial venting then you must set a vent time. This will be the length of time during which the vial is vented to atmosphere. Venting occurs immediately after the withdrawal time has elapsed. Default should be 0.3 min (18 seconds). Venting is used to allow for the safe handling of spent vials after analysis.

High Pressure Sampling—If you are using the high pressure injection option, you will set the injection pressure here. You must first enable the option on the Option tab. This option is available if you have PPC installed. You can enable it on the PPC tab (as Inject psi). For more information see the section, *Principles of High Pressure Sampling* in the Method Development chapter of this guide.

Water Trap—In cryofocusing, water is removed from the sample prior to enrichment using the water adsorption trap. Water from the sample is removed by adsorption onto a hygroscopic salt.

Operating Modes—Select Constant mode if you want to use the same thermostatting time for all samples that are analyzed by this method. Constant mode is the standard mode of operation. When you select constant mode, overlapping thermostatting is enabled if you have an HS 40 or HS 110 with the 15-vial oven.

In Progressive mode the thermostatting time for a series of samples increases automatically for each vial. Sample 1 is thermostatted for the value entered. Sample 2 is thermostatted for twice the entered value, Sample 3 for three times the entered value, and so on.

Select Multiple Headspace Extraction (MHE) mode if you want to perform multiple headspace extractions. The MHE function uses from 2 to 9 extraction steps per sample vial and pressurizes, injects, and vents each vial between extractions.

Number of Injections—This parameter defines the number of extraction steps in multiple headspace extraction methods. Valid settings are 1 to 9 injections.

Shaker—If installed and selected, the shaker can decrease the time needed for equilibration by providing continuous mixing of the sample in the vial during the equilibration process. See information on the Vial Shaker accessory in the *Accessories* chapter later this manual for more details on the shaker option as well as considerations when setting the duration of thermostatting time when the shaker is used.

The shaker only starts an automatic shaking program when a headspace method which utilizes the shaker, is started.

In MHE mode, the optional shaker must be switched off. Shaking is not possible during MHE analyses since the needle remains inserted in the vial during all of the analyses.

NOTE: See additional options that are available in the section "Status Tab Option Tab for the HS 40/110 Trap" on page 122.

The Status Tab PPC Tab

Programmed Pneumatic Control (PPC) is the electronic control of the carrier gas pressure. You can set the carrier gas pressure from the PPC tab, then the PPC control module regulates pressure to that set-point.

Column Pressure—This is the carrier gas pressure for the HS system. The carrier gas is used to pressurize the vials and then carry the sample through the transfer line to the GC injector or column. You will set the carrier pressure on this tab even if the PPC module is not installed on your instrument.

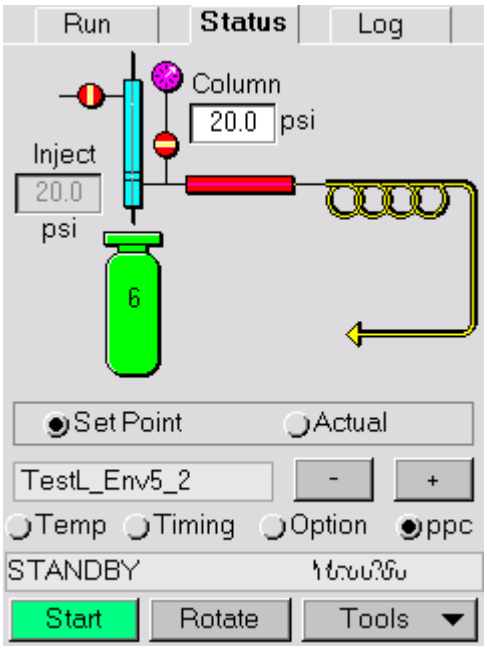


Figure 38 PPC Tab

Inject Pressure—This option is available and the pressure set if the system is PPC equipped and high pressure injection has been selected on the Option tab (see *page 114*). The Inject Pressure is applied to the sampling needle and the column head pressure from start of the pressurization time until the end of the withdrawal time and/or the end of the vial venting time if vial venting has been selected.

HS 40/110 Trap

Status Tab Temp

The Temperature tab on the Status tab for the HS 40/110 trap offers an additional option of inputting the trap temperatures.

Trap Low Temperature—This is the temperature to be used for the absorption of the analytes during trap loading. It should be low enough in order to trap the components of interest but not too low. Temperatures that are too low could cause moisture condensation in the trap itself. The default value is 40 ºC. The range of acceptable values is 0 to 100 ºC, in increments of 1 ºC. The minimum attainable temperature is approximately 2 ºC above the ambient temperature.

Trap High Temperature—This is the temperature to be used for the trapped components of vaporization and their subsequent injection into the column. It should be high enough to ensure fast, complete vaporization of the sample. The default value is 280 ºC. The range of acceptable values is 0 to 400 ºC in increments of 1 ºC.

NOTE: The default Trap Maximum Temperature is 400 ºC. This value depends on the trap material.

The name of the method that is currently loaded on the Run tab is also displayed. The settings displayed here are from this method. You can change the settings and then save the revised method.

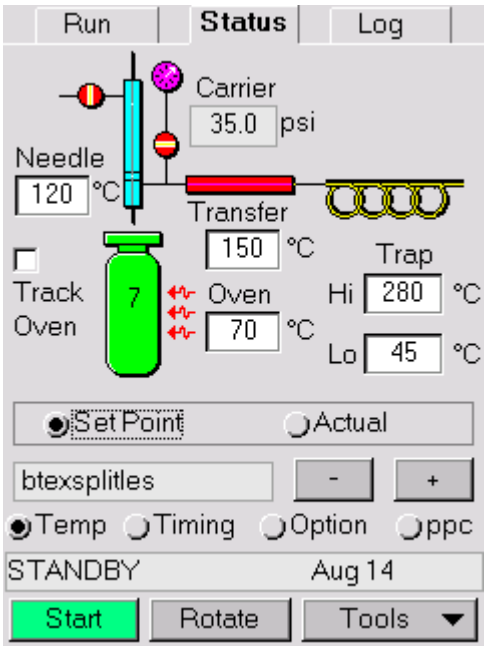


Figure 39 HS 40/110 Trap Status Page Temp Tab

Status Tab Timing Tab for the HS 40/110 Trap

The Timing tab on the Status tab for the HS 40/110 trap offers some options for the trap.

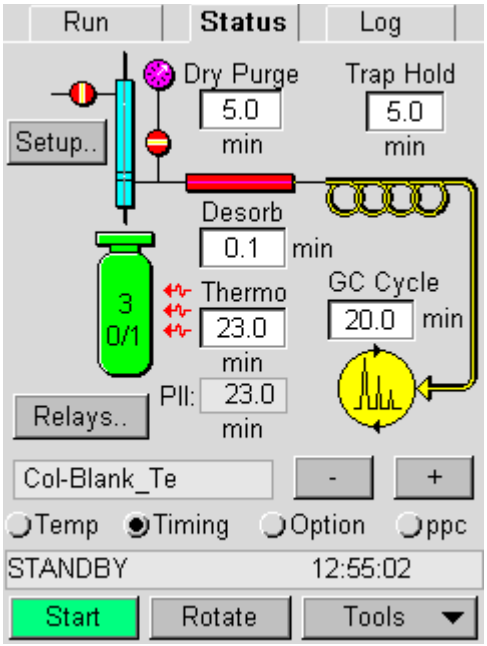


Figure 40 HS 40/110 Trap Status Tab Timing Tab

Dry Purge — This option allows you to input the number of minutes required for the dry purge. The dry purge allows for helium to purge the trap of water at the lowest trap temperature entered and at the desorb flow rate. The amount of time needed for the dry purge will vary depending on the thermostatting temperature, the type of sample you are running and the Headspace Trap Cycle time. If the FID extinguishes during your analysis you will have to increase the dry purge time.

The **Trap Dry Purge** range of values is 0.1-99 min, in steps of 0.1 min. The **Trap Dry Purge** is carried out under a pressure equal to the used in the **Desorb** step.

The **Desorb time** is the time that it takes the analytes to be released from the trap into the transfer line.

Trap Hold — This option allows you to input the number of minutes that the trap will maintain the maximum temperature. In order to release the analytes from the trap, the maximum temperature must be maintained for a sufficient amount of time. A recommended trap hold time is five minutes. In addition, this will help clean up the trap to prepare for the next sample.

Setup — By touching the **Setup** button the HS Trap Timing screen will appear. This allows you to set the HS Trap Timing based on the cycles, pressure and decay time.

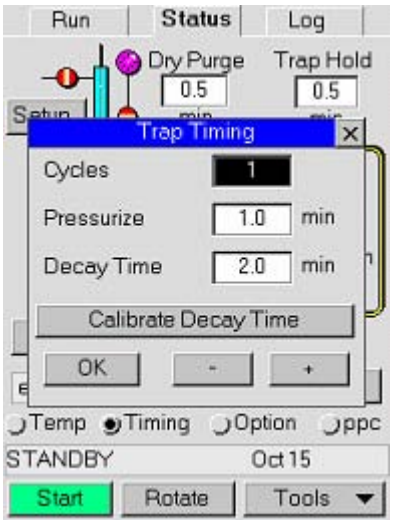


Figure 41 HS 40/110 Trap Setup Screen

The Timing screen has the following variables:

Cycles—you can set the cycle number from 1 to 4. This number represents the number of times that the vial pressurization and trap load cycles will be performed per vial. For example, should you select 4 cycles and a vial pressure of 40 psi. More than 99% of the material will be adsorbed and focused in the HS 40/110 trap.

Pressurization—After equilibrium has been reached, the vial is pressurized by the carrier gas to a pressure equal to the Vial pressure (See PPC tab). You can set the pressurization time to any value between 0.1 and 999 minutes. The default value is 1 minute.

Decay—The time for the complete headspace vapor to be decayed out of the vial on to the trap.

To calibrate the decay time press the **Calibrate Decay Time** button to bring up the calibration screen.

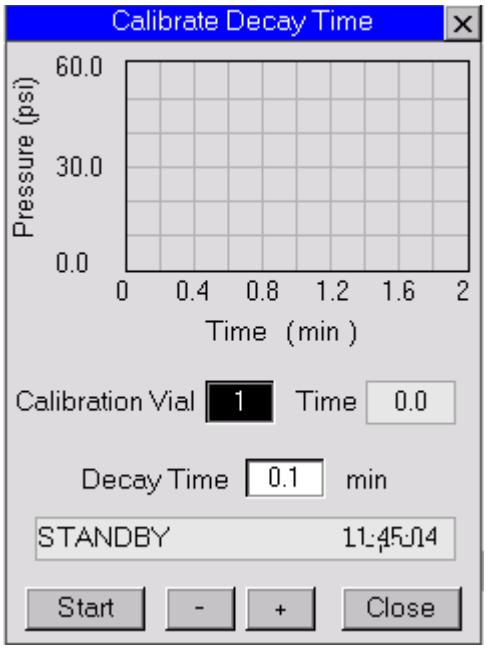


Figure 42 HS 40/110 Trap Calibrate Decay Time Screen

Place a vial of a typical sample in position 1. The HS 40/110 trap will thermostat your calibration vial for two minutes, then pressurize it to the vial pressure in the method. The calibration vial needs to have the same volume and composition as the samples. This will determine the decay time and this decay time can be entered in the decay time position.

NOTE: Later you will be able to determine if you had a vial that was not sealed properly or was under filled or over filled.

Gas Leaks Detected by "Monitor Vial Integrity" - Dynamic

Leak Test

Gas leaks can also be detected by the option **Monitor Vial Integrity** (see the *Preferences* section later in this section.)

Here it is fundamental that the Decay curve produced by the Calibrate Decay Time procedure gives a good decay profile. See in earlier chapter the Calibrate Decay Time description and notes.

To run this calibration, the following conditions should be strictly met:

- A non-leaking vial sealing and septum.
- The "calibration" sample should be identical to the ones to be analyzed.
- The correct sample volume must be used, the same as the volume which will be used for samples to be run.
- Check that the calibration curve profile is good. See the next figure for an example of a good decay profile. Here a safe Decay Time would be set at 1.6 min.

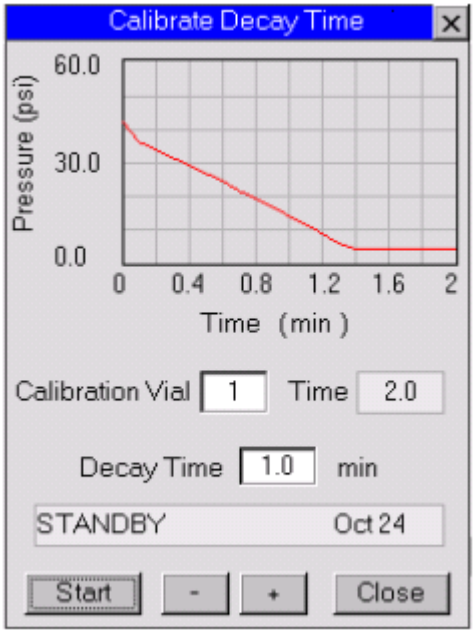


Figure 43 Calibrate Decay Time

For a given Vial Pressure, the decay slope and time will depend mainly on the sample volume.

- Smaller sample volume (larger headspace volume): small slope/gradient - slow pressure decay.
- Larger sample volume (smaller headspace volume): large slope/gradient - fast pressure decay.

Next, you should choose **Monitor Vial Integrity** in the SETUP tab of the **Preferences** screen.

Tools button ------> **Preferences** screen-------> **Setup** tab.

With this option enabled, the system will monitor and check each vial run in the method and report in the **Log** tab any deviation from the calibration curve profile; i.e.

- Slow pressure decay
- Fast pressure decay (see the following figure)

If **Stop Vial On Error** is also enabled in the **Setup** tab, the system will stop the run if a vial with such a mechanical problem is spotted.

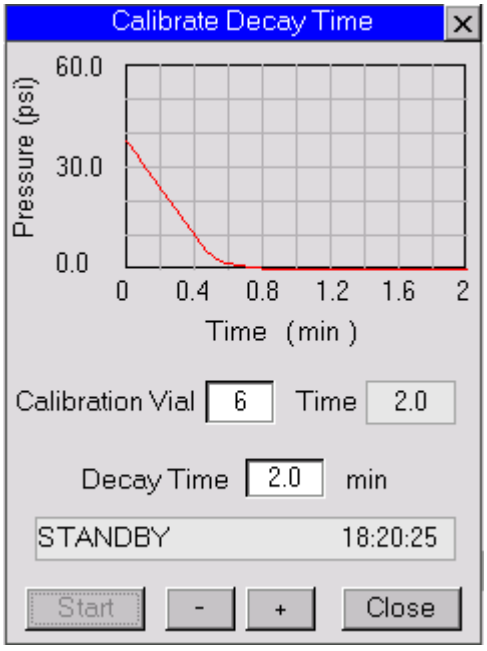


Figure 44 Example of Fast Pressure Decay Curve

If the calibration curve is correct and the same vials in content and volume are run, then a leak is probably present. It could be due to:

- Leak in the vial seal or septum.
- Leak due to needle sealing upper and lower O-rings or even the needle itself (scratched or dirty or bent).
- Leak at the tee-piece and connections between the sampling head and the back (in-board) inlet of the trap.
- Leak at the front end of the Trap (PTFE ferrule). Decay Flow is increased beyond the regulated value by the Trap Dry Purge Fixed Pr. Regulator (~50ml/min).
- Leak at the connection of the transfer line or column to the Sampling Head.
- The isolation flow is not correct (too low). The decay pressure branches to Transfer Line / column. Isolation flow

should be 12-15 ml/min.

- The Trap Dry Purge flow is high. The Fixed Pressure Regulator is possibly misadjusted or defective and gives higher flow (it should be ~50 ml/min).

Status Screen, Option Tab for the HS 40/110 Trap

The Option Tab for the HS 40/110 trap provides some additional options from those just listed with the Headspace only Options tab. On the Operating mode menu, select Trap.

In the Option screen for the HS 40/110 trap, you can set the following options:

- **Outlet Split:** If a split is desirable in your analysis, you should check this box. This setting will instruct the system to keep SV2, in the Needle purge outlet, open during the Desorption/Injection step. This split flow is preset in the factory at 15 ml/min and is not accessible to the user.
- **Dry Purge:** If Trap Dry purge is desirable, you should check this box. Dry purge pressure will be the same as the desorb pressure.
- **Internal Standard:** This field will not appear if this option is not installed in the HS Trap unit. If the internal standard. is present, you can inform the system that an internal standard will be used by checking this box.

*NOTE: When using the **Trap** in the Operating Mode you do **not** have an option for Inject Mode because you are sampling the entire vapor.*

The Operating Mode has the following choices in the drop down window in addition to the standard headspace modes:

- **Trap**-Standard trap mode using over-lapping thermostatting. Use the trap for concentrating the analytes before injection into the GC column.
- **Trap Clean**-uses high temperatures to vent the contaminates from the trap.
- **Trap Test**-a dummy injection is made into the GC where the material from the trap using the trap high temp is sent down

the column for analysis.

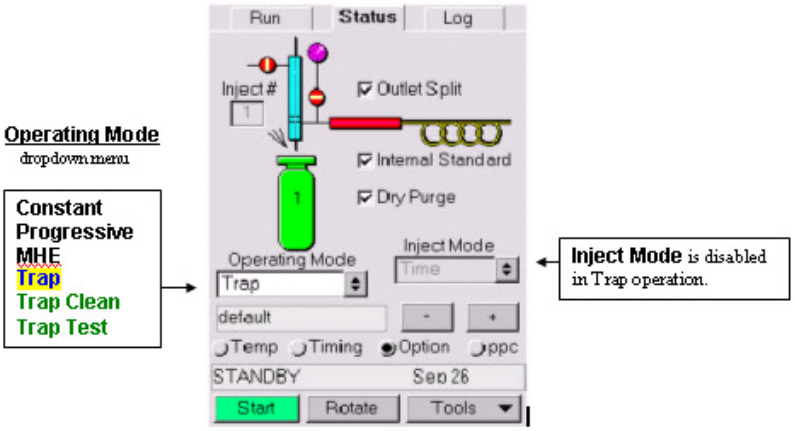


Figure 45 HS 40/110 Trap Status Tab Option Tab

Status Screen PPC Tab for the HS 40/110 Trap

The PPC tab on the Status screen for the HS 40/110 trap has the following options for the trap. The **vial** PSI box allows you to set the pressure value for the vial in the vial pressurization and the trap transfer step.

Column Pressure—This is the pressure that the PPC will apply at the end of the desorb step as the carrier gas column head pressure during the analysis. Its value depends on the column characteristics, the GC oven temperature program and the analysis requirements. It range of values is 0-60 psi or 0.1 psi.

Vial Pressure—This is the pressure the PPC applies during the step of Vial Pressurization. 40-50psi is a reasonable pressure value that will allow the system to transfer the maximum of the headspace vapor volume to the Trap with a minimum number of cycles. Too high a pressure would subject the system to rather rigorous conditions that may lead to gas leaks.

The range of values is 0-60psi, in increments of 0.1psi.

Desorb Pressure (Trap Only)**—**This is the pressure that the PPC applies to the Trap during the Desorption step.

The range of values is 0-60 psi, in increments of 0.1psi.

NOTE: During the Trap Dry Purge step, the PPC applies the same pressure to the Trap as the one set for the Desorption step.

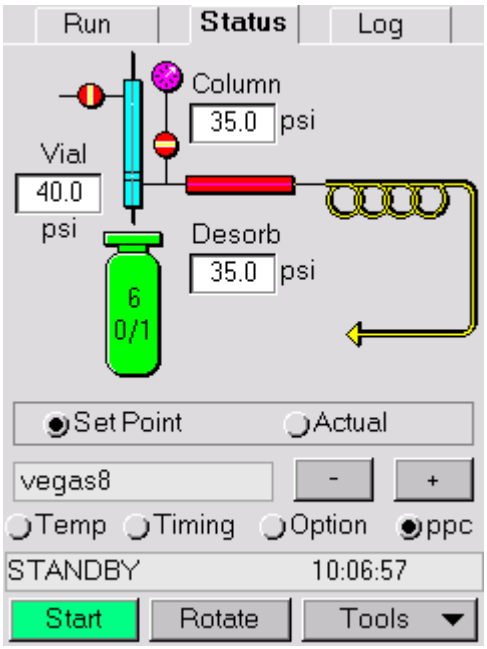


Figure 46 HS 40/110 Trap Status Tab PPC Tab

The Log Tab

Log Report—The Log tab allows you to view the analysis history of the headspace. An entry will be made whenever a vial is analyzed. Entries will also be made if an error occurs (for example, Vial Integrity).

The start time of the current analysis or sequence is displayed along with information on completed runs.

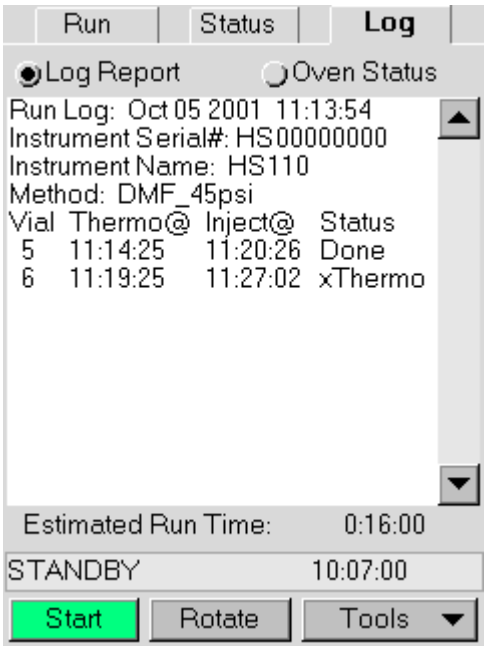


Figure 47 Log Report Tab

Oven Status—If you are operating the HS 40 or HS 110 with the 15-vial oven, you can view the status of the vials in the oven.

The current temperature of the oven is displayed. The number of the vials that are in the oven is also shown as is the location of the vial that is currently being sampled.

The current temperature of the heated oven and the direction of rotation are also shown.

The vial number, the method with which it was analyzed, the thermostatting time and the injection time plus the status of the run are displayed. If the sequence has been completed, then the time at which it was completed is also displayed.

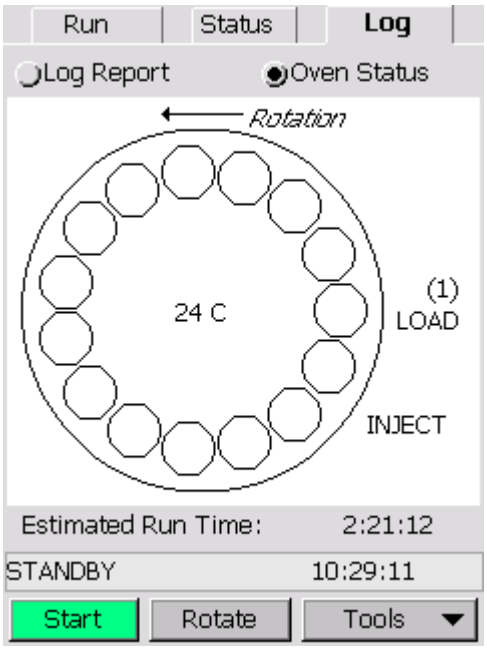


Figure 48 Oven Status Tab

The information is overwritten when a new sequence is started.

Decay (HS40/110 Trap only)—. If you set up your pressure decay properly (the time for the complete headspace vapor to be decayed out of the vial on to the trap) and the pressure in the vial is not consistent with your setup, an error message will be generated and displayed in this log provided that the **Monitor Vial Integrity** is chosen. For example, in the next screen, is a fast pressure decay for vial 15. There could also be a slow pressure decay for the vial that decays slower than the set decay time.

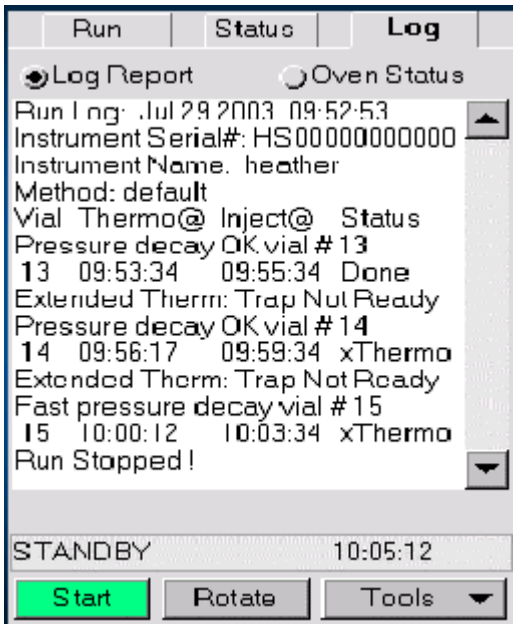


Figure 49 Pressure Decay Log Screen

Tools

Pressing the Tools button opens a pop-up menu of tools that enable you to perform various functions such as creating and editing methods and configuring the options available on your instrument.

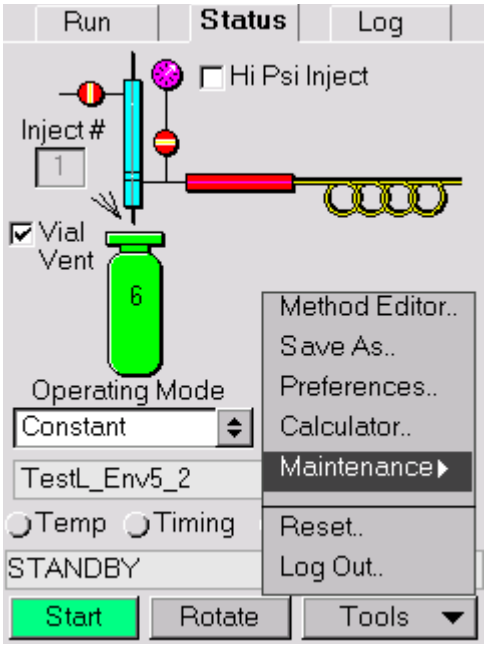


Figure 50 Oven Status Screen Tools Menu

Method Editor

The Method Editor command opens the Method Editor tab. This tab allows you to create and store methods for your application. You can store up to 9 methods. Once the methods have been saved, they can be recalled at any time for use in a sequence or can be recalled for editing. The Method Editor tab is similar to the Status tab in appearance. See *Chapter 5* for details on developing and testing methods.

Open the File Menu:

New Method—To create a new method, open the method editor, enter the desired method parameters and then save the method.

Open —To revise an existing method, select the Open command from the File menu. Select the method that you want to edit and press OK.

Save/Save As —Use these commands to save new methods or update existing ones.

To create a new HS method:

1. Open the Method Editor tab and enter or update the desired method parameters.

2. Select **Save** if you are updating an exiting method. The Method will be saved with the same name.

3. Select **Save** As if you are creating a new method. Touch the highlighted name field. Enter a new name for the method by pressing the name field. An alphabetic keypad is displayed. Enter the desired name. To enter numeric characters, press the Num key. To enter upper case text, press Cap.

4. Press **Alpha** to return to the alphabetic keypad.

*NOTE: If a name is already displayed in the name field press the **Red Return** key to delete the characters.*

5. Press **OK** to enter the new name for your method and then press **OK** to save the method.

6. Select **Exit Editor** from the File menu.

Activate—This command makes the currently selected command active. The method is loaded from memory and the instrument is heated or cooled to the new settings.

Delete —Use this command to remove an existing method from memory. You can store up to 9 methods. If you need to store more methods, you must use the PC control software. The control software allows you to store a greater number of methods for various applications.

Exit Editor—The Exit command closes the Method Editor and returns you to the default screen.

Test

Maintenance

The Maintenance command provides access to the following tests and maintenance support.

- Leak Test
- Valve Leak Test (HS 40/110 Trap only)
- Column Isolate (HS 40/110 Trap only)

These tests and maintenance procedures will be presented and discussed in the *Routine Maintenance* chapter.

The test command provides access to the leak test and the cryofocusing test. Use the leak test to ensure that your HS sytem is leak tight. See *Leak Testing the Headspace* in the Installation chapter for details on performing the leak test.

NOTE: Cryo Test is disabled in the HS Trap version.

The cryofocusing test enables you to determine the length of time required to reach the cryofocusing temperature. You will need this information to set the Pre-cryofocusing time. See *Pre/Post-Cryofocusing Time (Headspace Only)* on page 204 for details on performing the cryo test.

Log Out (Headspace and HS 40/110 Trap)

The Log Out command closes the tabbed interface and logs out. The HS splash screen is displayed.

Calculator

You can use the calculator in standard view to do simple calculations. The calculator can be used to do MHE calculations.

To perform a simple calculation:

1. Enter the first number in the calculation.
2. Click+ to add, - to subtract, x to multiply, or / to divide.

3. Enter the next number in the calculation.
4. Enter any remaining operators and numbers.
5. Click =.
6. Sqrt calculates the root of the displayed number. X^2 calculates the square of the displayed number.
7. Press the close button (x) on upper right corner of the title bar of the window to close the calculator.
8. If you are performing MHE runs, you can enter the integrated peak area and press MHE. Enter the integrated area of the second peak and then press MHE again. Enter as many runs as you have performed and press the = button after the last button to calculate the total concentration.

Reset

Use the Reset command to reset the instrument. This may be because of a fatal error or software problem. If the instrument is reset, you must load your last active method and allow the HS to reach all of its set points before beginning an analysis. If you reset the HS during a sequence adjust the start and stop vials so that the sequence includes only the vials that have not been analyzed.

NOTE: When you begin the new sequence the log information from the previous sequence is lost.

Preferences Tab (Headspace and HS 40/ 110 Trap)

You use the Preference tab to set the configuration options for the HS. The selections you make here enable or disable various options on the Status screen and in the Method Editor.

The Preferences screen contains four tabs related to the operation of the HS:

- **Run** tab
- **Config** tab
- **Set Up** tab

- **Connect** tab

The Run tab sets the options for the running samples. The Config tab contains information on the type of carrier gas, pressure units, various calibrations, trap setup etc. The Setup tab displays various HS options information. The Connect tab contains access to the communication parameters required for RS-232 connections and the Date/Time button.

Run Tab

The Preferences Run tab configures the options on the Status Run tab. Utilizing the options on the Preferences Run tab allow you to simplify the operation of the instrument for routine analysis.

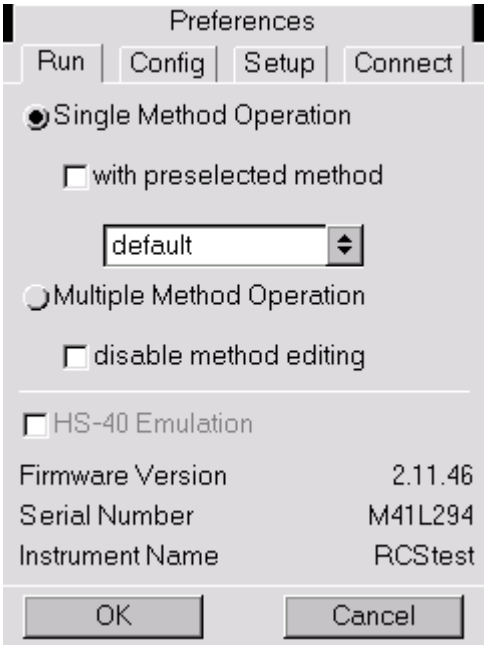


Figure 51

Single Method Operation—If you select single method operation the options on the Run tab allow you to enter a range of vials to be analyzed and allow you to select a method from a drop-down menu. These instructions are provided on the Status Run tab.

Single Method Operation with a Pre-selected Method—This option allows you to select the method to be used for the selected vial range. The Run tab is further simplified to allow the user to enter the vial range only. The method you select here displays on the Run tab. Enable the option and then select the desired method from the drop-down list of pre-programmed methods.

Multiple Method Operation—If you have selected multiple method operation, users are able to use any of the pre-programmed methods to run analyses or create a sequence.

Multiple Method Operation with Method Editing Disabled— You can disable method editing by selecting this option. The user can select any of the pre-programmed methods to run analyses or create a sequence, but the user cannot edit the methods in any way.

HS-40 Emulation—If you are using an HS 110, you can configure it to operate as an HS 40. If you enable this command, only vials from the outer ring will be loaded and the crane will not be used. In HS 40 emulation, the load and unload positions are not reserved. You can place samples in these positions.

You can use this mode if you are using low volume vials with adaptors or if you have fewer than 40 samples to run.

Show—Use this option to hide the Relay option on the Timing tab. If you need to program timed events to control external devices, ensure that the Relay option is enabled.

Config Tab

Carrier Gas—Select the type of carrier gas being used and the units to be displayed when a pressure value must be entered to complete a method.

Number of Injections—You can set the number of injections to be made before an indicator is displayed to the user that maintenance is required. Once the needle maintenance has been completed, as outlined in *The Sampling Needle* on page 243, then you can reset the counter by pressing the Reset button.

Maintenance Alarm—The needle needs to be maintained periodically. After every 500 injections the needle holder should be

removed to clean the needle from abraded sealing material. It is only necessary to change the sampling needle when it is damaged, or when you wish to change to another needle type. See *The Sampling Needle* on page 243 for needle maintenance instructions.

You can check the number of injections that have been made and then adjust the maintenance interval at any time.

HS 40/110 Trap Status Tab Config Tab

The trap can be heated to 400 °C without damaging the packing, but care must be taken that the analytes do not degrade at the high temperature. For example, if the halogenated hydrocarbons are present in the sample, the temperature should not exceed 325 °C. When the trap is heated to 325 °C, trimethyl benzenes are released quantitatively. For higher boiling species it may be necessary to use a higher trap temperature.

This page offers some additional options for the HS 40/110 trap.

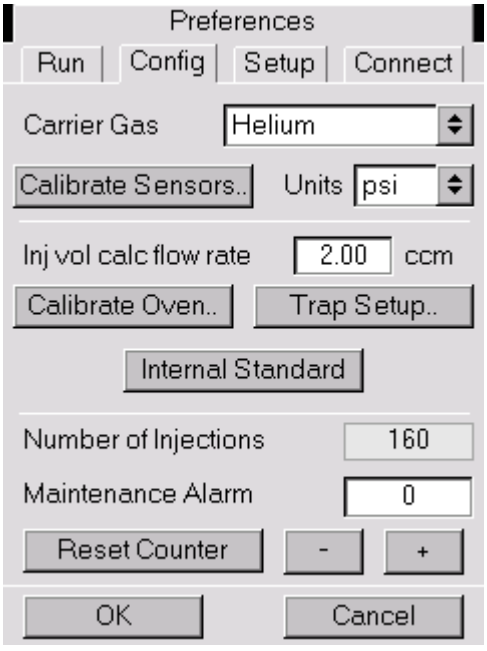


Figure 52 HS 40/110 Trap Status Tab Config Tab

Calibrate Oven — This button provides direct access to the oven calibration dialog.

Trap Setup — This button allows you to select the trap heating (slow or fast) rate and a value for the maximum temperature.

NOTE: The speed of Trap temperature rise and the maximum allowable Trap temperature can be selected and set on the Trap Setup.
***Tools*---><*Preferences*---><*Config* *tab*---><*Trap Setup*.**

You can choose either Fast or Slow Heat Rate from the drop down list.The default Trap Maximum Temperature is 400 ºC. It depends upon the Trap material. Unauthorized changes to higher values should be avoided.

CAUTION	*Follow the sorbant manufacturers recommendations for the upper trap temperature. If the temperature is set too high the trap and instrument could be severely damaged.*

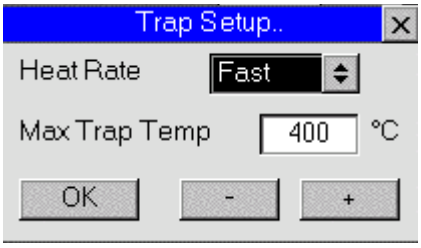


Figure 53

Internal Standard—This button allows you to input the loop load, loop equilibrium and the inject time.

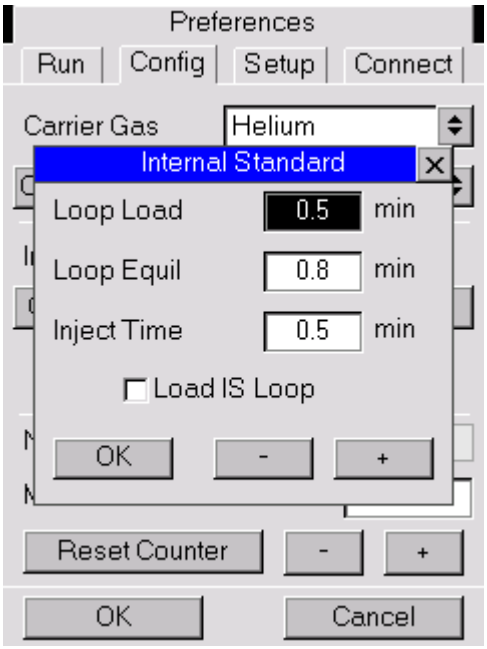


Figure 54

Loop Load—The time required to fill the sample loop.

Loop Equil—The time required for the sample loop to drop to a constant pressure.

Injection Time—The time required for the carrier gas to inject the standard into the vial.

Load IS Loop—This box is checked off only when you are first calculating the loop equilibrium time.

Setup Tab

The options on the Setup tab provide access to vial and system configuration.

Password Protection—You can protect the instrument from unauthorized access using the password protection. When enabled, users must enter a password to gain access to the interface.

To enable and use this option:

1. Open the Setup tab.
2. Press the **Password Protection** check box. A check mark will appear in the box to enable the option.
3. To create a password, touch the gray bar to the right of Password Protection. The password window will then appear.
4. Touch the new entry box and the Alpha/Numeric Entry window appears.
5. Enter the desired password, using letters (Alpha tab) and/or numbers (Num tab). The blue shift key changes letters to/from upper case. Touch **OK**.
6. Touch to confirm:entry box. Repeat the password entry. Touch **OK**.
7. Press **OK** to close the Preferences tab. This confirms your selection. If you press Cancel, then the settings on this tab revert to the default settings.

Priority Vials—This feature can be used only if the vial under counting has a magazine position of #10 or higher. This feature can also be used to insert vials, into an analysis sequence that has already been started. Once the vials have been placed in the priority vial position and the priority command is issued, the priority vials are inserted into the analysis sequence. The analysis of the priority vials does not affect any ongoing thermostatting of vials or analysis.

This parameter reserves sample magazine locations for a priority run. You can select #39 - #40, #40 or None. On the HS 110, you can select #109-110, #110 or None. The priority vial option is not available on the HS 16.

NOTE: If two priority vials are selected, the higher numbered position will be analyzed first.

Once you have enabled the priority vial locations, the locations are not analyzed unless you issue the Priority command. Touch the Priority Run bar at the bottom of the Status screen.If you issue the Priority run command, the current analysis will be completed and then the priority vials will be loaded and analyzed. Once the priority

vials have been completed, the HS returns to where it left off and continues analyzing the vials as specified on the Run tab.

After a method or sequence has been started, the Rotate button, located on the bottom of the display, changes to the Priority vial button. Press the Priority run button to initiate the analysis of the priority vials.

The priority vials are analyzed according to the current method.

The Priority feature only appears if priority vials have been selected from the Priority Vials menu. When you start a priority run, the priority vials are loaded into the oven carousel as soon as possible. The priority vials are analyzed using the current method. After starting and during a priority run the status Priority Run is displayed on the status bar.

NOTE: It is not possible to perform a priority Run in Progressive Mode.

Alarm Option—Use the vial alarm option to warn you when the current method or sequence is about to be completed. You enter the number of vials before the end, when the alarm will sound. If you enter “2”, when the second from last vial is loaded into thermostatting oven, an audible alarm will sound. Press the screen to acknowledge the alarm. Enter a value of 0 to disable the vial alarm option.

Vent Time (Headspace only)**—**The vial venting time is the length of time during which the vial is vented to atmosphere. If you enable this option, venting occurs immediately after the withdrawal time. The default venting time should be 0.3 minutes (18 seconds).

WARNING

If the compounds contained in the vials are hazardous, this option should be disabled and the vials should be vented into a fume hood or other well ventilated area to avoid exposure to hazardous vapors. Sample vials just unloaded from the thermostatted oven into the magazine can be very hot and may still be under pressure. Cool and vent the sample vials before you open or dispose of them.

Economy Mode—Economy mode allows the instrument to remain on while conserving power and carrier gas. To use economy mode you must enable the option and then enter the desired settings. When the instrument goes into economy all of the heaters are switched off, the needle purge valve (SV2) is closed, and the touch screen display dims.

Any key press interrupts the Economy Mode and the instrument returns to Standby. A visual note and an acoustic effect accompany the activation of economy mode.

To enable and use this option:

1. Open the Setup tab.
2. Press the **Economy Mode** check box. A check mark will appear in the box to enable the option.
3. Press the **Config** button to open the configuration dialog box.

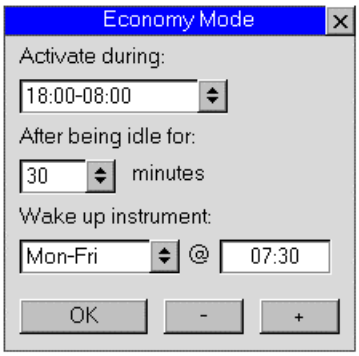


Figure 55 Economy Mode Dialog Box

4. The HS only goes into economy mode after a predetermined interval during which there is no activity. You can select this time. If you select 0, then the instrument will go into economy mode at the beginning of the selected interval, providing an analysis is not in progress. If you enter 30 minutes, then the instrument will go into economy mode, 30 minutes after all analyses have been completed.
5. Now enter the time for the HS to “wake up”. At the “wake up” time, the HS switches on the heaters and re-activates the purge valve. The instrument configures

itself according to the method specified on the Run tab.

6. Once you have entered the desired settings, press **OK** to accept the settings and close the tab.
7. Confirm that the date and time are correct. If necessary, enter the correct date and time.

Display Font-You can select from a number of different display fonts to customize your touch screen. To change the font:

1. Press the **Font** option and select the desired font from the drop down list.
2. Press **OK** to close the dialog box. You must log out and then open the interface again before the font change will take effect.

HS 40/110 Trap Setup Tab

Stop on Vial Error —This option will stop the run if there is a vial error such as a missing vial or there is something wrong with the vial.

Monitor Vial Integrity-This option will report on the Log tab any deviation of a vial pressure decay from the Decay Time set in the method (see **Calibrate Decay Time**).

If the **Monitor Vial Integrity Error** and the **Stop on Vial Error** are enabled in this tab, the system will terminate the counting if three consecutive vials fail the Vial Integrity test.

If the **Stop** on **Vial Error** is not enabled, the system will report the vial discrepancies on the Log tab, but will not stop the counting.

Connect Tab

Key Clicks—This option, if enabled sounds a tone each time a button is pressed. This feedback confirms that you have pressed a key.

Bar Code—The bar code reader is a future upgrade. Contact PerkinElmer for more information.

Baud Rate—Refer to the computer setup options to determine the baud rate, as this value will depend on the computer. The HS baud rate can be set from 300 to 57600 to match the computer's setting.

Handshake—Select the desired Handshake mode. Choose None, Xon/Xoff, or hardware. Normally, you will use None.

Language—You have a number of language options. You can select the language to be displayed on the touch screen display. Select the desired option from the drop-down menu. Press OK to confirm the selection and close the tab. The touch screen will now be displayed in the selected language.

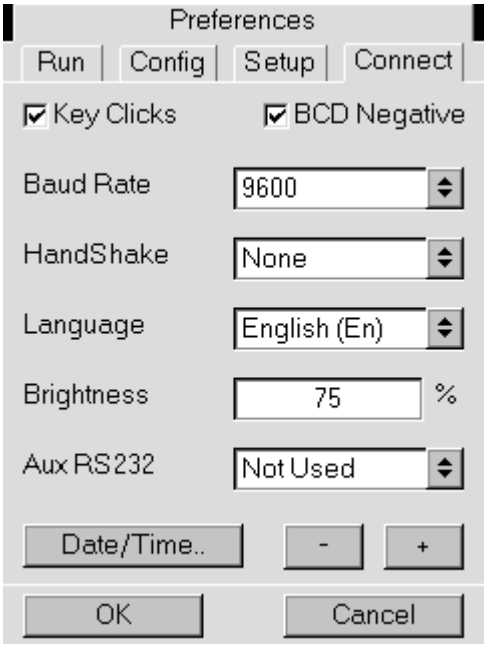


Figure 56 Connect Tab

Brightness—The brightness controls the touch screen display. Set the brightness so that the display is clearly visible under all lab conditions.

Auxiliary RS-232—This port is currently not available.

Date/Time—Press the date/time option to enter the correct date and time. The Time/Date dialog box opens as shown below. Select the desired date format and then enter the correct date. Next enter the desired time format and the correct time. Press OK to accept the new time.

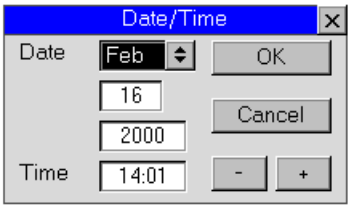


Figure 57 Date/Time Dialog Box

Starting a Run (TurboMatrix Headspace and HS 40/110 Trap)

Preparing Samples

In some methods pre-cleaning the vials (by washing with a detergent solution followed by rinsing with distilled water and drying in an oven) is recommended. Normally, the headspace vials are sufficiently clean and no pretreatment is necessary. In fact, such washing, particularly with a detergent, may add impurities to the vial!

The main source for blank peaks from an empty vial is not contamination on the vial's inner surface but bleeding of the septum and/or impurities in the air filling the vial. Such volatile contaminants can be eliminated by storing the empty vials in a clean room and/or by purging them with an inert, pure gas before the sample is added to the vial[1].

NOTE: If you are handling hazardous samples, you may need to perform the following steps in a fumehood.

To prepare sample vials:

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 48

1. Place the vial in a vial holder rack (N930-1304). The rack is recommended to keep vials upright during filling and capping.
2. Use a syringe or an appropriate pipet to fill the sample vial with liquid samples. Do not fill the vial over the maximum fill volume for liquid samples. Observe the maximum filling volume of 15 mL for liquid samples when using 22 mL sample vials. Ensure the sample does not come in contact with the top of the vial.
3. Load the cap. Place the star spring into the cap and then, using tweezers or forceps, place the seal on top of the spring. Push the seal into the cap so that it is flat. Place the cap on the vial.
4. Place the crimper over the cap. Place one hand over the stamper to stabilize the crimper on top of the vial. Use the other hand to squeeze the crimper. Keep the hand crimper level when sealing the cap. See the *Accessories* chapter *Sample Vial* section for further details.

The vials for HS-GC analysis are produced in large quantities, with a constant volume. The specified nominal volume is usually very close to the actual volume. In many methods the actual vial volume must be known. If the work at hand involves a new batch or a new supplier It is advisable to determine this value. This can be done by completely filling a number of vials with water, weighing them, and then using the mean value for future calculations.[1] For quantitative analysis identical sample volumes are required.

Loading the Magazine

The sample vial locations in the magazine are numbered 1 to 16, 1 to 40, or 1 to 110 depending on the instrument. See the *Accessories* chapter *Sample Vial* section for details of sealing sample vials.

All vial locations can be filled on the HS 16 magazine. The vial will be lifted from its location into the oven. When the analysis is completed the vial is lowered to its location.

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 46.

On instruments with the 15-vial oven, it is possible to turn the instrument off while vials are still in the oven. When the instrument is turned on again, it will attempt to unload the oven. You must provide an empty spot in the outside ring of the magazine so that the HS can unload the oven.

When you are loading the HS 40, you can place vials in all positions except for one. You must leave one spot open in order for the HS to unload any vials that may be present in the oven.

When you are loading the HS 110 you must leave the load and unload spots empty. You must also leave one further location on the outside ring empty for the HS to unload any vials that may be present in the oven. The HS will search the outer ring until it locates the empty position. To reduce the initialization time, leave location #1 empty. This is the first location that the instrument will look for the empty position.

Press the rotate button to move the magazine one quadrant at a time, to access vial locations under the loading port of the thermostatted oven.

If you are using the smaller 9 ml vials, you must use the low volume vial adapters (P/N N612-0110).

NOTE: You can only load these low-volume vials in the outer ring of the HS 110 magazine.

WARNING

Sample vials recently unloaded from the oven can be very hot. When automatic venting is switched off, the sample vials are still under pressure when unloaded from the oven. Ensure that sample vials are cool before venting them and disposing of the sample.

Single Method Operation on the HS

Once you have set up the HS and entered a method for your application you are ready to begin analyzing your samples.

To start a run:

1. Ensure the GC is ready.

2. Open the **Run** tab. If you have selected single method operation, instructions will be displayed on the tab.

3. If you are running a single method and the method has been pre-selected then you only need to enter the start and stop vials. Press the **Start vial entry** box and then press the plus or minus button to select the desired start vial.

4. Enter the desired end vial.

5. If you are running a single method, then select vials and enter the start and stop vial number. Choose the method to be used, from the drop-down box.

6. The Start button should be green. Press the **Start** button.

7. The button will change to a red Stop button.The Rotate button will also change to Priority.

The method can be stopped at any time by pressing the Stop button. A confirmation pop-up window will be displayed, press Yes to confirm that you want to stop the run. The analysis on the current vial is aborted and the vial will be unloaded and returned to the magazine. The method will be interrupted and the instrument will revert to Standby.

Multiple Method (Sequence) Operation

You can create a sequence of methods if you have varied applications. Multiple method operation must be enabled on the Preferences tab. You must also have created and tested all of the methods required for your sequence.

Your HS application may require the use of multiple method for analysis of multiple vials. It is also possible to run a sequence of methods on a single vial if required.

To start multiple method operation:

1. Select the **Sequence** option.

2. Press the **Start vial entry** box and then press the plus or

minus button to select the desired start vial.

3. Enter the desired end vial.

4. From the method drop-down box, select the method to be used for the selected range of vials.

5. Press the **Add** button to add the entry to the sequence.

6. Press the **Start vial entry** box again and enter the start/ end vials. Select the method to be used for the selected range of vials from the drop-down box and then press Add to add the entry to the sequence.

7. Repeat these steps until you have created the desired sequence. To delete an entry from the sequence, select the entry in the list and press the **Delete** button. To change the vial range, you must add a new entry with the revised vial range and then delete the old entry.

8. If you edit the methods called by your sequence the sequence will use the revised methods. If you need to edit a method but do not want to affect your sequence, then save the revised method with a different name.

NOTE: You will not be able to edit any of the selected methods if method editing has been disabled from the Preferences tab.

9. Press the Green **Start** button to run the sequence.

The HS will configure itself based on the first method. The instrument status will be Not Ready until the instrument reaches all of the set points. Once it has reached the set points, the vial will be loaded and the analyses will continue as determined by the method. If you have the HS 40 or HS 110 with the 15-vial oven, vials will be loaded at regular intervals as determined by the PII time.

Using the Tray Rotation Feature While Running A Vial Sequence

To optimize the TurboMatrix throughput cycle and avoid analyzing empty vial positions, Firmware version 2.14.83 for the HS-40 and

HS-110 includes a Tray Rotation countdown dialog. The main objective of this dialog is to rotate the autosampler tray enough to make the vial positions under the oven accessible, alert users when the instrument will be ready to load the next vial, and rotate the autosampler tray back to its position so that the TurboMatrix can continue with the analysis.

To access this functionality while a sample sequence is being analyzed, the Rotate button becomes available after the run has started. The button label changes to Load once a run is started (see figure on left). (The Load button will only be enabled if the Priority Vials feature is set to None under Preferences.) Pressing Load will activate the Tray Rotation countdown dialog (see figure on right) and rotate the tray to enable access the next available vial locations.

Each time the Rotate button in the Tray Rotation countdown dialog is pressed, the tray continues to rotate (approximately 90 degrees) to provide access to additional vials. The Tray Rotation countdown dialog will be open and active during the countdown time displayed at the top of the window. This countdown timer expires approximately 10 seconds before the next autosampler action is due to activate (e.g., load another vial, rotate sample tray, etc.).

Control/Function	Description
Last vial loaded	Displays the last vial loaded.
Available positions*	This feature informs you as to which positions are available for loading new samples. Some of the positions may appear to be empty because the samples are loaded into the Headspace. This will help the user avoid the 'in use' position. The vials displayed will be from the next vial position to the upper range of a program step. i.e. program step 1 to 10 next vial to be analyzed is 6 'load in available positions from 6 to 10'
OK	The tray also rotates back when you press on the **OK** button. However when **Available positions** parameter has a '-' indicating that no vials remain scheduled for sampling, pressing **OK** will not rotate the tray back to its original position. **Note:** To stop the entire sequence, you must close the modal Countdown dialog first before the **Stop** button can be enabled.
Rotate	The tray rotates 90° counterclockwise each time it is pressed.

*NOTE: When the Available positions parameter has a '-' indicating that no vials remain scheduled for sampling, pressing **OK** will not rotate the tray back to its original position.*

Creating a Sequence

You can use a sequence of methods to analyze a series of vials. The options on the Run tab allow you to select a range of vials and then specify a method to analyze the samples. You then select the next

range of vials and select another method. You can also analyze the same range of vials using different methods. The sequence can be used on a daily basis or it can be edited as required.

You will not be able to edit any of these methods if method editing has been disabled from the Preferences tab.

To create a new sequence:

1. Select the **Run** tab.
2. Press the **Start vial entry** box and then press the plus or minus button to select the desired start vial. Next, enter the desired end vial.
3. From the method drop-down box, select the method to be used for the selected range of vials.
4. Press the **Add** button to add the entry to the sequence.
5. Repeat these steps until you have created the desired sequence. To delete an entry from the sequence, select the entry in the list and press the **Delete** button. To change the vial range, you must add a new entry with the revised vial range and then delete the old entry.
6. If you edit the methods called by your sequence the sequence will use the revised methods. If you need to edit a method but do not want to affect your sequence, then save the revised method with a different name.

You use up to 8 methods in your sequence. If you need to use more than 8 methods, you must use the PC control software.

NOTE: You will not be able to edit any of the selected methods if method editing has been disabled from the Tools button.

7. Press the green **Start** button to run the sequence.

The HS will configure itself based on the method parameters. The instrument status will be Not Ready until the instrument reaches all of the set points, at which time it will become Ready. When the GC and the data handling system are ready, a vial will be loaded into the oven. The vial will be thermostatted and the analyses will continue as determined by the method.

If you are using the an HS 40 or HS 110 with the 15-vial oven, the first vial will be loaded, and then vials are loaded as required based on the time established by overlapping thermostatting.

Editing a Sequence

To delete an entry from the sequence, select the entry in the list and press the Delete button. To change the vial range, you must add a new entry with the revised vial range and then delete the old entry.

If you edit the methods called by your sequence the sequence will use the revised methods. If you need to edit a method but do not want to affect your sequence, then save the revised method with a different name.

Logic Flow Diagram - A Description (HS 40/110 Trap Only)

NOTE: The Trap is added to the HS in order to offer a pre-concentration of almost all the HS vapor produced in the sample vials by the preceding thermostatting.

Figure 58 is a logic flow diagram that shows the steps in sample preparation up to the Desorption/Injection-analysis carried out by a Headspace autosampler equipped with a Trap.

When an HS method is started, the first sample vial is loaded into the thermostatting oven (15-position oven, up to 12 vials can be loaded simultaneously).

1. Vial thermostatting: The system starts with the vial thermostatting at a temperature and for a time known to give the largest vapor volume of the compounds of interest.

2. Vial Pressurization: Next, the needle is lowered into the headspace of the vial and the vial is pressurized with carrier gas at a pressure and for a time set in the method (Vial Pressure, Pressurization Time).

3. Pressure Decay: With the needle still in the vial, the

carrier flow to the vial is interrupted.
Lack of external pressure, forces the already pressurized headspace vapor to escape on a reverse flow, through the needle then the Trap (back to front), and vent through the Purge outlet.
With the Trap cold (default 40 °C), the compounds in the vapor are adsorbed in the Trap.
The system will repeat steps 2 and 3 (Pressurization-Pressure Decay) for the number of cycles (maximum 4 cycles) set in the method. The correct number of cycles is the minimum number which results in the removal of practically all of the analyte vapors. This is determined by successively analyzing identical vials with the same method, altering only the number of Pressurize/Decay cycles. The smallest cycle number resulting in the highest vapor transfer (indicated by highest peak area) is the most suitable.

4. Needle Withdrawal: After the last cycle, the needle is withdrawn from the vial and this one is returned to the magazine.

5. Dry Purging the Trap: Next, is the optional step of the Trap Dry Purging if removal of moisture or/and certain volatiles is desired.
   The system applies a forward carrier gas flow through the cold trap at low trap temperature set in method, from front to back and out of the Trap Dry Purge outlet (˜50 ml/min).
   The analytes in the sample headspace vapor are now adsorbed, thus pre-concentrated in the dried Trap. The Trap is ready for the analytes desorption.
   After the Dry Purge step, the system interrupts the Isolation Flow to transfer line and column. The PPC pressure will then take over.

6. The system will then proceed to the **Desorption** step only if the GC is ready. If not, it will wait until the GC becomes **Ready**.

7. Desorption: With the GC ready, the system will proceed to desorption. It will rapidly heat the trap to the **High Temperature** set in the method. Simultaneously, it will

apply a low pressure for a short time. Both parameters are set in the method (**Desorb Pressure** and **Desorb Time**). This desorb pressure will create a reverse carrier gas flow through the trap (from back to front) via an appropriate switching of the solenoid valves SV1 and SV9.
GC analysis program starts at this step.
After the initial short Desorb time, the system switches the PPC module to the appropriate column pressure that sweeps the desorbed analytes out of the trap and into the transfer line and the column for the GC analysis. The column pressure value is set in the HS Trap method.

8. Trap Hold: This keeps the trap at the desorption temperature for the **Trap Hold** time that we have set in the method. At the end of this time, the system interrupts the heating of the trap and starts cooling it with a fan, back to the low trap temperature set in the method (default 40 °C).

9. Simultaneous with the Trap Desorption, the system checks whether this is the last vial in the method. If yes, it terminates the HS Trap operation and goes to standby. If not, it moves to the next vial which it loads into the thermostatting oven to start its preparation.

10. The term **PII** Algorithm (**P**eriod from **I**njection to **I**njection algorithm) that appears in the flow diagram, denotes a software that enables the HS to calculate the period from injection to injection from the timing values that are entered in the method and the GC cycle time. The PII value is shown on the Timing tab of the HS screen.
    For optimum sample throughput (i.e. the greatest number of samples analyzed in a given time), it is essential that the PII value is only slightly longer than the GC cycle time. The PII value shows the effects of the timing changes in the analysis on the vial throughput.
    In a 15-vial oven, it is possible to overlap the thermostatting time of the vials and reduce the PII.

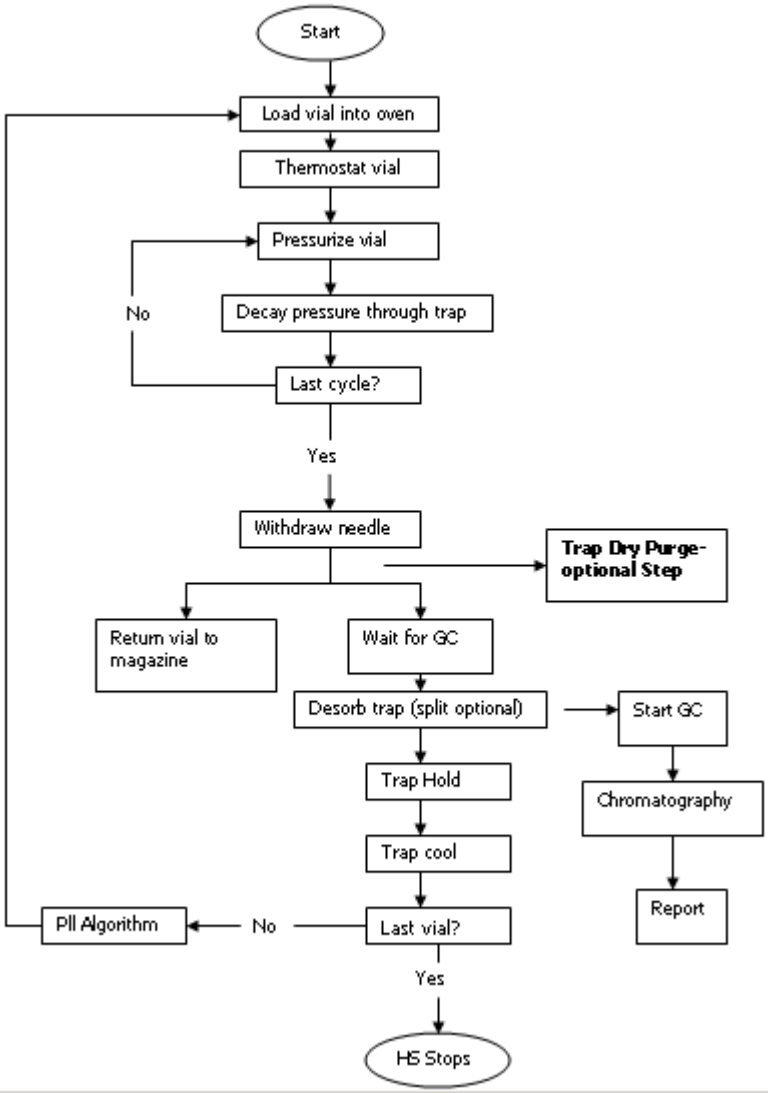


Figure 58 HS Trap Logic flow Diagram

High Pressure Sampling (Headspace Only)

The high pressure sampling option is automatically available if you have the PPC option installed.

The high pressure sampling technique is used to prevent pre-injection. Pre-injection occurs when the internal pressure of the sample vial is greater than the sampling head pressure. In such cases the sample expands onto the transfer capillary as soon as the sampling needle moves down into the vial. This causes split peaks and double peaking on the chromatogram. High pressure sampling permits sampling in such cases, without these secondary effects.

A higher pressure (P2) is applied during pressurization of the vial and injection of the sample. After injection of the sample (i.e. immediately after withdrawal time and vent time), and when the needle is in the standby position, the pressure returns to P1.

High pressure sampling is necessary if the vial pressure exceeds the column head pressure. The standard balanced pressure sampling technique requires the column head pressure to be higher than the internal vapor pressure in the thermostatted sample vial.

When using wide-bore capillary columns (0.53 mm internal diameter), or high thermostatting temperatures, it is possible that the internal pressure in the vial, generated by the partial vapor pressures of the sample components, can exceed the column head pressure. In this case, the differential pressure between column head pressure and vial pressure becomes negative.

The high pressure sampling accessory can be used with packed columns and open tubular capillary columns. It can be operated independently or in combination with other installed pneumatic accessories such as the cryofocusing accessory.

HS 40/110 Trap (Isolation Flow-Pre-Injection Peaks)

For the HS 40/110 trap the isolation flow should be carefully adjusted to 5 to 10 ml above the analytical column flow in order to prevent Pre-Injection peaks.

For details, see the paragraph **Loss of Sensitivity-Pre-Injection Peaks** in the *Troubleshooting* chapter.

Shutdown (Headspace and HS 40/110 Trap)

Normally you do not need to shut down the instrument. You can put the instrument into economy mode overnight and over the weekend. See *Setup Tab* on page 141. If the instrument will not be used for more than two days, you may opt to shut it down.

If the instrument is to be disconnected from the GC be sure to cap the end the fused silica transfer line to prevent contamination of the system.

To shut down the automatic headspace sampler:

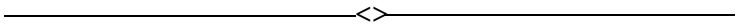
1. Stop the running Headspace Method by pressing the **Stop** button.
2. Wait until all the sample vials have been unloaded from the thermostatted oven.
3. Unload the magazine. Press the **Rotate** button to access vials that are under the oven
4. Press the power switch to turn the HS off.

*NOTE: Before proceeding to next step, ensure that GC and injector/detector conditions meet those in the **Carrier Gas Shut Off** paragraph in the **Routine Maintenance** chapter.*

5. Turn off the carrier gas supply.
6. Turn off any related accessories and close the gas supplies.

With the HS 40/110 trap the column isolation mode will stay on and maintain column flow even when the trap instrument is powered off. You can power down the HS 40/110 trap and still have flow going through the column. If you are using an MS you can leave the MS pumped down.

Accessories *4*



Options Board

An options board is available for the instruments. This board provides: Four programmable, time-dependent switch relays, the BCD interface, one RS-232C port and support for the shaker and HS 110 automation module. The HS 110 utilizes the options board for control of the crane and robotic arm that are used to load the vials into the oven.

The BCD option and the shaker both require the option board to be installed and enabled.

NOTE: The shaker is not available for the HS-16.

Timed Events

The optional timed event connector has terminals for 6 programmable, relays. External devices may be timed by these relays.

CAUTION	*Input voltage must not exceed 50 V (DC). Input current must not exceed 0.5 A (DC).*

You may program the events using the event tab.

To program events:

1. Open the Status tab or the Method Editor tab and select the **Timing** tab.
2. Press the **Relay** button to open the Event Relays tab.

NOTE: If the Relay button is not displayed on the Timing tab, it must be enabled from the Preferences Run tab. See Run Tab on page 137.

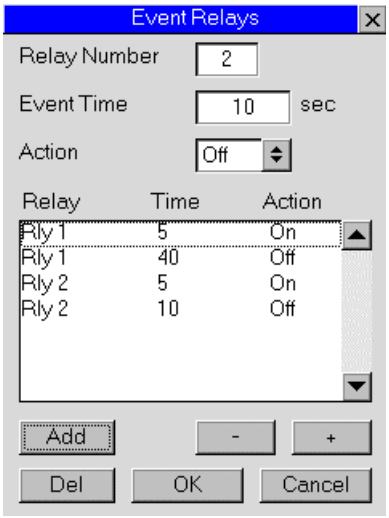


Figure 59 Event Relays Dialog Box

3. Enter the relay number that you want to activate or deactivate.
4. Enter the Event time and the required action. Turning a relay on and off requires two events.
5. Press the **Add** button to add the event to the listing. To delete an event from the list, select the event and then press the **Delete** button.
6. Repeat this step for each event that you require. You can enter up to 6 events.
7. Press **OK** to accept all of the events and close the window.
8. If you are using the Method Editor, save your method. If you are using the Status tab, you must enter all the method parameters and save the new method.
9. Run an analysis to test that your devices are connected properly and your events are programmed correctly.

Example: To turn relay 1 on for 1 minute, 30 seconds after the current analysis has started, you would enter: an event time of 30 sec, Relay Number 1 and Action would be On. You would now enter a second event: an event time of 90 sec, Relay Number 1 and Action would be Off.

The following timing diagram shows various devices being activated, during each analysis. A device can also be activated for more than one analysis as shown by relay #3.

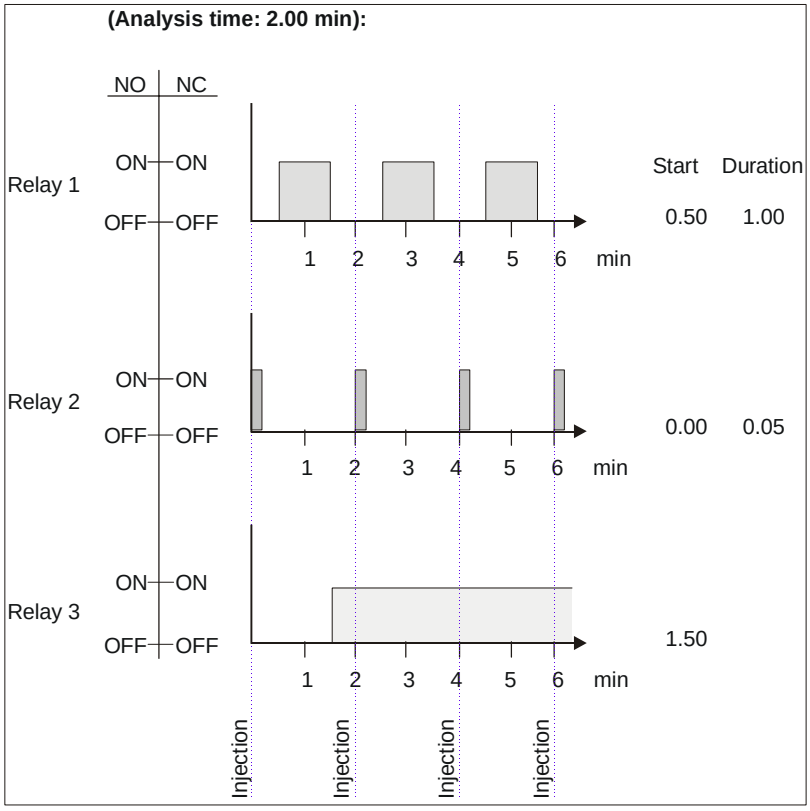


Figure 60 Timed Event Diagram

NOTE: Timed events are not included in the PII calculation.

BCD Interface

The Binary Coded Decimals (BCD) interface is supplied on the options board. The BCD interface is used to transfer the sample vial numbers to an external instrument such as an integrator.

The BCD interface has two configuration parameters: BCD Data Logic and BCD Data Format.

BCD Data Logic—The BCD interface data channel contacts are set as follows:

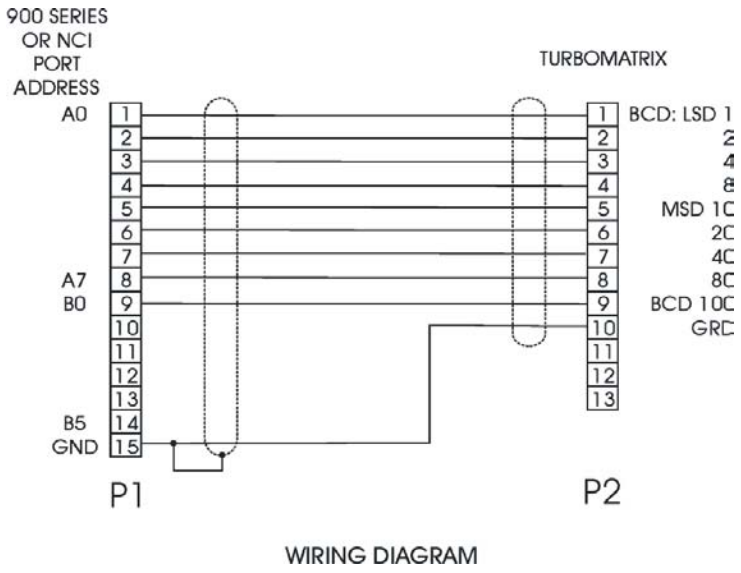


Figure 61BCD Interface Data Channel Contacts

The BCD data channels (contacts 1 to 13) are set to negative logic. Negative is the default.

Negative is the default. If you wish to change from negative to positive logic in the software under Tools select **Preference** in this screen the “connect the logic can be changed to positive.

The Vial Shaker Accessory

The frequency scanning shaker accessory may be installed on the HS 40 or HS 110. The options board is required for the shaker option. If you have an HS 40, the options board must be installed. The options board is standard on the HS 110.

The shaker can decrease the time needed for equilibration by providing continuous mixing of the sample in the vial during the equilibration process. When applying the shaker, it is important that

the sample be in resonance with the shaker frequency to obtain the desired mechanical mixing effect. In the case of liquid samples, the resonance frequency depends on the sample viscosity and its volume. To overcome this problem, the frequency scanning shaker varies its frequency automatically during equilibration, through a broad frequency range, so that each sample in the oven reaches its resonance frequency.

The shaker option is installed in the factory. If you order the accessory after your instrument has been delivered, a service engineer must install the option. Please contact PerkinElmer for further information.

PPC

Programmed Pneumatic Control (PPC) is the electronic control of pressure for sampling and the GC column. The PPC control module regulates pressure using an electronically driven variable flow restrictor. The control module also contains pressure transducers to provide feedback for complete monitoring.

The PPC system is composed of one valve. The carrier valve is used to maintain the pressure of the gas through the system

The module will be calibrated when the instrument is installed and does not need to be calibrated again.

See *PPC Tab* on page 216 for details about setting the sampling pressure.

The PPC option is installed in the factory. If you order the accessory after your instrument has been delivered, a service engineer must install the option. Please contact PerkinElmer for further information.

Composite Zero-Dilution Split Injector Liner

Description

The zero-dilution split injector liner is composed of an inner hourglass insert and an outer liner. There is a sufficient gap between the outer wall of the insert and the inner wall of the outer liner to allow gas flow between the two. The insert is flared at the top to help with the insertion of the fused silica transfer line from the HS. The bottom of the insert is beveled to ensure that the insert does not seal against the outer liner thus ensuring gas flow between the two.

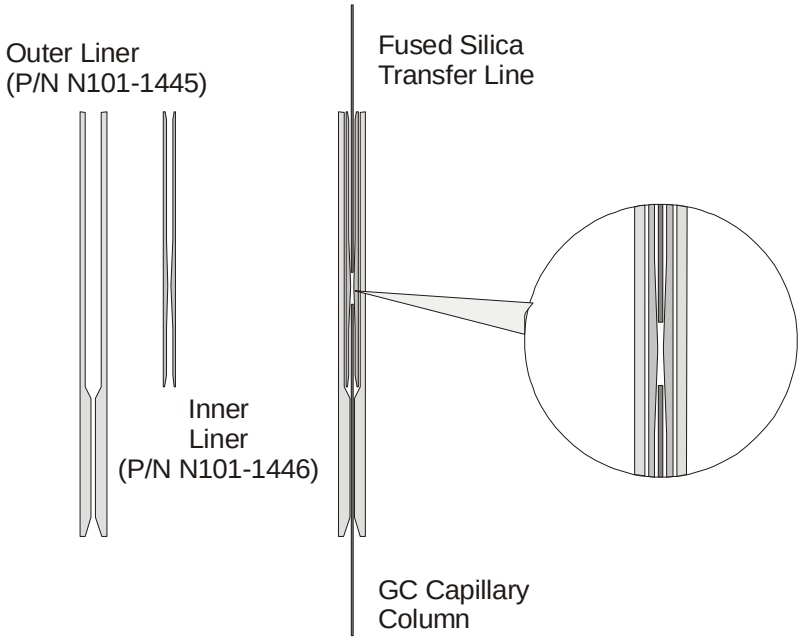


Figure 62 Zero-Dilution Split Injector Liner

The assembled liner is installed inside the injector in the same manner as a normal injector liner. The fused silica transfer line is installed at the top of the injector and the chromatographic column is introduced through the bottom of the injector.

The GC column is pushed up through the outer liner and insert until it rests against the hourglass constriction. The column is further inserted and lifts the hourglass insert up until the constriction stops as it contacts the end of the transfer line. The GC column is then withdrawn about 2mm to leave that distance between the end of the transfer line. The insert is held and is loosely sealed on the end of the column by gravity. The end of the column is then secured and sealed at that position by a nut and ferrule.

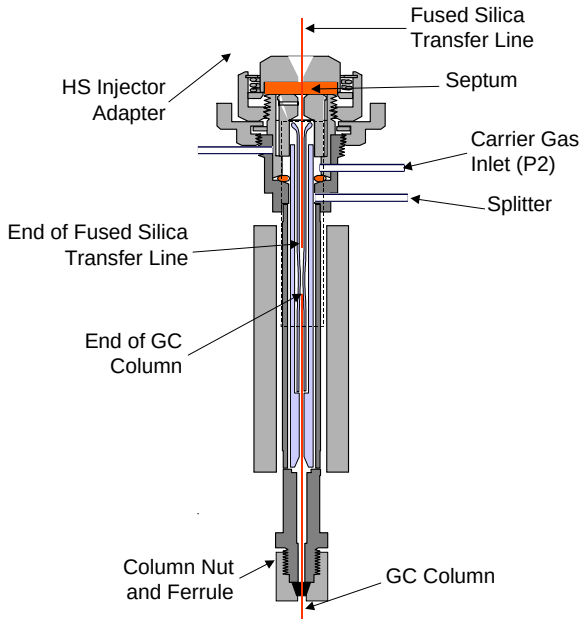


Figure 63 Cross Section of the Zero-Dilution Split Injector Liner Installed in the GC Injector

During sampling, the vial is first pressurized (P1) with carrier gas. P1 must be higher than the column head pressure at the GC injector (P2). Carrier is gas supplied to the injector, via the normal carrier gas supply P2. Excess carrier gas is vented through the injector splitter at a rate of 15 ml/min or more.

During the sample injection, the sample will elute from the transfer line, into the hourglass and immediately enter the chromatographic column. Excess sample eluting from the transfer line will exit

through the gap between the end of the transfer line and the constriction in the hourglass and will mix with carrier gas flowing though the injector outer-liner and out through the split vent.

The excess sample flowing out of the hourglass acts as a barrier to the carrier gas flowing through the outer-liner and prevents it from reaching the inlet to the chromatographic column and so no dilution or dispersion of the sample takes place.

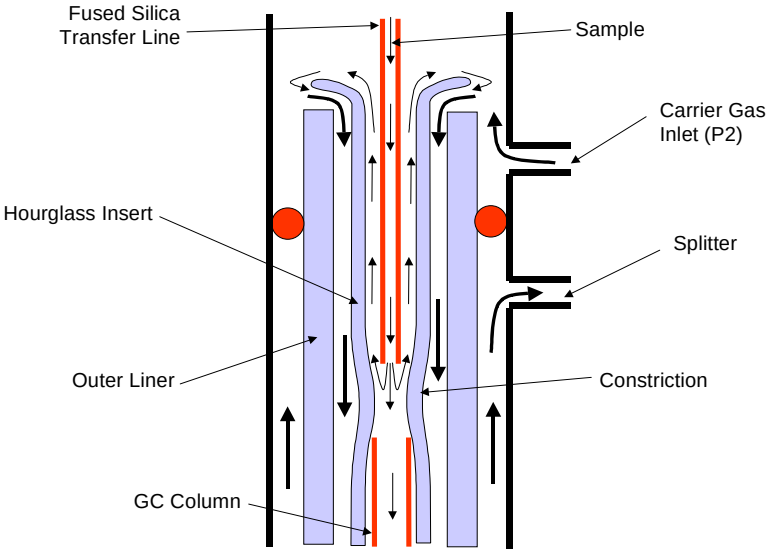


Figure 64 Sample Flow Through the Zero Dilution Liner

Installing the Zero Dilution Liner

To install the zero dilution liner in the GC injector:

1. Turn off the GC and the HS. Allow the GC oven and injector to cool.
2. Remove the column and existing injector liner. Refer to the GC user's manual for instructions.
3. Using gloves, check the outer and inner liner components of the new composite liner for contamination or damage.

NOTE: Do not touch any parts of the liner with your fingers, as you will contaminate the injector.

4. Insert the inner liner into the outer liner.
5. Install the composite liner into the injector using a standard o-ring. The outer liner should be gently inserted into the injector.
6. Replace the injector head and secure with a wrench.
7. Thread a length of 0.32 mm i.d. deactivated fused silica tubing through the transfer line and attach it to the HS sampling head as outlined in *Installing the Heated Transfer Line at the HS Needle Unit (TurboMatrix Headspace Only)* on page 62.
8. The other end of the tubing should be threaded through the septum in the HS septum nut. Score and cut the fused silica so that 62 mm is left protruding from the septum (57 mm from the edge of the septum nut).

NOTE: This length can be shorter (down to 20 mm) if 0.53 mm i.d. columns are being used.

9. Insert the fused silica tubing into the injector.
10. Insert the column into the base of the injector and push it up through the liner until it just reaches a stop (about 8 cm). Secure the HS injector adapter

NOTE: Do not push the column too hard.

11. At this point, the fused silica transfer line and the column should be at either side of the restriction in the inner liner. Withdraw the column about 2 mm and tighten the column ferrule.
12. Establish a flow of carrier gas and leak test your GC connections.

Operation of the Zero Dilution Liner

The liner operates well as the pressure drops across the transfer line. A pressure at 0.5 psig or above 2.5 psig is recommended (~20ml/min).

The GC split flow should be set to 10 ml/min or more. Higher flow rates will just waste carrier gas.

Cryofocusing Accessory

The cryofocusing accessory is available for the HS 110 and HS 40.

NOTE: The cryofocusing accessory is not available on the HS-16.

The HS component of this option is installed in the factory. The GC component must be installed on-site. Refer to the cryofocusing manual for details of installation. If you order the accessory after your instrument has been delivered, a service engineer must install the option. Please contact PerkinElmer for further information.

Cryofocusing (or cold trapping) is the technique of sample concentration through the application of low temperature. The sample is cooled at the GC column inlet and the volatile components in the headspace are separated from the rest of the unretained gases and hence concentrated.

The volume of the headspace gas sample can be increased and so the detection limit can be lowered by a factor of up to 50. This is advantageous when you are using capillary columns, which can normally only handle small sample volumes in the range of 100-300 uL.

Cooling increases the partition coefficient of the volatile components in the stationary phase; they thus pass through the column more slowly than the carrier gas while the air contained in the sample continues to flow at the same speed as the carrier gas. In this way, the components of interest are separated from the original large volume of air contained in the original sample and pre-concentrated at the head of the column.

Principle of Operation

In the cryofocusing technique, a portion of the quartz capillary column is fed through a PTFE tube through which a coolant gas is passed in the opposite direction to the carrier gas in the column. The coolant gas is cooled by passing it through a copper coil immersed in a Dewar vessel containing liquid nitrogen. The gas flow is metered by a valve connected to the HS. For efficient cooling during cryofocusing the temperature of the GC oven should be as low as possible.

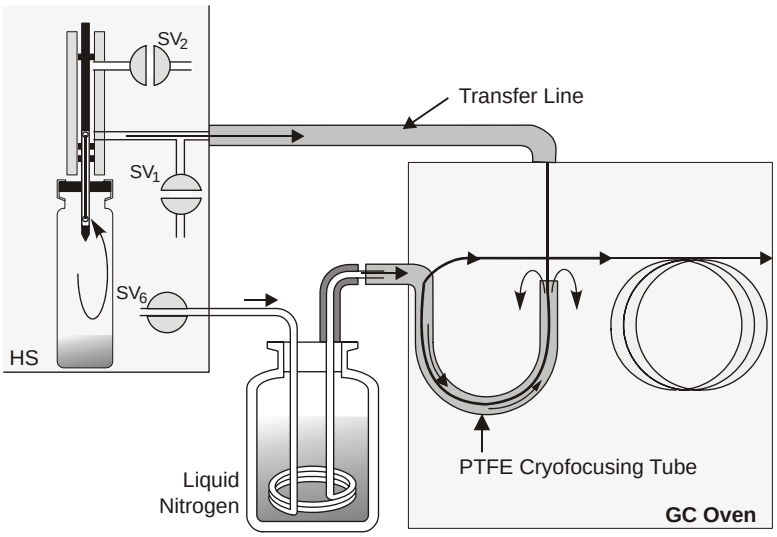


Figure 65 Cryofocusing Accessory

The pre-cryofocusing duration allows the head of the GC column to cool to the set cryofocusing temperature. The post-cryofocusing time maintains the head of the column at the low temp until the sample has been collected and unretained compounds have cleared the column. These values are determined when optimizing the cryofocusing. The total cryofocusing time comprises the pre- (before sample injection), focusing time (during injection) and post- (after injection) cryofocusing times.

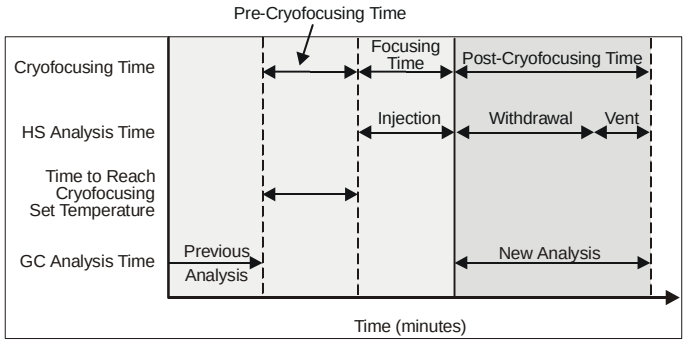


Figure 66 Setting the Pre and Post Cryofocusing Time

Cryofocusing with the Water Adsorption Trap

In cryofocusing using the water adsorption trap, water is removed from the sample prior to enrichment. This is done by adsorption onto a hygroscopic salt. The schematic below illustrates how this functions during the injection phase.

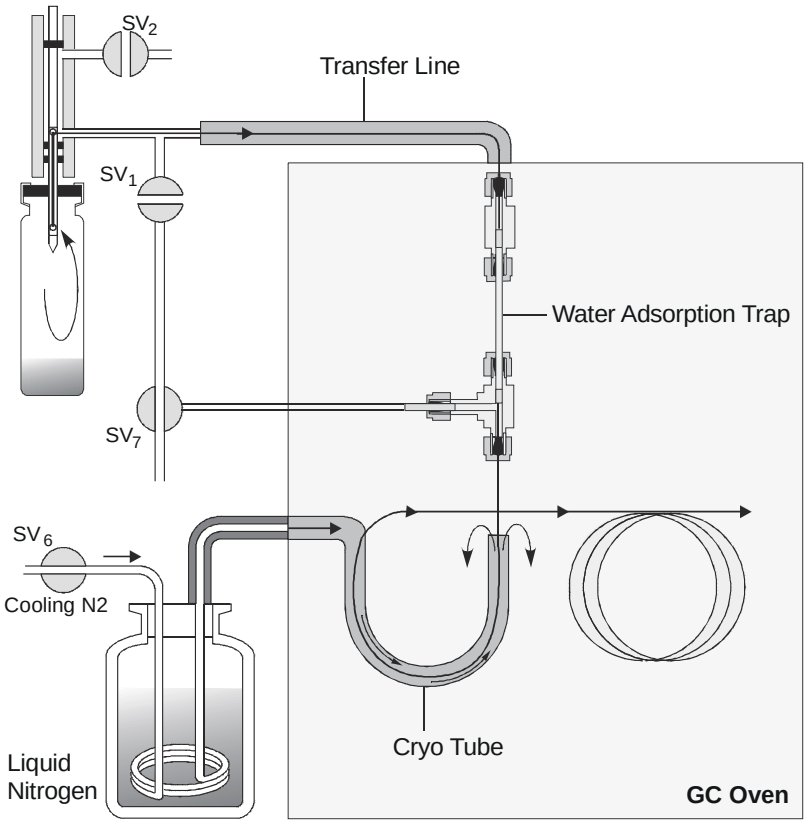


Figure 67 Cryofocusing with the Water Adsorption Trap

Gas flows through SV7 in the direction of SV1 when the needle is in the vial. Gas flows through SV7 in direction of the T-union and adsorption trap when the needle is out of the vial.

The water trap is backflushed automatically at the end of the withdrawal and vent time and it continues until the next vial is pressurized.

When you enable the water trap on the Options tab, the HS switches SV7 on and off, thus controlling the backflush flow through the water trap. The water trap can be regenerated by maintaining the GC oven temperature at 120°C for several minutes before beginning the next analysis.

Sample Vials

PerkinElmer 22 mL crimped top sample vials, P/N N9306079 (100 vials) or P/N B0104236 (1000 vials) and PerkinElmer 22 mL screw top sample vials, P/N N9306075 (100 vials) or P/N N9306078 (1000 vials) are carefully selected for HS operation. They are maintained under stringent quality control standards.

NOTE: Using any other vials from a different manufacturer is not covered under the warranty.

Please observe the following information:

- It is possible that a few vials in a batch are not within tolerance. If in doubt, we recommend that you check the sample vials using the vial gauge (P/N B0151737) provided with the instrument. Do **not** use unsuitable vials.
- Check the safety closure for reliable tightness after sealing the sample vial.
- Use only felt tip pens to mark sample vials. Adhesive labels may jam in the oven.
- Sample vials recently unloaded from the thermostatted oven into the magazine can be very hot and may still be under pressure. Cool and vent the sample vials before you open or dispose of them.

Using the Vial Gauge to Check Sample Vials

In order to ensure rapid and reproducible thermostatting the vials fit exactly in the oven. As vials are heated, in the oven, the glass expands slightly. If you are not using suitable vials it is possible that

vials will stick in the oven. With the vial gauge (P/N B0151737) you can check the sample vials for:

- Outside diameter and circularity of the vial body and neck;
- Concentricity of the vial neck with respect to the vial body;
- Height of the vial.

If in doubt, check the vials as shown below and discard any unsuitable vials. Also check the vials visually for any damage.

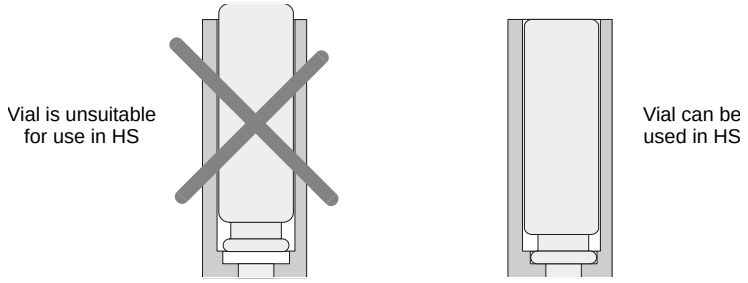


Figure 68 Using the Vial Gauge

Crimped Top Sample Vials

The patented safety closures (star springs) incorporate pressure relief features which guarantee safe operation with the high pressure that may develop during thermostatting. If a vial should burst, you risk injury from glass splinters and possible damage to the instrument.

NOTE: Such damage is not covered under the warranty.

Use only the cap removal tool (P/N N9301270) to open the sample vials. See *Decapping the Vials* on page 274. Carefully check the sample vials after cleaning for hairline cracks and damage before reuse. Do not use unsuitable vials. Replace reused sample vials regularly. PerkinElmer vials are guaranteed only for single use.

Hand Crimper for Crimped Top Vials

The hand crimper (P/N N9302785) is required for sealing the vials. You cannot seal the vials correctly without the crimper. A bench top (P/N N6621006) and electronic crimpers (P/N N9302595) are also available. Refer to the PerkinElmer catalogue for more information.

Sealing the Hand Crimped Vials

NOTE: If you are handling hazardous samples, you may need to perform the following steps in a fume hood.

To seal sample vials:

1. Place the vial in a vial holder rack (N930-1304). The rack is recommended in order to keep vials upright during filling and capping.
2. Use a syringe to fill the sample vial with liquid samples. Do not fill the vial over the maximum fill volume for liquid samples. **Observe the maximum filling volume of 15 mL for liquid samples when using 20 mL sample vials. Ensure the sample does not come in contact with the top of the vial**.
3. Load the cap. Place the star spring into the cap and then, using tweezers or forceps, place the seal on top of the spring. Push the seal into the cap so that it is flat. Place the cap on the vial.

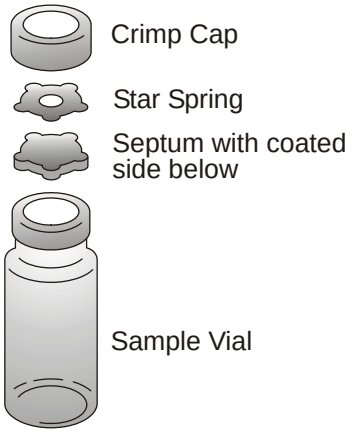


Figure 69 Safety Closure Assembly

4. Place the hand crimper (P/N N9302785) over the cap. When using the hand crimper. Place one hand over the stamper to stabilize the crimper on top of the vial. Use the other hand to squeeze the crimper. Keep the hand crimper level when sealing the cap.
5. Release the crimper and check the seal. The cap and septum must not be kinked or damaged. The safety

closure must be firmly crimped to the neck of the vial so that it cannot rotate freely, but it should not be deformed in any way.

6. If the vial is not sealed properly, remove the cap, using the cap removal tool (P/N N9301270) or the electronic hand decapper (P/N N9302595) and reseal the vial. If the vial still cannot be sealed properly, you may need to adjust the hand crimper. See *Adjusting the Hand Crimper* on page 271 for more information.

Screw Top Sample Vials

If not properly secured and sealed, the screw cap vials may leak. In order to prevent this from occurring, please insure that the septa is seated securely in the cap prior to tightening. In addition, the vial cap should be securely screwed down, with the cap and vial threads properly aligned.

Accessories (Screw and Crimp Cap Vials)

Unsafe high pressure formed in the vial during thermostatting may be due to the use of a solvent with a boiling point that is too low for the application. Operator error or inexperience may also contribute to unsafe thermostatting conditions. For example, if an aqueous sample is thermostatted to 80°C, the internal water vapor pressure is only 47 kPa. If, however, the sample is thermostatted at 180°C, the internal pressure would be as high as 10^3 kPa, with a high risk of vial explosion.

The internal pressure in the vial presses the septum disk against a star spring (crimp top only) and the aluminum cap, which has a small, curved slot. At pressures above 500 kPa, this slot is deformed and an artificial leak opens, venting the internal pressure to atmosphere. This safety closure requires flat septum disks and will not work with rubber stoppers. [1]

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997 Perkinelmer P/N N1011210), p. 51

The most common source of sample loss or degradation is poorly sealed vials. If your application requires sealing more than 10 vials on a daily basis you may want to consider the bench top or electric vial crimper to ensure your vials are sealed correctly.

Seals

The seals are manufactured to high standards and made of carefully chosen materials. Because of this, the amount of residual volatile that may evaporate at higher sampling temperatures is minimized. Ensure that the seals do not become contaminated once the bag is opened. Your lab atmosphere may be loaded with many volatiles that can contaminate the seals and vials. Don't touch the face of the seal that will contact the sample. It is good practice to handle the seals with tweezers or forceps only. Do not use your fingers.

For trace analysis best results will be obtained when the seals are preheated for up to 12 hours at sampling temperature. In this case, you must ensure that the seals don't touch each other and are well ventilated in a clean atmosphere. Using a nitrogen atmosphere will decrease the possibility of contamination.

Seals are available in four types. Your choice of seal will depend upon your application. See *Sample Vials and Seals* on page 337 for part numbers.

*NOTE: Do **not** exceed the maximum septa temperature when thermostatting samples.*

*NOTE: The sample needle temperature can **not** exceed the maximum temperature of the septa.*

Butyl Rubber Septa—for temperatures up to a maximum of 100 ºC, very weak interference peaks with FID, low permeability.

PTFE Coated Butyl Rubber Septa—for temperatures up to a maximum of 100 ºC, very weak interference peaks with FID, lowest permeability.

Aluminum Coated Silicone Septa—for temperatures up to a maximum of 120 ºC, very weak interference peaks with FID, very low permeability.

*NOTE: Do **not** use the aluminum coated silicone septa for needle temperatures exceeding 120ºC.*

PTFE Coated Silicone Septa—for temperatures up to a maximum of 210 ºC weak interference peaks with FID, very low permeability.

Headspace Control Software

The Control Software is designed to run under Microsoft Windows. The software provides the means to operate the instrument though a PC. You can also create methods, and sequences and log information about the status of the HS.

The Control Software runs in combination with TotalChrom. It is easy to install and use and allows you to:

- Control your HS
- Create and test new headspace methods
- Create sequences based on stored methods
- View graphical status and instrument control
- Log events that may affect analysis results
- Create printed method or sequence reports

A computer is not required to run the HS. If however you have a large number of varied applications then the PC control software is recommended. If you do opt to use the HS Control Software, your computer must be capable of running the PC Software and TotalChrom. If a TurboMass detector is also a component of the system, then the computer that is supplied with the mass spectrometer can be used to run HS control.

You require the following hardware and software to connect your computer directly to the HS and run the control software:

NOTE: Additional hardware may be required if you are running your data handling system from the same computer. Refer to the documentation supplied with your data handling system for details.

The minimum hardware configuration requirements are:

Windows 2000/2003 Servers:

- Processor Intel Pentium, 933 MHz
- Memory 256 MB RAM

Windows 2000 Clients

- Processor Intel Pentium, 933 MHz
- Memory 256 MB RAM

Windows XP Clients

- Processor Intel Pentium, 933 MHz
- Memory 256 MB RAM
- Workstation

The operating system requirements are:

- Windows 2000 with Service Pack 3, Window XP with Service Pack 1

The minimum hardware configuration requirements are:

- Windows 2000 or XP
- Processor Intel Pentium, 933 MHz
- Memory 256 MB RAM
- A Windows compatible printer is required in order to obtain printed reports.
- A mouse or another pointing device supported by Windows.
- A serial port for direct connection to your instrument using the communication cable kit (P/N M0417035). The serial port, to which the HS is connected, cannot be shared with another serial device. You may require additional serial ports for an NCI, a mass spectrometer, and other serial devices.

NOTE: The communication cable is a null modem cable

- A CD-ROM drive for installing the control software.

Refer to the Control Software Manual for details on the installation and operation of the control software.

HS 40/110 Trap Accessories

The HS 40/110 trap offers some options and accessories.

The Integrated Trap

This feature is available on the TurboMatrix 40 Trap and TurboMatrix110 Trap options as a factory installed accessory only. It cannot be fitted at the same time as the cryo-trap.

IS Addition

This feature is available on the TurboMatrix 40 Trap and TurboMatrix 110 trap options as a factory installed accessory or as a field-installed upgrade on systems where the trap has been preciously installed.

Vial Integrity Testing

This feature is standard on the trap based systems for the TurboMatrix 40 Trap and TurboMatrix 110 Trap options.

Vial Temperature Calibration

This feature is standard for the HS16, HS40 and HS110 options.

Method Development 5

Introduction

When you are creating a new method, normally you will optimize the method by performing analyses at various settings. You can use the Status tab to enter the desired parameters and options and then run a few samples to evaluate the method. You can then adjust parameters accordingly and run more samples until you obtain the desired results. Once you have the method you want you can use the **Save As** command in the **Tools** menu to save the new method. You can then update the method at any time or create new methods based on the current one, using the **Method Editor** and the **Edit** command.

Principles of Headspace and Headspace Trap Analysis

Headspace analysis is the analysis of the vapor lying in equilibrium over a solid/liquid sample in a sealed container.

For practical headspace analysis the sample is sealed in a vapor tight vial, placed in a thermostatted oven and heated to a preset temperature.

When equilibrium is reached between the solid/liquid phase and the vapor phase, the sample vial contains the volatile material in equilibrium between the solid/liquid sample and the vapor lying over it.

A defined amount of the vapor is taken and carried to the column in the gas chromatograph for analysis. Total vapor in the case of the headspace trap is taken and carried to the column in the gas chromatograph for analysis.

With this technique only volatile substances reach the column, the non-volatile substances remain in the sample vial.

Using this technique, samples containing constituents which are unsuitable for injection with a syringe can be analyzed (e.g. soil, polymers, highly viscous liquids).

Suitable fields of application are in the analysis of polymers, certifying of the volatile components in drinks and foodstuffs, blood alcohol levels, water and environmental analysis.

The HS Sampling Technique

The HS employs a unique sampling technique - a pneumatic pressure balanced system. The headspace sample is introduced onto the column without resorting to a gas syringe, thus avoiding fractionation due to pressure changes in the syringe. Since the needle is sealed, there are no losses of headspace gas during transfer. The sample injection is executed in three steps:

Thermostatting Phase—During the thermostatting phase (Standby) the sampling needle is in the upper position. The carrier gas flows through solenoid valve V1, to the column; at the same time the needle cylinder is purged by a small cross flow vented through solenoid valve V2 and needle valve V2. The cross flow prevents carry over between injections

Pressurization Phase—After completion of the thermostatting time, the sampling needle moves to the lower position, piercing the sample vial septum. The carrier gas flows into the vial headspace, pressurizing it to equal the sampling head pressure (P1).

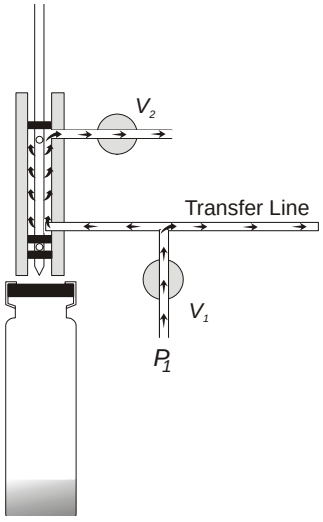


Figure 70 Thermostatting Phase

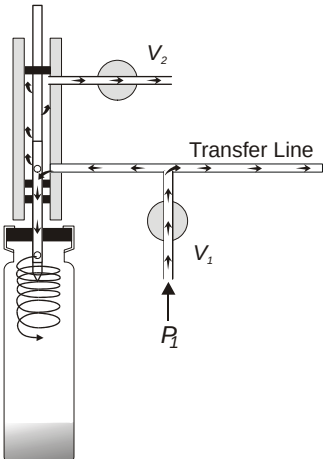


Figure 71 Pressurization Phase

Injection (or Trap Load) Phase —After the pressurization phase, the solenoid valves V1, and V2 are closed, stopping the carrier gas flow. The compressed gas in the vial flows onto the column or trap. After the pre-selected injection time the solenoid valves V1, and V2 are again opened, completing the sampling phase. The carrier gas now flows directly onto the column and branches to the sample vial, preventing additional sample vapor reaching the column.

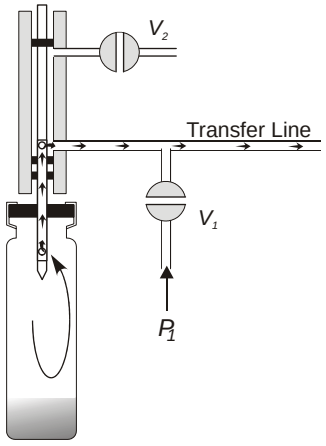


Figure 72 Injection Phase

HS 40/110 Trap Sampling Technique

NOTE: The previous three illustrations show a generalized flow and do not represent the exact plumbing for the HS 40/110 trap.

This sampling technique incorporates some additional steps to the HS Sampling Technique. The HS 40/110 trap Sampling technique and the HS Sampling Technique share the Thermostatting, Pressurization and Injection (or Trap Load) phases (see the explanation of these phases on the previous page). Unique to the HS 40/110 trap are the Pressure Decay phase, the Dry Purge phase and Desorb (split/splitless) phase.

Pressure Decay Phase—This phase allows for the pressure inside the vial to decay into the HS 40/110 trap. The column isolation flow prevents branching of the decay flow to the Transfer Line/GC column and maintains the flow down the column.

Dry Purge Phase—This phase allows you to input the number of minutes required for the dry purge.The dry purge allows helium to pass over the trap to purge the trap of water.The amount of time needed for the dry purge will vary depending on the type of sample you are running and the HS Trap **Cycles** number.

Desorb Phase—The Desorb phase is the time for the sample to be desorbed from the HS 40/110 trap and focused onto the GC column.

The figure below is a generalized plumbing diagram.

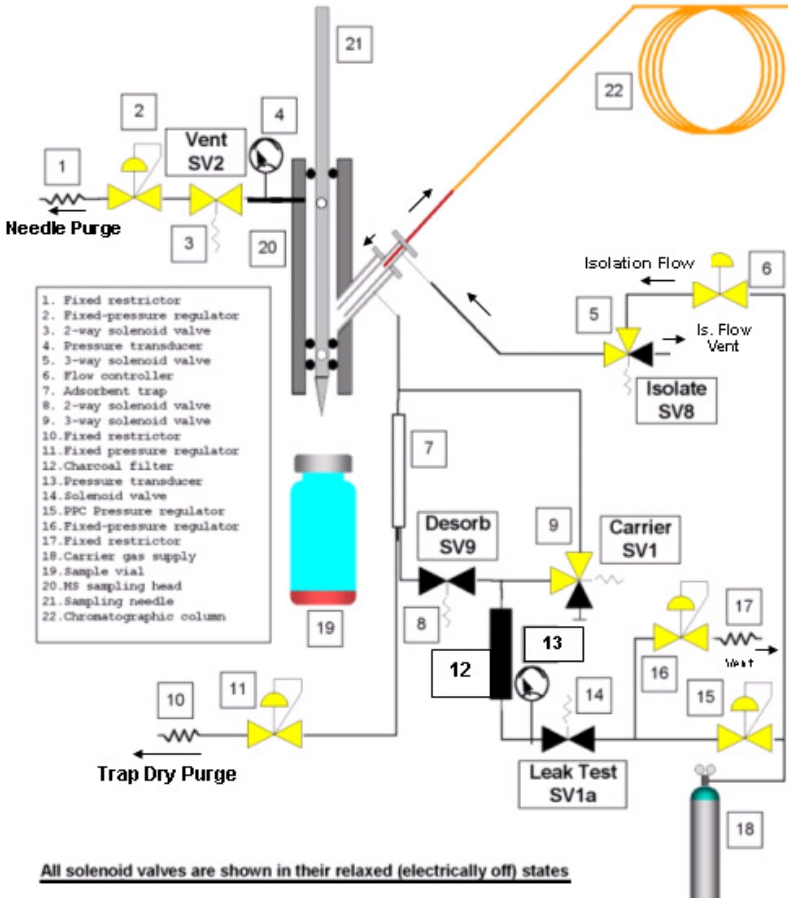


Figure 73

Creating a New Method

To create a new method:

1. Press the **Tools** button and then select **Method Editor** from the drop-down box.
2. To create a new method, press the **New** button. The **New Method** tab opens. The New Method tab is identical to the **Status** tab. It contains four tabs that will allow you to set up various analysis parameters.

   For a **Headspace only** unit:

3. Press the **Timing** tab. Enter values for the Pressurization time, the Inject time, the Withdrawal time (Headspace only) and the **Cycle** time (Headspace/Trap). A PII time will be calculated after you have entered values for each parameter. If the cryofocusing option (Headspace) is installed, enter the pre-cryofocusing time and the post-cryofocusing times (Headspace only).
4. On the **Temperature** tab, press the **Needle** box to activate the option and then press the Plus or Minus keys to enter the desired value. Enter the Transfer Line and thermostatting temperatures in the same manner. If the cryofocusing option (Headspace) is installed, enter the cryofocusing temperature (Headspace only). See *Cryofocusing Temperature (Headspace Only)* on page 193 for details.
5. Press the **Options** tab and enable any options that are required to complete your method. Select the desired operating mode. If you are using MHE mode, you must set certain parameters accordingly. See *MHE Theory and Calculations (Headspace Only)* on page 229 for details.
6. If you have PPC installed, press the **PPC** tab and **Carrier pressure**. If you are using high pressure injections, enter the desired value for this option.
7. A default method name is entered. You can change the method name by pressing the default name. An alphabetic keypad is displayed. Enter the desired sequence name. To enter numeric characters, press the

Num key. Press **Alpha** to return to the alphabetic keypad.

8. Press **OK** to enter the new name for your method and then press Save to save your new method. Press Done to complete the selection and return to the Run tab.

To edit an existing method:

1. Press the **Tools** button and then select **Method Editor** from the drop-down box.
2. Select one of the methods from the list and press the **Edit** button. The Edit Method tab opens. The Edit Method tab is almost the same as the Status tab. It contains four tabs that will allow you to set up various analysis parameters. The parameters for the existing method are displayed.
3. Edit the parameters required for the updated method.
4. Press the **Options** tab and enable any options that are required to complete your method. Select the desired operating mode. If you are using MHE mode, you must set certain parameters accordingly. See *MHE Theory and Calculations (Headspace Only)* on page 229 for details.
5. Press **Save** to save your updated method. Select another method to edit or press Done to complete the selection and return to the **Run** tab.

Temperature Tab (Headspace and HS 40/110 Trap)

Needle and Transfer Line Temperatures

The needle temperature should be set high enough to prevent condensation but not so high that the septum is burned with a needle that is too hot. For best reproducibility, set the needle temperature to a value 5-10 ºC higher than the sample temperature (HS oven temp).

This also applies to the transfer line. Set the temperature at or slightly above (~5-10 ºC) the thermostatting temperature. You must also consider the GC oven and injector temperatures. Do not set the transfer line below the injector temperature.

You must remember that in the heated transfer line, the headspace gas is a mixture of air with trace concentrations of the analytes; thus too high a temperature may cause sample decomposition by oxidation.[1]

Temperature Mode

Three temperatures can be combined so that when you raise or lower the combined temperature, all three settings are adjusted. If for example you enable the **Combined** option and raise the temperature by 5 ºC, then the needle, the transfer line and the thermostatting temperature will all be raised by 5 ºC. If you choose the **Separate** option, then each temperature can be set independently.

Track Oven (Headspace and HS 40/110 Trap): The **Combined** temperature option is activated if you enable the **Track Oven** by checking it's box.

NOTE: The needle and transfer line must be maintained at 5-10°C higher than the thermostatting temperature.

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 71

Thermostatting Temperature (Headspace and HS 40/110 Trap)

Before withdrawing a headspace sample for analysis, the vials are thermostatted until equilibrium between the two phases is reached. In the HS 16 one vial is thermostatted. With the HS 40 and the HS 110 you have the option of thermostatting up to 12 vials.

NOTE: Although the thermostatting oven has 15 positions the software will only allow up to 12 vials for simulaneous thermostatting.

The thermostatting temperature you set here will depend on your sample and the sample matrix. There are many factors to be considered when you are setting the thermostatting temperature. A detailed discussion of the effects of temperature on vapor pressure, partition coefficients and headspace sensitivity is provided in Static Headspace Gas Chromatography, Theory and Practice by Bruno Kolb and Leslie S. Ettre (P/N N1011210).

You can set the thermostatting temperature to any value between 35 and 210 ºC, in steps of 1 ºC. If you set the thermostatting temperature to 0, the heaters are turned off.

Although the HS will allow you to set the thermostatting temperature to a maximum value of 210 ºC, the vial seals are rated for lower temperatures (see the *Seals* section, earlier in the chapter). The seals will begin to vent when the internal vial pressure exceeds 73 psi (500 kPa).

Track Oven (TurboMatrix Headspace and HS 40/110 trap Temperatures): The **Combined** temperatures option is activated if you enable the **Track Oven** by checking its box.

NOTE: If you set the needle/transfer line or thermostatting temperature to a value below room temperature, the whole instrument must be cooled, i.e. in an environmental chamber to a temperature 5 ºC below the thermostatting temperature. This is outside the operational specifications of the HS.

HS 40/110 Trap Temperatures

The speed of Trap temperature rise and the maximum allowable Trap temperature can be selected and set on the Trap Setup.

Tools--->**Preferences**--->**Config** tab--->**Trap** Setup...

You can choose either Fast or Slow Heat Rate from the drop down list.

The default Trap Maximum Temperature is 400 ºC. It depends upon the Trap material. Unauthorized changes to higher values should be avoided.

CAUTION	*Follow the manufacturers recommendations for the upper trap temperature. If the temperature is set too high the trap and instrument could be severely damaged.*

Cryofocusing Temperature (Headspace Only)

Cryofocusing (or cold trapping) is the technique of sample concentration through the application of low temperature. The injected analytes are pre-concentrated on the capillary column. The sample is cooled at the column inlet; the volatile components in the headspace are separated from the unretained gases and concentrated.

If the cryofocusing accessory (M041-3411) is installed and enabled, the cryofocusing temperature options will be displayed on the Temperature tab.

In the cryofocusing technique, a portion of the capillary column is fed through a Teflon® tube through which a coolant gas is passed. The coolant gas is cooled by passing it through a copper coil immersed in a Dewar vessel containing liquid nitrogen. The gas flow is switched on and off by means of a solenoid valve (SV6) fitted to the HS. This valve is controlled by the HS and the cryofocusing temperature is maintained by switching this valve. Refer to the user's manual for the Cryofocusing Accessory, (P/N M041-3578) for detailed instructions on operating the cryofocusing option.

The cryofocusing temperature can be set to any value between -100°C to -30°C. The value to which you set the temperature will be

determined by your application. For optimizing the coolant gas temperature for you application refer to Static Headspace Gas Chromatography, Theory and Practice by Bruno Kolb and Leslie S. Ettre (P/N N1011210).

Timing Tab

You can view the timing values for HS operation on the Timing tab. Once all the correct timing values have been entered the period from injection to injection (PII) is automatically calculated by the Headspace.

Pressurization Time (Headspace and HS 40/ 110 Trap)

After equilibrium has been reached, the vial is pressurized. The pressurization time is the duration you want the vial to be in the carrier gas flow. Excess gas pressure is vented through the needle purge port.

When you are using capillary columns, a pressurization period of 1-3 minutes is recommended. The pressurization time needs to be long enough to ensure homogeneity of the gas phase in the vial. The pressurization time should be at least two minutes for good reproducibility and three minutes for optimum reproducibility. For some applications where a short pressurization time is used to increase productivity, expect deterioration in performance.

Vial Pressurization-Carrier Pressure (Headspace Only)

In order to obtain good reproducibility it is important that the column head pressure be greater than the pressure in the sample vial. At the beginning of the pressurization phase when the needle moves down into the vial, pressure is released. Part of the pressure built-up between the injection port and the column head is released to the atmosphere through the hole located laterally at the lower part of the needle. Thus, if the difference in pressure between column head and vial is too small, part of the sample in the vial headspace will be

taken up in the carrier gas flow prior to the actual injection, resulting in an undesirable, double injection.

In order to prevent such double injections, the column head pressure should be greater than the vial pressure (Carrier Pressure in PPC tab) at the preselected thermostatting temperature, by at least a factor of 1.2 + 20 kPa; (100 kPa = 1 bar).

> **Example:** At a vial pressure of 50 kPa, the column head pressure should be greater than 80 kPa. If, chromatographic conditions demand a column pressure of 90 kPa, the above criterion is met (90 > 80).

The scale below shows the relationship between vial pressure and thermostatting temperature in samples with a water matrix:

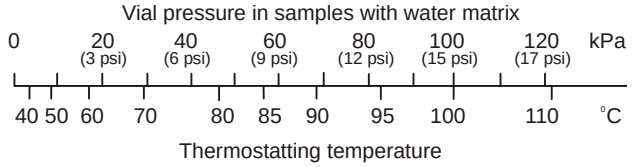


Figure 74 Relationship Between Vial Pressure and Thermostatting Temperature in Samples with a Water Matrix

Injection Time (Headspace Only)

The carrier gas supply line and the pressurized gas in the vial expand onto the column, resulting in a flow of the headspace gas from the vial to the column.

Since both the pressure in the vial and the transfer time can be set, the transferred volume of the headspace gas can be accurately controlled. A separate gas supply is not required for pressurization: the vial is pressurized to the carrier inlet pressure.

The sample injection time can be set to any value from: 0.00 to 9.9 min in steps of 0.01min and 0.1 min. The value that you set will depend on your application. You may need to determine the actual setting experimentally using known standards.

Thermostatting Time (Headspace and HS 40/ 110 Trap)

The time needed for equilibration depends on the diffusion of the volatile sample components from the sample and then back into the sample. The equilibration time cannot be predicted. Therefore, in the case of an unknown sample, particularly if no previous information is available, the required equilibration time must be established empirically. You can do this by preparing a number of vials with the same sample, thermostatting them for different times and then using identical analysis conditions, and plotting the resultant peak area against the thermostatting time.

Particularly useful for time-consuming kinetic measurements (Headspace only) is the automated progressive mode of thermostatting. For details see The *Options Tab* in the *Operation* chapter.

The equilibration time should be the shortest time the sample has to be thermostatted. The analytical result will not change if the thermostatting time is longer than the time needed for equilibration. However excessively long thermostatting should be avoided, because some samples may be sensitive to prolonged heating.

The equilibration time may be fairly long-in fact, generally longer than the analysis time. Starting the equilibration of the second sample only after the analysis of the first has been accomplished, will result in an unnecessary loss of time and sample throughput. To eliminate this unnecessary time delay for routine analyses, you can thermostat each sample for the same time and start it while the preceding sample is still being analyzed. This is the overlapping constant mode of thermostatting. You can only use overlapping thermostatting if you have the 15-vial oven option installed on the HS 40/110 only.

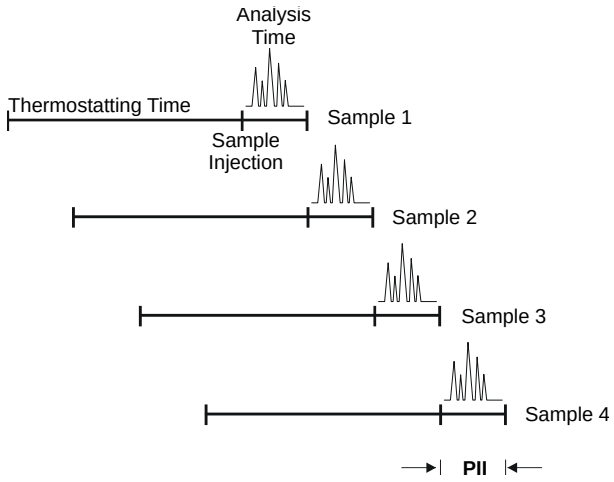


Figure 75 Overlapping Thermostatting

NOTE: In special cases you may need to work under non-equilibrium conditions.[1]

You can set this value to: 0.0 to 9.9 min in steps of 0.1 min, 10 to 99 min in steps of 1 min; and 100 to 990 min in steps of 10 min.

If you are using MHE mode (Headspace only), set the thermostatting time so that it is equal to the GC cycle time.

Injection Volume (Headspace Only)

The injection volume (uL) is based on the column flow rate and the injection time. It corresponds to the flow rate measured at the end of the column under normal atmospheric pressure and temperature conditions. The calculation of the injection time for the entered volume is based on the assumption that the vial pressure remains constant during sampling and no other gas is supplied to the column.

This parameter defines the sample injection volume. Valid range: 0 to 99 uL, 100 to 990, 1000 to 10000 uL, in steps of 1, 10 and 100 uL.

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997, Perkinelmer P/N N1011210), p. 118

Withdrawal Time (Headspace Only)

This is the length of time after the injection, before the sample needle is withdrawn from the sample vial or lowered into the vent position. During this time the needle remains in the vial.

You can set this value to: 0.0 to 9.9 min in steps of 0.1 min, 10 to 99 min in steps of 1 min.

When you are using the high pressure sampling option, you must set the withdrawal time correctly to avoid double injections. See *Setting the Withdrawal Time for High Pressure Sampling* earlier in this chapter.

Setup (HS 40/110 Trap Only)

Decay Time

After vial pressurization, the next step is to use the pressure in the vial in order to load the trap with the largest possible part of the headspace vapor (a reverse flow). Consequently, this step should last until the pressure in the vial has decayed to its lowest value.

The Decay time to be set in the method appears in the Trap Timing screen as shown below.

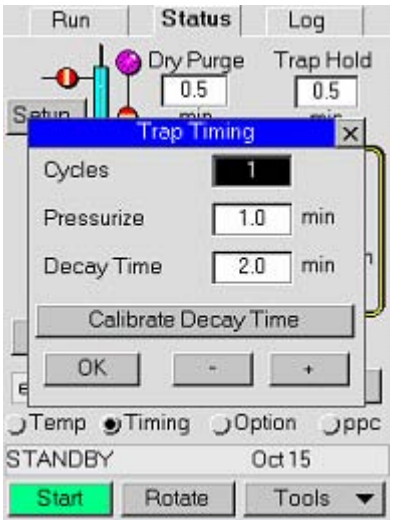


Figure 76

This value is decided after the Calibrate Decay Time procedure is chosen in the Trap Timing screen above.

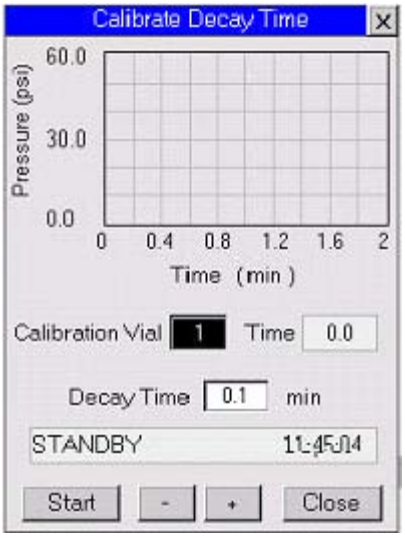


Figure 77

The above calibration is run with a calibration vial, identical in content (solvent) and sample volume with the vials to be analyzed.

When this procedure is started, the system thermostats this vial for a preset Thermostatting time of 2 minutes. Thermostatting temperature and subsequent Pressurization (Pressure and Time) are carried out under the values set in the method. The Pressure Decay in the **Calibration** lasts two minutes preset by the system.

At the end of this process, the system will display for you the pressure decay curve.

On choosing Calibrate Decay Time, loading the calibration vial and pressing the **Start** button, the following screen appears and this process begins.

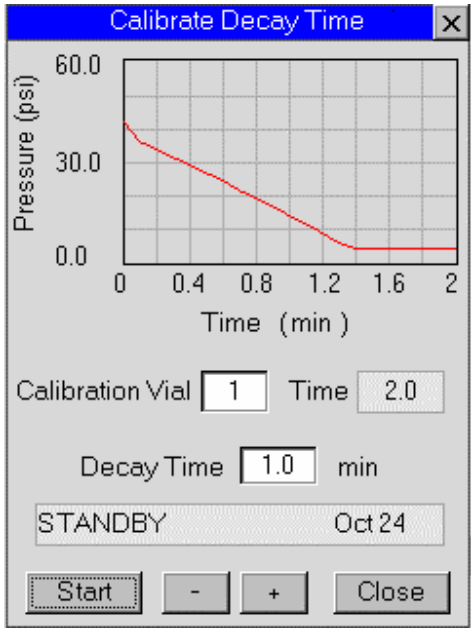


Figure 78

End of the Calibrate Decay Time function:

This is a correct Pressure Decay. The calibration curve is linear with a good slope. It reaches minimum pressure at around 1.4min. A safe Decay time in the method would be 1.6min.

The Pressure Decay curve should be a linear function with a proper slope to the vial lowest pressure after decay ends.

The Decay time should be set at a value safely beyond the pressure curve minimum but not too long as this will unnecessarily prolong the overall vial handling time.

The Decay Time range of values is 0.1-99.9 min, in steps of 0.1 minute.

NOTE: This unique Decay curve profile feature is memorized and used throughout the vials run in order to Monitor Vial Integrity, alerting the operator if the decay curve for any sample run under the method is not consistent with the curve in the memory. This could indicate improper vial septum seal or incorrect sample volume. Leaks of a different origin and cause are also immediately detected. The system optionally reports a wrong curve in the Log tab or, it even stops the run (choice of the Stop on Vial Error option).

*With the **Monitor Vial Integrity** and the **Stop and Vial Error** enabled. The system terminates the counting after three consecutive vials have failed the Vial Integrity test.*

This feature and various aspects on it, will be handled in the Troubleshooting chapter.

Cycles

From the Timing tab and the Setup. button, we can set the Cycles number in the Trap Timing pop-up screen:

Timing tab---->Setup button----->Trap Timing screen

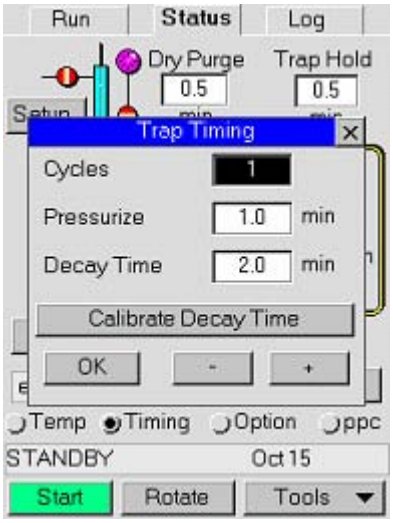


Figure 79

The Cycles number can be set from 1 to 4 cycles and is the number of times Vial Pressurization and Trap Load will be performed per vial. Less than 1% of the headspace vapor is lost as a vial residue. More than 99% is adsorbed and focused in the HS 40/110 trap.

Pressurization Time

After equilibrium has been reached, the vial is pressurized by the carrier gas to a pressure equal to the vial pressure and for a time set as Pressurize time in the Trap Timing screen that pops-up if you touch the **Setup.** button.

Timing tab---->Setup button----->Trap Timing screen

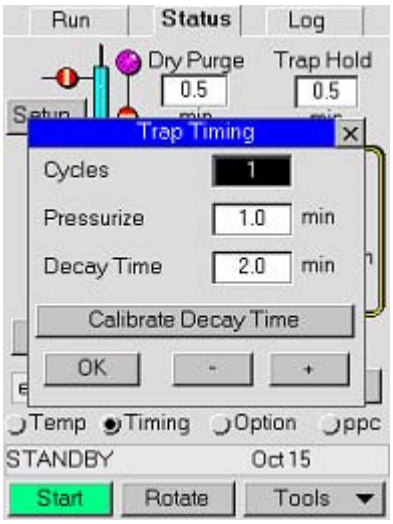


Figure 80

You can set the pressurization time to any value from 0.1 to 999 minutes, in steps of 0.1 min. The default is 1 minute.

Trap Hold (HS 40/110 Trap Only)

This option allows you to input the number of minutes that the trap will maintain the maximum temperature. In order to release the analyte from the trap and completely clean the trap from sample residue to ready it for next sample, the maximum temperature must be maintained for a sufficient amount of time. A recommended trap hold time is five minutes.

Its range of values is 0-999 minutes, in steps of 0.1 minute.

Dry Purge Time (HS 40/110 Trap Only)

After trap loading is the optional step of removing the moisture from the trap tube.

This option allows you to input the number of minutes required for the dry purge.The dry purge allows helium to pass over the trap to purge the trap of water.The amount of time needed for the dry purge

will vary depending on the type of sample you are running and the HS Trap Cycles number. The higher the number of Trap re-loadings, the higher the HS oven temp and the more moisture your sample has, the higher the moisture will condense.

Note: If the FID extinguishes during your analysis you will have to increase the dry purge time.

Desorb Time (HS 40/110 Trap Only)

Desorb time is the time for the sample to be desorbed from the HS 40/110 trap to the transfer line/column (GC). Typically for a 10 ml sample 0.5 min is plenty of time.

Pre/Post-Cryofocusing Time (Headspace Only)

These optional parameters appear only if the cryofocusing accessory is installed and the option has been enabled. The pre-cryofocusing time takes place before the sample injection.

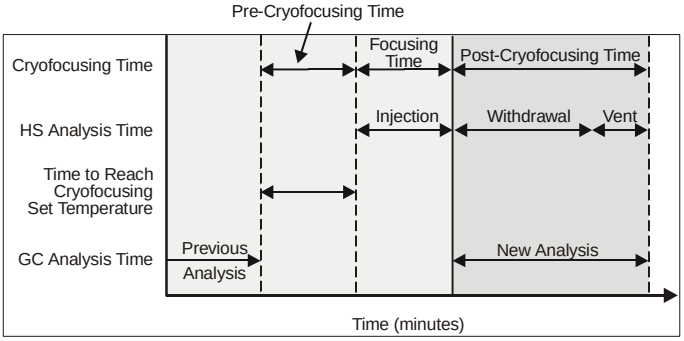


Figure 81 Setting the Pre and Post Cryofocusing Time

NOTE: The reference point for the cryofocusing times is always the end of the injection time.

The system parameters pre and post-cryofocusing time are used to optimize the cryofocusing duration before and after sample injection.

The cryofocusing duration determined when optimizing the cryofocusing system should be used as a guideline for the pre-cryofocusing time in the headspace method. The total cryofocusing time comprises the pre- (before and during sample injection), focus time (inject time) and post- (after injection) cryofocusing times.

The pre-cryofocusing time must be sufficiently longer to ensure that the headspace has reached the required low temperature before an injection begins. Typically, this would be at least two minutes before the injection time begins, depending on the pressure/flow of the cooling gas. During the pre-cryofocusing time, the GC oven should be less than 45°C to ensure adequate cooling of the trap.

The pre-cryofocusing time can be measured using the Cryo Test on the Test and Diagnostics option on the Tools menu.

To establish the pre-cryofocusing time:

1. Enable the Cryofocusing option and water trap (if it is installed) on the Options tab.

NOTE: Check that the GC is cooled and in the ready state.

2. On the Temperature tab, enter the desired cryo temperature. This is the temperature the head of the column will be cooled to.
3. Select the Tools menu and then select Test. On the pop-up menu select Cryo Test.
4. Press OK to begin the test. The HS begins to cool the head of the column. It also begins to measure the time until the set temperature has been reached.
5. Once the set temperature has been reached, the test is stopped. The displayed time is the pre-cryofocusing time.

The post-cryofocusing time represents the time after the end of the injection, during which the analytes, which are still in the water trap and other void volumes, are transferred to and focused on the headspace before the start of chromatography. An adequate post-cryofocusing time is required to ensure that the peaks are sharp and do not tail as a result of the above mentioned void volume effects, etc.

The post-cryofocusing time should be longer than the withdrawal time plus vent time up to a maximum of 2-3 minutes. Typically, you should set the post-cryofocusing time to approximately 1 minute.

Cryofocusing is started prior to sample injection. The first 8” of the column are cooled to the cryofocusing set point before the injection is made. It is therefore important to make sure that a pre-cryofocusing time is selected so that it does not interfere with the chromatography of the previous run.

This is of particular importance when operating in Constant mode, as this involves the simultaneous thermostatting of several samples. In such cases the cycle time must be prolonged by an amount equal to the cryofocusing time.

In Progressive mode, a prolonged cycle time is not necessary, as only one sample is thermostatted.

Post-cryofocusing enables particularly volatile components to be separated from the broader air peak, which is necessary, for example when you are using ECD detection. Post-cryofocusing time can normally be very short; however, it should be at least equal to the injection time + withdrawal time.

You can set this value to: 0.0 to 9.9 min in steps of 0.1 min, or 10 to 99 min in steps of 1 minute.

The time taken for chromatography of the sample is determined by the GC method. You must enter a value for the GC Cycle Time into the HS.

The Option Tab

The Options tab provides access to the headspace method options. There are some options that you may need for some methods but may not need for others. You can enable vial venting, the shaker, cryofocusing and high pressure injection from this tab. In the case of the headspace trap instrument add IS (internal standard), outlet split and dry purge. The high pressure injection option is only available if you have the PPC option installed. The cryofocusing option is only available if it has been installed on both the HS and the GC column oven.

You also select the operating mode and the injection mode from this tab.

Injection Mode (Headspace Only)

The volume of the injected sample can be entered as an injection **Time** or an injection **Volume**. The injection volume (uL) is based on your entry for the column flow rate. The injection volume corresponds to the flow rate measured at the end of the column under normal atmospheric pressure and temperature conditions.

for Headspace only, if you select injection volume as your mode of injection, specify the column flow rate and the desired injection volume. The HS will automatically calculate the corresponding injection time. If you select time as the injection mode, the HS will determine the injected volume from the time you have entered, on the timing tab, and the column flow rate.

The calculation of the injection time for the entered volume is based on the assumption that the vial pressure remains constant during the sampling and no other gas is supplied to the column.

In the case of balanced pressure sampling systems the injection volume can be controlled by the injection time. The injection time should be limited to a few seconds. During the injection time the transferred gas volume is determined by the actual gas velocity.

If we consider the same linear flow rate at the column inlet (u_i), you can calculate the transferred volume (V_H) injected onto the column from the injection time (t) and the cross section of the column (Q_c):

$$V_H = (Q_c)(u_i)t \qquad \textit{Equation 1}$$

The admissible sample volume onto an open-tubular column is limited by the column's sample capacity. Overloading the capillary column leads to band broadening, and this effect is independent of the method of sample introduction.

Principles of High Pressure Sampling (Headspace Only)

High Pressure Sampling—This technique utilizes internally regulated carrier gas. The carrier gas is supplied by the HS PPC module. If you are using the high pressure injection option, you will set the injection pressure here.

You must first enable the option on the Options tab. This option is available if you have PPC installed. You can enable it on the PPC tab. You will enter the desired injection pressure on the PPC tab in the Status tab or on the Method Editor tab.

High Pressure (P2) must be higher than the internal vapor pressure in the headspace sample vial. Ensure that pressure P2 does not exceed 500 kPa (70 psi).

Both P1 and P2 are supplied by the PPC module at V1. The PPC module automatically increases the pressure from P1 to P2 at the start of pressurization. The PPC module returns the pressure to P1 after the withdrawal time or vent time.

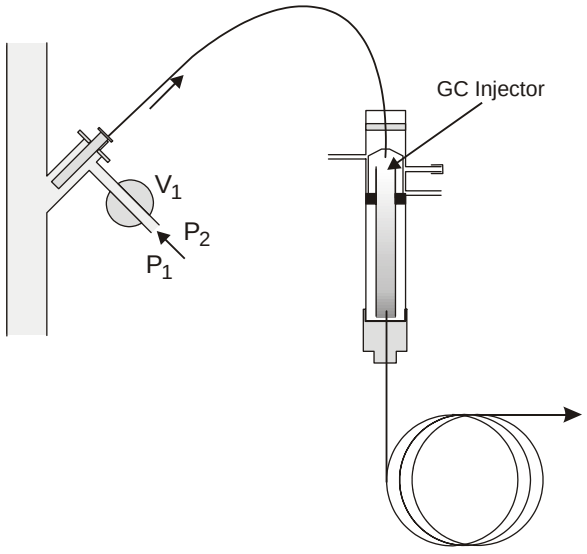


Figure 82 High Pressure Sampling

Standby—Carrier gas at pressure P1 is applied to the inlet of solenoid valve V1 by the PPC module. During sample thermostatting, the system is in the standby position. Carrier gas flows through V1 to the GC column. The needle purge flow enters the needle cylinder and vents via V2. See *Figure 83*.

Pressurization—At the end of the thermostatting time, the needle enters the sample vial. Simultaneously, the PPC module will now supply pressure P2. The sample vial is pressurized with carrier gas at pressure P2. See *Figure 84*.

P2 is also supplied to the GC. In direct connect mode this high pressure flows directly through the GC column. In the standard HS connection at the injector, the high pressure (P2) is supplied to the injector, but is reduced to P_{GC} as it passes through to the GC column.

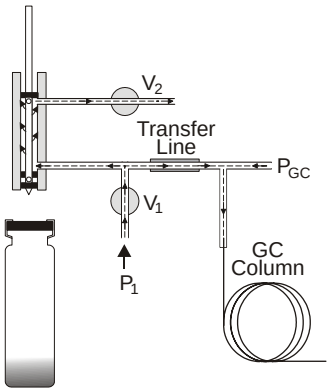


Figure 83 High Pressure - Standby

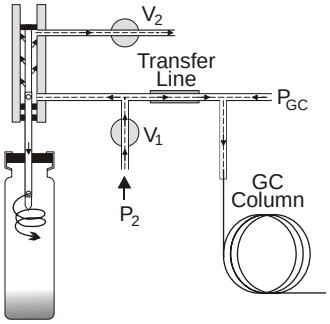


Figure 84 High Pressure - Pressurization

Sampling—At the end of the pressurization time, the PPC module stops the flow of carrier gas. Vapor from the vial headspace expands onto the column due to the pressure drop across the transfer line. See *Figure 85*.

Again, in direct connect mode this high pressure flows directly through the GC column. In the standard HS connection, P2 is supplied to the injector, but is reduced to P_{GC} as it passes through to the GC column.

At end of the injection time the solenoid valves V1 and V2 open. Carrier gas flows onto the column where separation of the sample takes place.

Withdrawal—At the end of the injection time, the needle withdraws from the sample vial. The system is once again pressurized to P2. In the direct connect configuration, high pressure carrier gas continues to flow over the GC column for the duration of the withdrawal time.

Vial Venting—At the end of the withdrawal time the sampling needle is positioned to vent the vial, if vial venting is enabled. When the sampling needle returns to the standby position, the PPC module establishes the original pressure (P1).

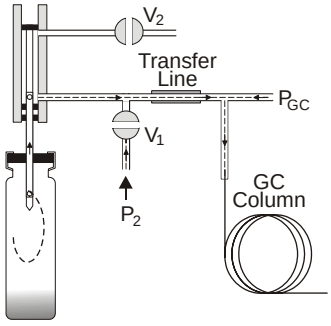


Figure 85 High Pressure - Sampling

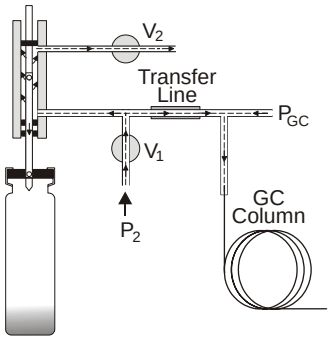


Figure 86 High Pressure - Withdrawal

Setting the Withdrawal Time

For High Pressure Sampling

The high pressure augments to the column head pressure until the end of the withdrawal time. The withdrawal time should be long enough to ensure that enough of the sample has moved onto the GC column before returning to normal pressure. This reduction in pressure causes a temporary reverse in the gas flow at the column head. This should only have an effect on the carrier gas - not on the sample!

When working with wide-bore capillary columns (0.53 mm i.d.) or with short quartz capillaries (0.32 mm i.d.) a short withdrawal time is recommended (e.g. 0.1 min). This avoids unfavorable GC conditions (very fast chromatograms) caused by the extreme flow rates due to the high pressure (P2).

If the withdrawal time is too short, split peaks and loss of injection volume may result.

If the withdrawal time is too long, the chromatographic separation may be unacceptable due to the extreme flow rate through the column.

Water Trap

In cryofocusing, water is removed from the sample prior to enrichment using the water adsorption trap. Water from the sample is removed by adsorption onto a hygroscopic salt.

The water adsorption trap is necessary to prevent the GC column from becoming blocked by the formation of ice.

Operating Modes (Headspace Only)

Constant Mode—Constant mode is the standard mode. In this mode, the sample vials have the same thermostatting time. This mode is generally used for all routine operations.

The instrument software calculates the PII value (Period from Injection to Injection) from the parameter values entered. The PII value is shown on the display. For optimum sample throughput (i.e. the greatest number of samples analyzed in the shortest time), it is essential that the PII value is only slightly longer than the cycle time of the selected GC method.

If the above conditions can be fulfilled, then the thermostatting times for the individual samples will overlap.

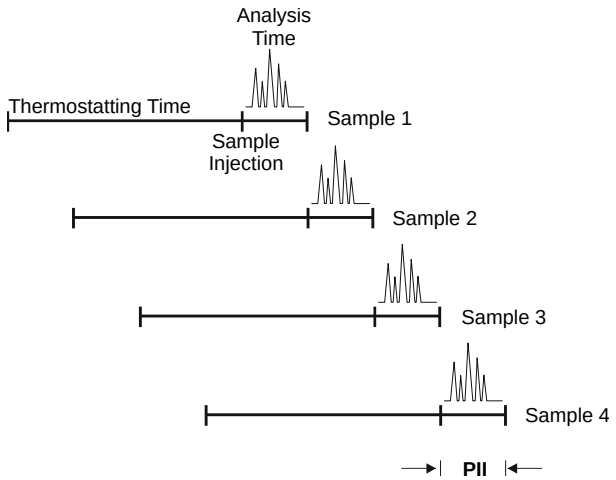


Figure 87 Overlapping Thermostatting

In the worst case, the samples will be sequentially thermostatted. You can normally achieve optimum thermostatting conditions by making slight adjustments to the thermostatting time or the GC Cycle time.

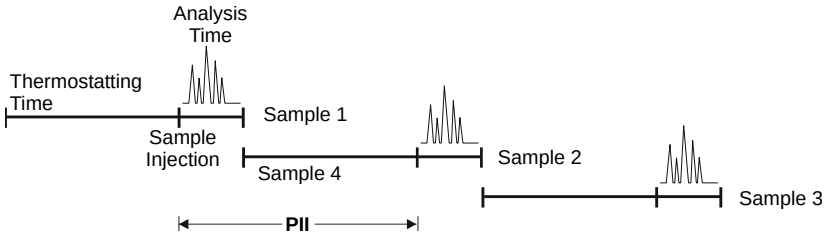


Figure 88 Sequential Thermostatting

MHE Mode—The MHE mode (Multiple Headspace Extraction) is used to quantitatively determine an analyte in a sample. It is especially useful in the analysis of voliatiles in insoluble samples. Such samples cannot be quantitatively analyzed directly by headspace gas chromatography, since it is not possible to prepare reference solutions. This function is used for method development and validation as well as for quantitative analysis of difficult solid samples.

NOTE: Does not work with compounds with a high concentration of Potassium.

In MHE mode the headspace gas in the sample vial is extracted and analyzed successively with pressurization of the sample vial between each extraction and automatic venting between extractions. Up to nine extractions can be taken with the HS. During venting a portion of the headspace vapor escapes.

As each successive extraction and analysis is run, the peak areas of the constituents decrease. If the extraction is run to exhaustion, it is only necessary to sum the peak areas of the analyte to determine its concentration in the sample.

In practice it is not necessary to run the extraction to exhaustion. After a minimum of two extraction steps, the sum of the peak areas can be calculated from the geometric progression.

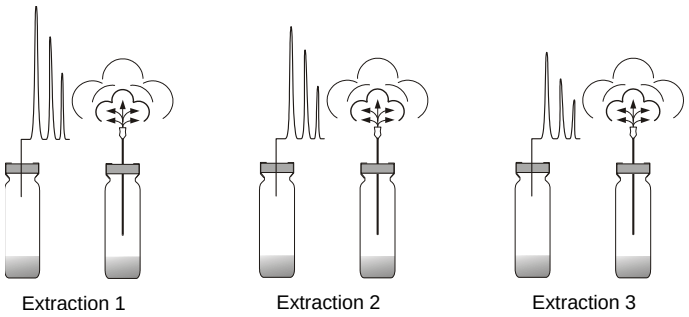


Figure 89 MHE Mode

Progressive Mode—This function is used for initial method development to determine the thermostatting time required for a specific application. The function can also be used for kinetic studies. Vial position one must be filled. For every vial position after position one, the thermostatting time will be increased by n times the initial thermostatting time where n is the sample number.

In progressive mode, the thermostatting time of the nth sample is n times the value chosen for the 1st sample. For example Sample 1 is thermostatted for the value entered. Sample 2 is thermostatted for twice the entered value, Sample 3 for three times the entered value, and so on.

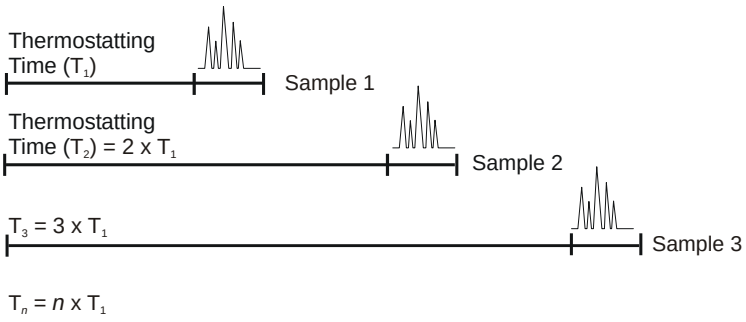


Figure 90 Thermostatting Time Using Progressive Mode

Number of Injections

This parameter defines the number of extraction steps in multiple headspace extraction methods. Valid settings are 1 to 9 injections.

If you take two consecutive aliquots from the headspace of the same vial, although the partition coefficient (K) remains constant, the peak area obtained for the second aliquot may be smaller than the area obtained in the first analysis. If you continue taking successive aliquots from the vial's headspace, the total amount of the analyte present will further decline, eventually becoming totally exhausted. Thus, the sum of the amounts of the analyte removed in the individual extractions will be equal to the total amount of analyte present in the original sample. This is what we call the multiple headspace extraction method. Its advantage is that by extracting the whole amount of the analyte, any effect of the sample matrix is eliminated and quantitative determination of the total amount present in the headspace vapor can only be calculated.

In practice, you will not carry out the extractions indefinitely: from a limited number of consecutive extractions the peak area corresponding to the total amount of analyte present is obtained by extrapolation, based on mathematical relationships.[1]

For the HS 40/110 trap Only:

The Operating Mode has the following choices in the drop down window in addition to the standard headspace modes:

- **Trap-**Use the trap for concentrating the analytes onto the absorbent trap before injection into the GC column.
- **Trap Clean**-without a vial, uses high temperatures to vent the contaminants from the trap.
- **Trap Test**-without a vial, as a "blank" injection is made into the GC where the material from the trap only using the trap high temp is sent down the column for analysis.

Shaker

The shaker can decrease the time needed for equilibration by providing continuous mixing of the sample in the vial during the equilibration process. When applying the shaker, it is important that the sample be in resonance with the shaker frequency to obtain the desired mechanical mixing effect. In the case of liquid samples, the

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 40-41

resonance frequency depends on the sample viscosity and its volume. To overcome this problem, the frequency scanning shaker varies its frequency automatically during equilibration, through a broad frequency range, so that each sample in the oven reaches its resonance frequency.

A shaker is recommended for the determination of non-polar volatile organic compounds (VOCs) in aqueous solutions exceeding a volume of about 3 mL. On the other hand, shakers do not have much effect on polar compounds in aqueous solutions.

Equilibration of a solid sample in general, but not necessarily always, takes longer than the equilibration of a liquid sample, and it depends on the structure of the solid and also on the equilibration temperature.

Long equilibration times are often characteristic of solid samples, where the diameter and thickness of solid particles will determine the time of diffusion. The porosity and surface area of the solid sample is important and influences the speed of equilibration. Therefore, some porous solid samples with a high surface area often have surprisingly short equilibration times.[1]

The activated sample shaker remains in standby and only starts an automatic shaking program when a headspace method, that utilizes the shaker, is started. It is possible to shake some vials and not others using a sequence of methods.

In MHE mode the optional shaker remains switched off as the needle remains in the vial between analyses.

PPC Tab

Programmed Pneumatic Control (PPC) is the electronic control of pressures for inlet, and auxiliary gases.

The PPC module is standard in the **Headspace with Trap** version. The PPC control modules regulate pressures using electronically

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 121-123

driven variable flow restrictor. The control modules also contain pressure transducers to provide feedback for complete monitoring.

A PPC controller board drives the variable restrictors on the control modules by comparing actual pressures with set points determined from user entered values.

You can set the carrier gas pressure from the PPC tab.

a) Headspace only version:

Carrier Pressure: Set the pressure required for the GC column and the analysis temperature program.

You will set the carrier pressure on this tab even if the PPC module is not installed on your instrument.

There are a number of considerations when you are setting the carrier gas pressure. If you have connected the HS at the GC injector and you are using split injection, then the carrier must be set so that the HS carrier pressure is approximately 5 psig higher than the GC column head pressure.

b) Headspace/Trap version:

Column: Set the pressure required for the GC column and the analysis temperature program.

Set also **Vial** pressurization and **Desorb** pressure values.

If you are using a **Direct** or **On-column** connection, then you are also supplying the GC carrier gas and you must consider the type of column and the GC temperature program when you are setting the carrier pressure.

You may need to connect a flow meter to the end of the column to accurately measure the column flow rates.

High Pressure Sampling—The high pressure sampling technique is necessary if the vial pressure exceeds the column head pressure. The standard balanced pressure sampling technique requires the column head pressure to be higher than the internal vapor pressure in the thermostatted sample vial.

When using wide-bore capillary columns (0.53 mm i.d.), or high thermostatting temperatures, it is possible that the internal pressure in the vial, generated by the partial vapor pressures of the sample components, can exceed the column head pressure. The differential pressure between column head pressure and vial pressure becomes negative.

In such cases the sample expands onto the separating column as soon as the sampling needle moves down into the vial at the start of the pressurization phase, causing double peaking and split peaks in the chromatogram. The high pressure sampling technique permits sampling in such cases, without these secondary effects.

The maximum injection pressure must not exceed 73 psi (500 kPa). The minimum pressure can be determined as follows:

NOTE: Confirm that your temperatures are at set point and that the headspace is ready.

1. Extend the pressurization time to approximately 20 minutes. Load a vial containing a sample into the magazine and start a run. You do not need to save this method. You must be in the Status tab, or the HS will attempt to run the method or sequence on the Run tab.
2. Wait until the thermostatting time has elapsed. Press the [Run] key on the GC, at the start of the pressurization time.
3. Should a chromatogram appear during the pressurization time, the setting for injection pressure is too low. Repeat this procedure at a higher injection pressure until the pre-injection of the sample is eliminated.

This option is available if you have PPC installed. You can enable it on the PPC tab. See *Cryofocusing Accessory* on page 172 for a detailed description of this option and some operating guidelines.

Split Sampling (Headspace Only)

Split sampling is recommended for most headspace applications using capillary columns. Using this technique, the sample is delivered more efficiently through the transfer line. A pressure drop of 5 to 7 psi (35 to 50 kPa) can be generated across the transfer line,

depending on the split flow. This can be useful when operating short capillary columns or wide bore capillary columns at a limited head pressure, while the headspace vial equilibrium pressure is high.

Typically, when changing from splitless sampling to split sampling, there is no reduction, or only a very limited reduction, in sensitivity. This is due to the operating principle of pressure balanced sampling where during injection, carrier gas is replaced by sample gas from the pressurized headspace vial. The headspace vapors are not diluted by carrier gas during the sample transfer unless additional carrier gas is supplied by the GC pneumatics.

For split injection, a split/splitless or programmable split/splitless (PSS) injector must be installed in the GC. When performing split sampling, the carrier gas pressure (P1) is supplied by the HS. PPC module to control the pressure (P_{GC}) in the split injector.

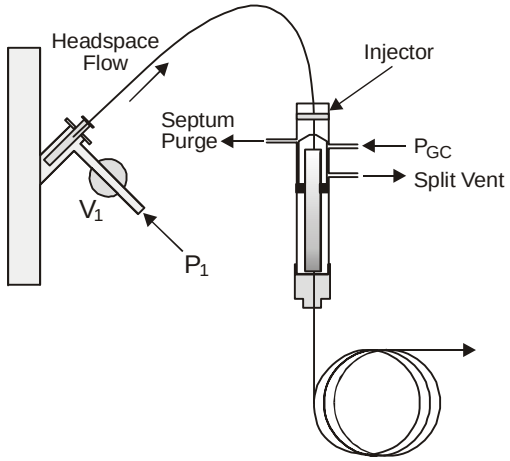


Figure 91 Split Sampling

In dual pressure regulated, split sampling, as seen in *Figure 91*, carrier gas continuously purges the split/splitless injector to avoid back diffusion and sample carryover. When operating with PPC control, a minimum flow of approximately 2 mL/min should be supplied by the GC's injector split pneumatic modules to purge the GC carrier gas lines. Keeping this flow smaller than the septum purge flow ensures, that no dilution of the injected headspace sample

takes place in the injector. A higher flow from the GC pneumatics can be applied to dilute the injected sample if necessary.

In order to prevent pre-injections, P1 should be greater than the headspace pressure in the vial, at the preselected thermostatting temperature.

P1 = 1.2 x Headspace Pressure + 20 kPa ***Equation 2***

P1 should be at least 30-50 kPa (4-7 psi) higher than P_{GC} to account for the pressure drop caused by the split. The split vent flow should be at least 2 mL/min.

For a graph on equilibrium pressure for aqueous samples as a function of thermostatting temperature, see *Figure 74*. The equilibrium pressure can also be measured directly from a thermostatted vial using the vial pressure gauge (P/N B0501377).

The following diagram indicates the pressures in the sample vial, sampling head and column head in the system with split operation:

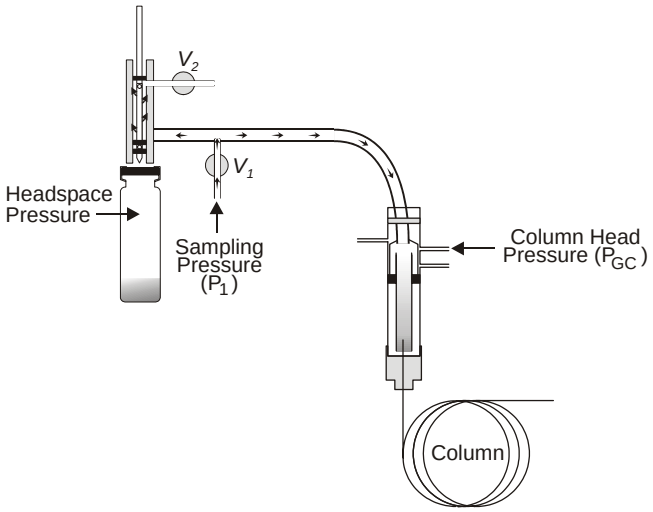


Figure 92 System Pressure During Split Operation

Example Setting P1 and P_{GC} for split operation: Aqueous sample is to be thermostatted at 80 °C. A P1 of 17 psi (120 kPa) is required for split operation, and a column head pressure of 10 psi (70 kPa) is required.

1. Set P1 to 17 psi (120 kPa).
2. Adjust the split flow on the GC until you reach 9 psi (60 kPa).
3. Adjust P_{GC} until you reach the required pressure of 10 psi (70 kPa).

In order to connect your HS to a split/splitless or PSS injector, you must be familiar with the operation of your GC. The instructions provided below are specific to the AutoSystem XL or the Clarus 500. Refer to your AutoSystem XL User's Manual (P/N 09936073) or the Clarus 500 User's Guide (P/N 09936625) for detailed operational instructions. If you are using another GC, you must adapt the following instructions accordingly.

To configure your GC for split sampling (PPC only):

1. Configure the Split/Splitless injector to be used in the AutoSystem XL, or the Clarus 500 for "HS-40" operation.
2. In the AutoSystem XL or the Clarus 500 configuration, set the Split Flow Offset to zero for your injector.
3. In the GC method, set the Split Flow to at least 2 mL/min.

NOTE: Split flow refers to the amount of carrier gas supplied to the injector from the GC. A low split flow will result in less dilution of the injected headspace gas, but will not deliver enough carrier gas to the GC injector to support a pressure program. The split flow can be changed during a chromatographic run through the GC method timed events.

4. After you determine a value for P1, open the PPC tab and enter the carrier gas pressure (P1).
5. Check your GC manual for the optimal column head pressure of your analytical column. Adjust the split flow until the GC shows a pressure 10 kPa below the optimal column head pressure.
6. Open the carrier gas regulator on the GC and increase the pressure P_{GC}, until you reach the optimal column head pressure as read on the GC gauge or PPC display.

If you are using an instrument equipped with PPC with an AutoSystem XL or the Clarus 500 that also has PPC, observe the following guidelines when you are creating a pressure program.

- The pressure of the GC injector should not be programmed to a higher value than the HS sampling head pressure.
- You should disable the PPC alarm in the AutoSystem XL or the Clarus 500 when performing pressure programming for headspace sampling.
- Pressure, flow, or velocity programming requires the split flow in the GC method to be set 50 mL/min greater than the sum of the column flow + GC septum purge flow + HS needle vent flow.
- The split flow is the total flow added to the system by the AutoSystem XL or the Clarus 500 pneumatic module.
- To calculate the split flow value, use the final/highest column flow in the flow/pressure program. The septum purge flow is approximately 3 mL/min, the HS needle purge flow is approximately 15 mL/min when you are using Helium. To the sum of these values, add 50 mL/min to obtain the required split flow to be entered into the GC method.
- Slow or fast flow programming may require a lesser or greater split flow setting. To maintain proper flow programming rates it is important not to have leaks in the system. See *Leak Testing* in the *Installation* chapter for details on leak testing the HS sampling system.

During overlapping thermostatting the HS may pressurize a vial before the previous analysis has been completed. To ensure that all vials are pressurized to the correct pressure, always consider the pressurization time when calculating the GC cycle time for the HS, if you are performing any of the following:

- Pressure programming
- Constant flow or constant linear carrier gas velocity over an oven temperature program
- Flow programming
- High pressure sampling

During headspace injection, the split flow added to the GC injector dilutes the headspace sample. For example, an HS transfer line flow

of 25 mL/min and a GC split flow of 25 mL/min results in a 1:1 dilution or split of the original headspace sample concentration.

Since additional carrier gas is supplied from the HS, the actual flow through the GC split vent must be measured by an external flow meter. The flow through the split vent should be equal:

Actual Split Flow = Transfer Line Flow - Column Flow + Split Set point ***Equation 3***

Splitless Sampling

In the splitless sampling technique the sampling head pressure is used to pressurize the vial and to supply the GC column flow. This technique is recommended for the HS 40/110 trap but could also be used just with the headspace. For reproducible analytical results the sampling head pressure must be greater than the headspace pressure in the vial. During pressurization the pressure in the vial equals the sampling head pressure. During injection the sample vapor flows to the column. The rate of the pressure decrease in the sample vial depends on the carrier gas flow.

Any GC injector can be used for splitless sampling. The injector is used only to provide a mechanically stable, heated adapter for the transfer line. Splitless sampling configurations are generally simple to set up in principle. The sample is transferred directly to the GC column or to the HS 40/110 trap. Split ratios and GC flow rates are not required.

Splitless operation is necessary for cryofocusing. High pressure sampling can also be used with splitless sampling. Splitless sampling is less suitable for capillary columns that have a low pressure drop (short and/or wide bore).

Examples of the two methods of splitless sampling are given below. In the direct connection configuration, the transfer line and the analytical column are directly connected together, using a butt connector, inside the GC oven. For on-column sampling, the GC capillary column is connected directly to the HS sampling head through the heated transfer line.

There are however, some disadvantages to this sampling method if using this only with headspace. Less flow through the sampling area may lead to an increase in sample carry over and thus increased

relative standard deviation (RSD) and broader peak with small k^1. This is due to less efficient sweeping of the needle area.

It is also easier to service and troubleshoot, since the HS and GC can easily be separated pneumatically to be leak tested separately but plugging the column end.

This configuration also offers less flexibility if you need do the occasional liquid injections to the GC injector. The column must be disconnected from the HS sampling head before and the GC column re-connected to the injector.

Direct Connection: In the direct connection configuration, the transfer line and the analytical column are directly connected together, using a butt connector, inside the GC oven. Two types of butt connectors are available, press fit connectors which can only be used once, and standard low-dead volume unions, which can be reused.

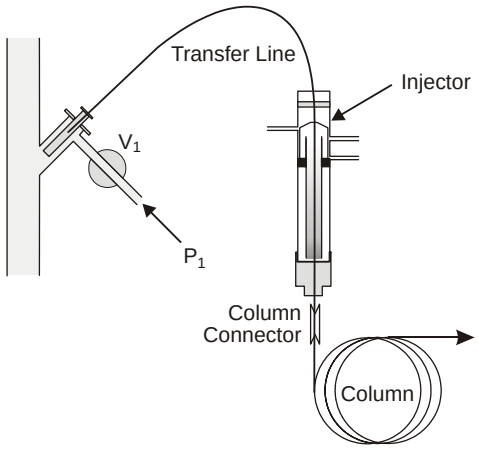


Figure 93 Splitless Sampling – Direct Connection

On-Column Connection: To perform on-column sampling, a 0.25 mm i.d. or 0.32 mm. i.d. capillary column can be connected directly to the HS needle area. Approximately 1.5 m of the capillary column is unwound from its holding cage and inserted from the GC oven, directly up through the GC injector and heated transfer line to the HS needle area. During operation, the temperature of the heated transfer line should not be allowed to exceed the maximum recommended temperature for the column stationary phase.

Splitless Sampling with the HS 40/110 Trap

HS 40/110 Trap Theory

The HS 40/110 trap uses a focusing trap (Pulsed Headspace Extraction or Trap) as its sampling operating mode. This approach uses a single or user specified number of pressurization and Trap Load steps to extract a high percentage of the headspace from the sample vial.

The HS40/110 Trap allows multiple headspace vapor loading steps into the cold trap, in order to remove from vial most of the headspace and so achieve a pre-concentration of the compounds of interest (up to 100 times lower detection limits).

An optional step after the last trap loading allows a cold dry purge of the trap to remove moisture and/or very volatile components.

Last, comes the desorption step of the pre-concentrated sample in the trap, by a fast increasing of the trap temperature and a simultaneous reverse carrier gas flow through the trap, in order to inject the sample to the transfer line and the following GC analytical column.

Throughout all of the preparative steps before desorption/injection, the transfer line and analytical column remain pneumatically isolated, constantly supplied with carrier gas through a pressure balance system (Isolation Flow).

In addition, TRAP CLEAN and TRAP TEST modes of operation offer trap cleaning as well as a trap cleaning quality test.

Built-in systems reveal possible sample leaks and/or not equal sample volumes and provide accurate calculation of the vial thermostatting temperature.

The Trap system is also supported by the Internal Standard option, that allows the addition to the sample, of selected gaseous internal standard components.

Another feature of the HS 40/110 trap is column isolation. This ensures GC/MS stability with the carrier gas flow into the GC being maintained during servicing of the headspace and trapping system

even when the headspace instrument is turned off. This feature is particularly useful when mass spectrometric (MS) detection is being used with the GC.

With the introduction of a trap, some modifications have been made to method development. The Method Editor has fields relating to the trap in each of the tab pages (Temperature, Times, Option and Pneumatics). See the *Operations* chapter of this guide for more details on modifying the Method Editor for the HS 40/110 trap.

Headspace Sampling

With Wide-Bore Capillary Columns

Wide-bore capillary columns with an I.D. of 0.53 mm generally do not have sufficient flow resistance for direct pneumatic headspace sampling. The sampling pressure must exceed the headspace pressure in the vial after thermostatting to avoid uncontrolled pre-injection.

If the column you are using requires a low inlet pressure, while the headspace sampling pressure must be kept at a higher level, you have two options:

When you are using a split/splitless injector in the GC, the required pressure drop can be established across the transfer line by setting a large split flow.

When you are using a packed column injector with no split function, or a direct connection from the transfer line to the column, the required pressure drop can be established across a flow restrictor attached to the outlet of the GC column. A restrictor is recommended between the wide bore column and the detector. The restrictor (P/N N9301354) has a 1.0 m x 0.18 mm I.D. A deactivated fused silica capillary is typical for this purpose.

Headspace Sampling

Using a Packed Column and a Packed Column Injector

Packed columns are normally used in splitless operation. They are typically installed onto a packed column injector, which has a separate flow controller for carrier gas.

To set up your headspace analysis using packed columns and a packed column injector:

1. Disconnect the HS heated transfer line from the packed column injector and replace the septum cap.

2. Make sure, the GC is released from external control.

3. From the AutoSystem XL keypad or the Clarus 500 touch screen, set the carrier gas flow to the level needed for your analysis, if you have PPC. If PPC is not installed on your GC adjust the flow controller to obtain the required head pressure.

4. From the AutoSystem XL keypad or the Clarus 500 touch screen, set the required oven temperature and allow the system to stabilize for one minute at the required oven temperature.

5. Press the Carrier Prog. button on the GC keypad for the AutoSystem XL or the Clarus 500 touch screen to access the carrier gas control display for the injector that that the HS 40/110n Trap is connected to. For the AutoSystem XL press the Arrow/Set key and read the GC injector pressure from the display. This is the carrier gas pressure required to obtain the required column flow. Make a note of this pressure for later use.

   On a manual system, read the column head pressure in the GC injector from the pressure gauge on the GC. This is the carrier gas pressure required to obtain the required column flow for the analysis. Record this pressure for later use.

6. Allow the GC oven to cool.

7. Adjust the carrier gas flow to the packed column injector to 2 - 5 mL/min (with the flow meter at the column).

NOTE: The bulk of the carrier gas is supplied from the HS. A small flow from the GC is required in order to purge the carrier gas line and the top part of the injector. This purge flow eliminates dead volume as well as back diffusion of headspace sample. It is an important precaution in order to avoid sample carry over and ghost peaks. A higher flow can be used, but it will result in dilution of the headspace sample.

8. Remove the septum cap from the packed column injector and connect the HS heated transfer line to the injector using the injector adapter. Take care not to break the fused silica transfer line. See the procedure *Installing the Heated Transfer Line at the GC Injector for Split Operation* in the **Installation** chapter.

9. Set the headspace carrier gas pressure (P1) to a value approximately 3 psi higher than the column pressure recorded in step #5. P1 must be higher than the headspace pressure in the thermostatted vial.

10. Increase the GC oven temperature to the operating temperature for the GC analysis.

11. Check that the pressure required for the operation of the column as recorded above in step #5 has been reached at the injector.

12. Adjust the HS carrier gas pressure further until the required column pressure (P2 + 3 psi) has been reached.

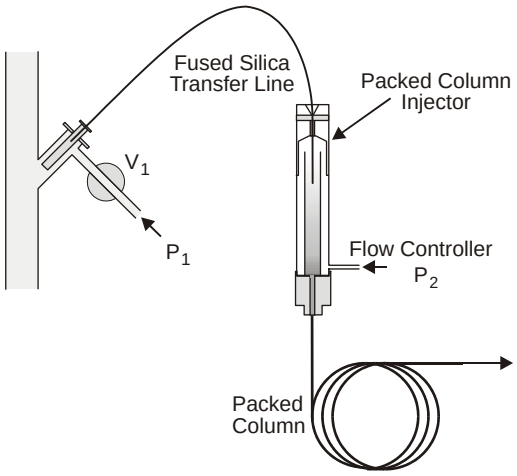


Figure 94 Sampling with Packed Columns

MHE Theory and Calculations (Headspace Only)

One way to do quantitative analysis using headspace is the Multiple Headspace Extraction (MHE) technique. This technique requires multiple extractions from the sample vial. The concentration of the volatile compound of interest is determined at each extraction step. Following each extraction, re-equilibration occurs between the two phases (liquid/solid and gas) in the sample vial, and although the concentration of the particular compound in both phases will be smaller than it was originally, their ratio (partition coefficient) remains the same.

In typical headspace analysis, it is not practical or possible for extractions from a sample vial to continue until all of the volatile compound is removed.

Using the MHE technique, however, it is unnecessary to perform extractions until all the volatile compound is exhausted, as the decrease in concentration of the compound in subsequent extraction steps follows the mathematical relationship of a first-order reaction. That is, the decreasing concentration over time is proportional to the prevailing concentration:

$$\frac{dC}{dt} = kC \qquad \textit{Equation 4}$$

where t is time, C is concentration, and k is a constant. Since the concentration at any time, C, depends on the initial concentration C_0 and the exponent k, the equation becomes:

$$C = C_0 e^{-kt} \qquad \textit{Equation 5}$$

Since the extractions are performed in steps, the time t may be replaced by the number of extraction steps, n. The initial concentration C0 is replaced by the peak area from the first extraction step, A1 which occurs at time t = 0 or n - 1. The constant k, which now includes instrument parameters, becomes k*:

$$A_n = A_1 e^{-k^*(n-1)} \qquad \textit{Equation 6}$$

This can be expressed in the form of a linear equation:

$$\ln A_n = -k^*(n-1) + \ln A_1 \qquad \textit{Equation 7}$$

A simple regression analysis provides a straight line plotted through the values of the peak areas derived from three or four extraction steps. This line gives the values for the slope (-k) and y intercept (A_0).

The total peak area of the volatile compound in the sample is then determined by summing the partial peak areas from each extraction step. Using Equation 7 as a geometric progression, the total area A_n becomes:

$$\sum A_n = A_0 (1 + e^{-k^*} + e^{-2k^*} + e^{-3k^*} \ldots) \qquad \textit{Equation 8}$$

Written in a more general form, the equation for the sum of the areas is:

$$\sum A_n = \frac{A_0}{1 - e^{-k*}} \qquad \textit{Equation 9}$$

Applying the values determined by the regression analysis, you will get at an extrapolated total peak area number which directly corresponds to the total amount of the compound present in the sample.

Once the total area for a particular component has been determined, its concentration in the sample can be calculated by using total peak areas derived from previous MHE analyses of a calibration standard[1].

1. PerkinElmer Inc. HS-40 Control Application (July 1990), p 31-32.

Routine Maintenance *6*

Introduction

This chapter describes the maintenance procedures that can be performed by the user.

WARNING

*Do **not** attempt to make adjustments, replacements or repairs to this instrument except as described in the accompanying user documentation.*

NOTE: This equipment requires no specified inspection or preventive maintenance to ensure the continuous functioning of its safety features.

The instrument is constructed with high quality components and requires little maintenance other than to keep it clean and free of dust. The HS does require some regular maintenance and replacement parts which will vary with usage. This will ensure optimum operating efficiency.

You should only perform the maintenance procedures described in this section.

NOTE: In most cases, the procedures in this chapter involve gaining access to internal parts of the instrument. It is therefore extremely important to heed all warnings regarding especially electrical and mechanical hazards. Carefully review the safety information in Chapter 1.

Before starting any maintenance:

- Switch off all the instruments in the system.
- Disconnect the instruments from the electrical supply.
- Allow hot parts of the instrument to cool down.
- Follow the maintenance instructions exactly as described in this manual.

Item/Operation	Frequency
Clean the Needle	Every 500 injections
Replace Needle Seal Assembly	Every 2500 injections
Replace o-ring seals	Up to 1500 injections
Change trap (HS 40/110 Trap Only)	As required
Run reproducibility test	As required
Clean the magazine	As required
Zero the PPC module	As required

Table 11 Maintenance Schedule

General Laboratory Cleanliness

Headspace Sampling and gas chromatography are very sensitive techniques. If proper precautions are not taken, the surrounding environment will contaminate your system.

NOTE: All users must be made aware of the circumstances that can lead to contamination of the system.

The degree to which precautions are necessary depends on the sensitivity required for your application. Trace level determinations at ppb or ppt levels require substantially more care than determinations at ppm levels. Background interference can be kept to a minimum if you take sensible precautions.

Cleaning and Decontamination

Before using any cleaning or decontamination methods except those specified by PerkinElmer, users should check with PerkinElmer that the proposed method will not damage the equipment.

Cleaning

In general, the instrument needs very little maintenance. You can clean the outside with a damp cloth with non-aggressive cleaning liquid. Other items that may need periodic cleaning:

Decontamination

If the instrument or an accessory requires decontamination before repair, maintenance, warranty or trade-in purposes at PerkinElmer, the responsible body should read the procedure and complete the certificate which is available on the PerkinElmer public website:

http://las.perkinelmer.com/OneSource/decontamination.htm

Follow the "Decontamination of Instrumentation and Associated Sub- assemblies" procedure and complete the "Certificate of Decontamination." The certificate is used to certify the decontamination process was completed before equipment can be returned to PerkinElmer.

Carrier Gas

The carrier gas is a major, potential source of contamination. Contamination can originate from the gas itself or from the tubing used to carry the gas. Always use high purity gases (≥99.999%).

NOTE: Appropriate filters should be placed in the carrier gas line close to the inlet of the HS to minimize the level of impurities in the carrier gas. Ensure that gas lines containing filters are protected from excess pressure as described in Gas Connections on page 75.

Tubing

Always use clean tubing, preferably made of copper or stainless steel, with the minimum possible number of joints. If necessary, clean the tubing by passing a stream of clean, inert gas through the tubing, while baking it in an oven at a temperature high enough to remove any trace organic solvents.

CAUTION	*Never clean carrier gas tubing with organic solvents. Any remaining traces of solvent will contaminate your system.*

Use compression fittings to make any joints in the tubing. Do not use soldered joints, especially if an ECD is to be used. The flux used in solder may contain a strongly electrophilic compound.

Sample Vials and Seals

CAUTION *Using sample vials, caps and septa other than those supplied by PerkinElmer may result in improper operation of the TurboMatrix Headspace or Trap Headspace Sampler. Damage to the instrument and/or loss of sample materials or data resulting from the use of sample vials, caps and septa* ***not*** *supplied by PerkinElmer may occur.* ***The subsequent service visit to remedy the situation, caused by the choice to use these non-PerkinElmer sample vials, caps and septa is not included under your warranty or service contract agreement.*** *Your Service Engineer can discuss the benefits of using only PerkinElmer sample vials, caps and septa.*

When samples have been collected, the vials should be analyzed as soon as possible.

WARNING *Using sample vials, caps and septa other than those supplied by PerkinElmer can result in damage to the instrument and/or injury if you attempt to remove the broken glass vials.*

Avoid storing vials that contain a sample in places where there are high levels of organic vapors, such as refrigerators, car trunks and airline storage hangars.

Store caps and septa in a clean environment (preferably a clean, heated desiccator) when they are not being used.

You may want to heat new septa to 100°C for 2 to 3 hours before putting them into use.

Important! Carrier Gas Shut Off

There are cases where you are instructed to switch off the carrier gas supply to the headspace.

However, in certain cases as in HS 40/110 trap the carrier gas should not be shut-off as the Isolation Flow (that protects the analytical column) will be stopped and you will starve your transfer line and detector of helium.

Under normal circumstances you can do all your maintenance and troubleshooting with the carrier gas on.

However in some cases you may be required to do maintenance that requires a complete shutdown of the carrier gas to prevent oxidation. You should **not** attempt maintenance in the Headspace or HS 40/110 trap unit, unless you have ensured the following in the GC:

1. If the detector is a flame one (FID, NPD, etc.), the flame should be off.
2. If the detector is an MS, the transfer line and ion source (if present) should be cool.
3. GC oven, injector and detector in the HS analytical column channel should be cool.

Following the above steps will protect you in case of being busy with the HS/TRAP maintenance or troubleshooting and you forget that the GC is still at a high temperature which will damage the column and possibly the transfer line if the carrier gas is cut off.

Reproducibility Test (Headspace)

Check the reproducibility of the HS sample injection system at regular intervals to ensure the reliability of your analysis results. To do this, create a method with the system parameters provided below. If you have already run this test, you simply need to load the saved method.

Run the test mixture ten times and save the results. The relative standard deviation for the peak heights, or peak areas, should be less than or equal to 1.8.

Test Column	Capillary Column, 25 m; 0.32 mm I.D. OV 101 (Permaphase DMS), 1 um	
Test Mixture	0.4% (v/v) ethanol in water (i.e. 1 mL ethanol in 250 mL water)	
Test Volume	2 mL test solution	
GC Parameters	Oven Temperature	Isothermal 60 ºC
	FID-Detector Sensitivity	High
	Detector Temperature	200 ºC
	Injector Temperature	120 ºC
	Carrier Gas	Helium
	Carrier Gas Pressure	100 kPa
	Split open	minus 0.2 min
	Split Flow	50 mL/min
	Analysis Time	2.5 min
Headspace Parameters	Vial	1-16, 1-40, 1-110
	Oven Temperature	60 ºC
	Needle Temperature	65 ºC
	Transfer Line Temperature	110 ºC
	GC Cycle Time	3 min
	Injections per Vial	1
	Thermostatting Time	22.0 min
	Pressurization Time	3.0 min
	Injection Time	0.04 min
	Withdrawal Time	0.5 min

Table 12 Capillary Column Parameters

Changing the Fuse

The fuses should normally not need to be changed. A blown fuse may indicate a more serious problem and you should contact your PerkinElmer service engineer unless you can identify a cause external to the instrument (incorrect line voltage, power surge, lightning, etc.).

The operating voltage of the instrument is set at the factory. Only a PerkinElmer service engineer or similarly qualified person is authorized to change it if required.

Before changing the fuse(s), observe the following precautions:

WARNING

To prevent potential injury to yourself and damage to the instrument, switch off all instruments in the system and disconnect them from the line power supply before you change any fuses.

To replace fuses:

1. Ensure that the power cord is disconnected from the power entry module on the rear left side of the instrument. The fuse drawer is located in the top portion of this module. Refer to *Figure 95*.
2. Carefully pry the fuse door open with a flathead screwdriver.
3. Use the flathead screwdriver to slide out the fuse drawer.
4. Remove the blown fuse and replace it with a new one of the correct type and rating. Consult the fuse chart. Both fuses must have the same rating.
5. Replace the drawer and close the fuse door.
6. Reconnect the line cord.
7. Resume normal instrument operation.

WARNING

For protection against fire hazard, replace only with the same type and rating of fuse.

Voltage @ 50/60 Hz	Fuse (250V rated)	Part Number
100	T 10A	M041-7002
120	T 10A	M041-7002
220	T 5A	M041-7038
230-240	T 5A	M041-7038

Table 13 HS Fuses

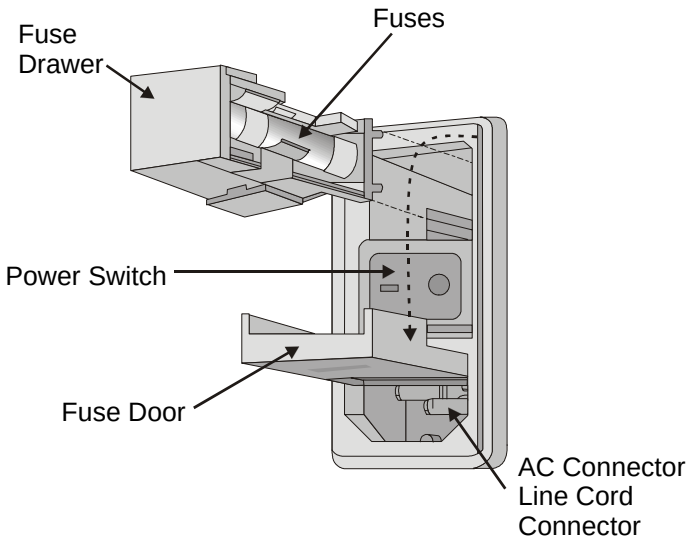


Figure 95 Replacing the Fuse

The Sampling Needle

Every 1500 injections, a maintenance display is shown indicating that the three o-ring seals on the needle need to be replaced. The injection interval can be changed depending on your application.

To acknowledge the message and clear the display, press the display. This display will be shown on two more occasions, and then again after the next interval of 2500 injections.

It is only necessary to change the sampling needle when it is damaged, or when you wish to change to another needle type. You may also want to replace the needle if you are changing applications. A platinum/iridium (Pt/Ir) needle is available for highly corrosive applications and Siltek® inert coated needle is available for reactive samples (this needle is shipped with the HS 40/110 trap).

Types of HS Needles

The jet needle can be used for both packed and capillary columns. Depending on the sample type, the jet needle is available in three different materials.

- Standard stainless steel needle (P/N B4000011). This needle has three grooves at the top of the needle to differentiate between the stainless steel needle and the Pt/Ir needle.
- Platinum/Iridium Needle for free volatile organic acids, bases and other corrosive compounds. The Pt/Ir needle has 4 grooves at the top of the needle (P/N B0510364).
- Siltek® inert coated needle (standard with the HS 40/110 trap)

If you have a manual pressure regulator the wide bore needles may also be used. Wide bore needles offer quicker pressurization of the sample vials.

Removing and Replacing the Needle (Headspace Only)

To change or replace the needle (see the following figure):

1. Turn off all heated zones by setting the temperature to 0. Allow approximately 30 minutes for the needle assembly and transfer line to cool down.
2. Once the system has cooled, switch off the instrument.

3. Turn off the gas supplies to the instrument.In the case of direct or on-column connections ensure that the flame is out if using an FID and that your oven/injectors and detectors are at room temperature.
4. Disconnect the instruments from the electrical supply.
5. Pull gently on the magnetic door release to open the door.
6. Loosen and remove the knurled needle nut.

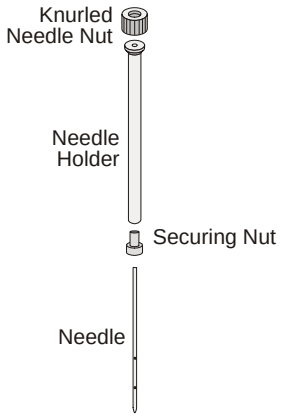


Figure 96 Changing the Sampling Needle

7. Lift the needle holder out of the rack.
8. Loosen the securing nut and pull the needle out.
9. Place the new needle in the needle holder nut. The top of the needle will butt up against the bottom of the needle holder.
10. Tighten the needle holder nut. The nut must be finger-tight.
11. Clean the needle with a lint free cloth or tissue. If necessary, dampen the cloth slightly with methanol.
12. Carefully slide the needle holder with the new needle back into the rack as far as the stop.

CAUTION	*Do not touch the lower half of the needle with your fingers.*

13. Rotate the needle so that the holes line up with the transfer line.
14. Securely hand-tighten the knurled nut.
15. Close the front panels and reconnect the instrument to the electrical supply.
16. Turn on the gases.
17. Power up the instrument.

Removing and Replacing the Needle (HS 40/ 110 Trap Only)

To change or replace the needle (see the previous figure):

1. Click on the **Tools** button. From the drop down menu select **Maintenance** and then select **Column Isolation Flow**. This will turn on the column isolation flow and your column will continue to get carrier gas while the needle is changed.
2. Pull gently on the magnetic door release to open the door.
3. Loosen and remove the knurled needle nut.
4. Lift the needle holder out of the rack.
5. Loosen the securing nut and pull the needle out.
6. Place the new needle in the needle holder nut. The top of the needle will butt up against the bottom of the needle holder.
7. Tighten the needle holder nut. The nut must be finger-tight.
8. Clean the needle with a lint free cloth or tissue. If necessary, dampen the cloth slightly with methanol.
9. Carefully slide the needle holder with the new needle back into the rack as far as the stop.

CAUTION	*Do not touch the lower half of the needle with your fingers.*

10. Rotate the needle so that the holes line up with the transfer line.

11. Securely hand-tighten the knurled nut.
12. Close the front panels and reconnect the instrument to the electrical supply.
13. Turn off the column isolation flow by pressing **Done** on the touch screen.

Cleaning the Jet Needle

Abraded sealing material from the vial septa may stick to the needle and can cause the needle to seal incorrectly during pressurization and withdrawal. A needle, coated with sealing material may also lead to unnecessary wear on the o-rings contained in the needle seal assemblies.

The recommended interval for cleaning the needle, will depend on the needle temperature and the type of septum being used. The minimum interval should be 500 injections. Avoid unnecessarily high needle temperatures. Usually 10 °C above the thermostatting temperature is sufficient.

To clean your jet needle:

1. Remove the needle as outlined in *Removing and Replacing the Needle (Headspace Only)* on page 243.
2. Clean the needle with a lint free cloth or tissue. If necessary, dampen the cloth slightly with methanol.

CAUTION	*Do not touch the lower half of the needle with your fingers.*

3. When you are wiping the needle, ensure that you do not force any material into the holes located on the side of the needle.
4. Blow filtered carrier gas through the needle.
5. Replace the needle assembly.

Changing the Upper Needle Seal Assembly

Check the needle seal assemblies (Part Number B0500833) every 1500 injections. Replace the o-rings and check the holders for damage. The seal assemblies (holders and o-rings) need to be replaced if they are scored or scratched on the top or bottom surfaces. The graphite-coated, Viton® o-rings (B0198110) should be changed every 1500 injections.

The special tools required to remove and replace the seals and o-rings are supplied in the shipping kit provided with the instrument.

To change the upper needle seal assembly:

1. Turn off all heated zones by setting the temperature to 0. Allow approximately 30 minutes for the needle assembly and transfer line to cool down.
2. Once the system has cooled, switch off the instrument.
3. Disconnect the instrument from the electrical supply.
4. Turn off the gas supplies to the instrument. In the case of direct or on-column connections ensure that the flame is out and that your oven/injectors and detectors are at room temperature.
5. Pull gently on the magnetic door release to open the door.

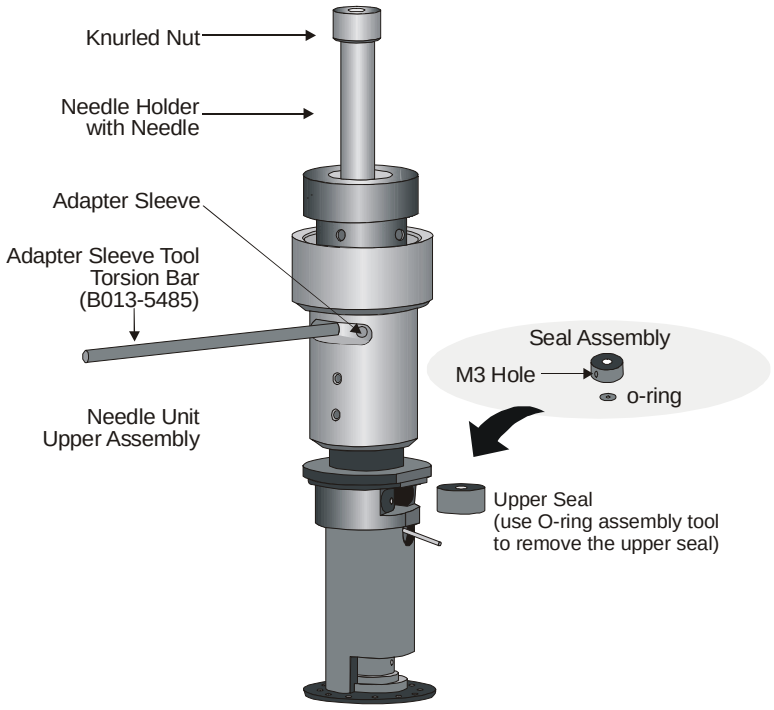


Figure 97 Changing the Upper Seal Assembly

6. Remove the needle holder and the needle as outlined in *Removing and Replacing the Needle (Headspace Only)* on page 243.
7. Blow filtered carrier gas through the needle.
8. Screw the O-ring assembly tool (P/N B013-1410) into the threaded hole (M3) of the upper needle seal assembly.
9. Loosen the adapter sleeve with the adapter sleeve tool (P/N M041-5330). You will gain access to the adapter sleeve through a window in the needle unit drive assembly. Use the tool to turn the adapter sleeve counterclockwise (left to right, facing instrument). This lifts the adapter sleeve up and allows you to remove the seal assembly.
10. Using the seal removal tool, gently pull out the needle seal assembly. If the seal assembly cannot be removed, then loosen adapter sleeve further.

11. Replace the o-ring. Refer to *Changing the O-Rings* on page 252. In the upper needle seal assembly there is only one O-ring located on the bottom of the seal assembly.

NOTE: Do not touch the new o-rings with your fingers. Use forceps or tweezers to remove the o-ring from its bag and place it on the seal assembly. If you touch the o-ring for any reason, throw it out and use a clean one.

12. Ensure that the correct needle seal assembly orientation is maintained. The side with the o-ring is placed down. The assembly must be correctly seated before securing it with the adapter sleeve.
13. Tighten the adapter sleeve. As you tighten the adapter sleeve the needle unit drive assembly will move the rack down. You will need to turn the rack counter clockwise to move it out of the way.

NOTE: Do not over-tighten the adapter sleeve as it will damage the seal assembly and cause it to leak.

14. Unscrew the seal removal tool from the threaded hole (M3) of the seal assembly.
15. Carefully slide the needle holder back into the rack as far as the stop.
16. Securely hand-tighten the knurled nut.

Changing the Lower Seal Assembly

The seal assemblies need to be replaced if they are scored or scratched on the top or bottom surfaces. The graphite-coated, Viton o-rings (B019-8110) should be changed every 1500 injections. The instructions below refer to items shown in *Figure 98.*

To change the lower seal assembly:

1. Turn off all heated zones by setting the temperature to 0. Allow approximately 30 minutes for the needle assembly and transfer line to cool down.
2. Once the system has cooled, switch off the instrument.
3. Disconnect the instrument from the electrical supply.

4. Pull gently on the magnetic door release of the front panel to open the door.
5. Remove the needle holder and the needle as outlined in *Removing and Replacing the Needle (Headspace Only)* on page 243.
6. Screw the seal removal tool into the threaded hole (M3) of the lower needle seal assembly. You will need to hold the removal tool as it will rotate once you begin to loosen the metal disc.
7. Place the spigot key into one of the holes in the metal disc.
8. Turn the metal disk clockwise (right to left when facing the instrument) to undo it as far as necessary to allow the lower seal assembly to be released.
9. Using the seal removal tool, gently pull out the needle seal assembly. Take note of the orientation of the seal assembly. You must retain this orientation.
10. Check the seal assembly and replace the o-rings as outlined in *Changing the O-Rings* on page 252. There are two o-rings in the lower needle seal assembly.

NOTE: Do not touch the new o-rings with your fingers. Use forceps or tweezers to remove the o-ring from its bag and place it on the seal assembly. If you touch the o-ring for any reason, throw it out and use a clean one.

11. Replace the seal assembly. Ensure the seal assembly is replaced in the same orientation as when it was removed. i.e. the same side must be facing up. Retain the seal removal tool in the seal assembly.

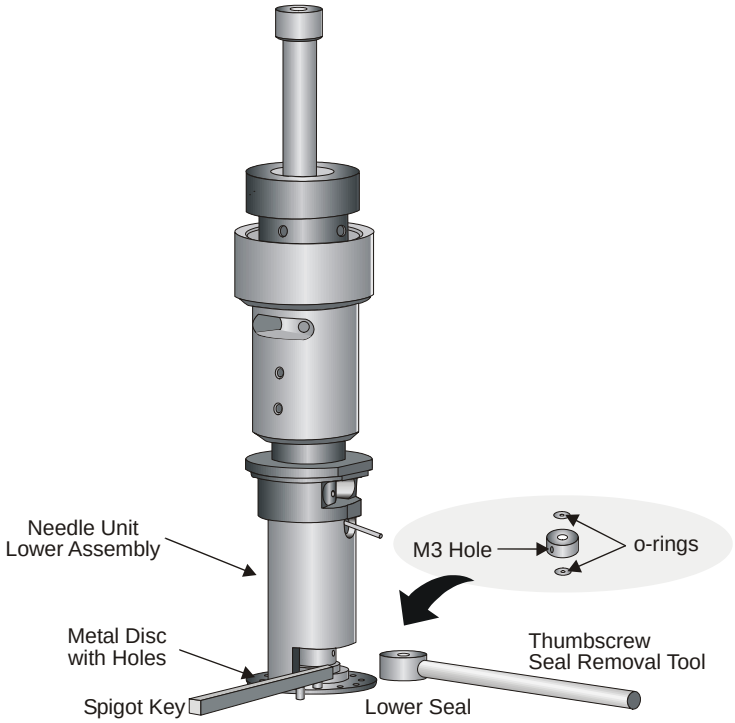


Figure 98 Changing the Lower Seal Assembly

12. Ensure that the needle seal assembly is correctly seated before securing it with the metal disc.Tighten the metal disk by hand enough to hold the seal assembly in place. Use the spigot key to turn the metal disk counter-clockwise.
13. Close the front panels and reconnect the instrument to the electrical supply.
14. Power up the instrument and turn on gases.
15. Perform a leak test to ensure the instrument is leak tight.

Changing the O-Rings

Normally it is not necessary to replace the whole needle seal assembly. In most cases it is only the o-rings that need to be changed. A special o-ring tool (B0147449) is supplied for removing and inserting the o-rings.

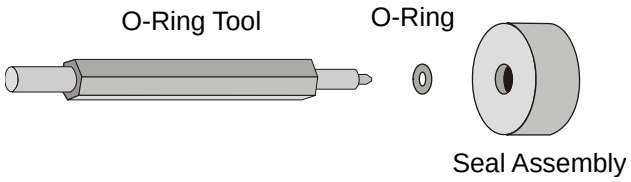


Figure 99 Changing the O-Ring Seals

To replace the o-ring:

1. Remove the needle seal assembly. See *Changing the Upper Needle Seal Assembly* on page 247 or *Changing the Lower Seal Assembly* on page 249.
2. Insert the narrow end of the o-ring tool into the o-ring.
3. Carefully press the tool to the side and remove the o-ring.
4. Take care not to damage the o-ring seat of the seal assembly.

NOTE: Do not touch the new o-rings with your fingers. Use forceps or tweezers to remove the o-ring from its bag and place it on the seal assembly. If you touch the o-ring for any reason, throw it out and use a clean one.

5. Place a replacement o-ring (P/N B0198110) into the seal assembly. The upper seal assembly contains one o-ring on the lower surface. The lower seal assembly contains two o-rings.
6. With the broader flat end of the o-ring tool press the o-ring into the seat. Take care not to damage the o-ring or the seat.
7. Replace the needle seal assembly. See *Changing the Upper Needle Seal Assembly* on page 247 or *Changing the Lower Seal Assembly* on page 249.

Changing the O-Ring in the HS 40/110 Trap

1. Turn off the instrument.
2. Check the conditions in the earlier section *Carrier Gas Shut Off* then shut off the gas supply.
3. Remove the knurled nut and the Dry Purge Assembly. See the following photo.
4. Using a 5/8 inch wrench, loosen the nut in the back by turning it 1/4 to 1/2 turn. See the following photo.

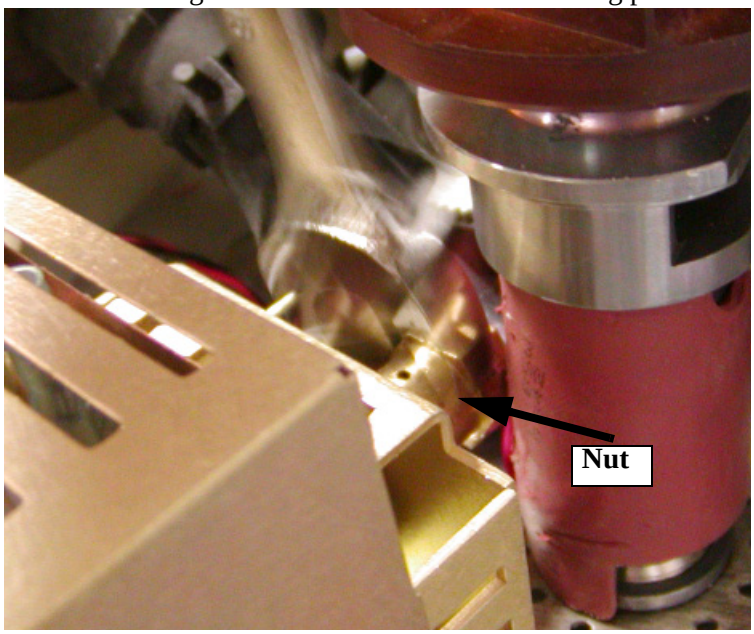


Figure 100

5. Use the plastic trap removal tool (P/N N6701077) to remove the trap. If you encounter any resistance loosen the nut, on the other side, some more. Twist the plastic removal tool and pull out slowly as you remove the trap.
6. Use a philips screw driver to loosen (but not remove) the three screws shown in the next photo.

NOTE: If the trap is broken or you cannot remove it easily you will also have to loosen the screws and then carefully remove the trap and any broken pieces.

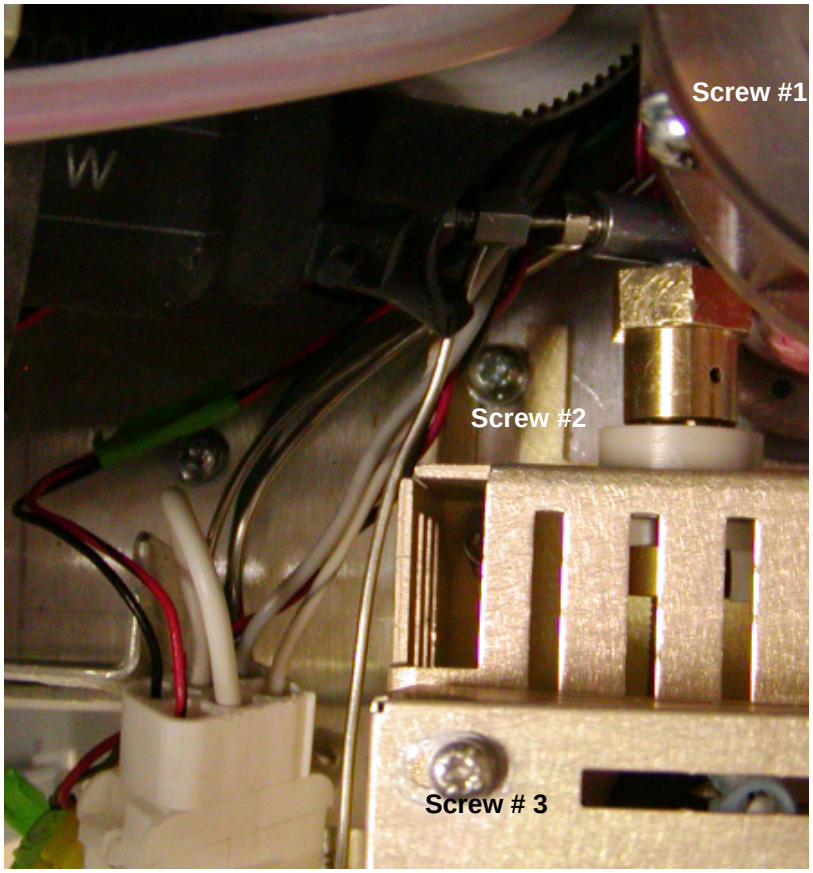


Figure 101

7. Slide the housing off and rest the housing on the carousel as shown in the next photo.

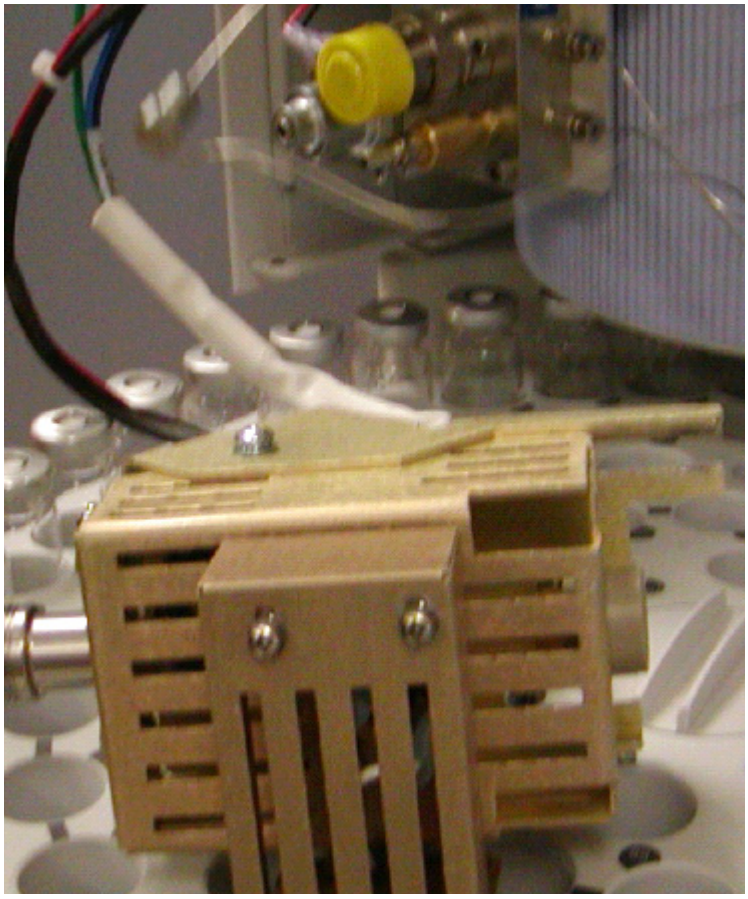


Figure 102

8. Remove the nut as shown in Figure 100. Use the Trap Alignment tool to remove the old O-ring. See the following photo for the location of the O-ring.

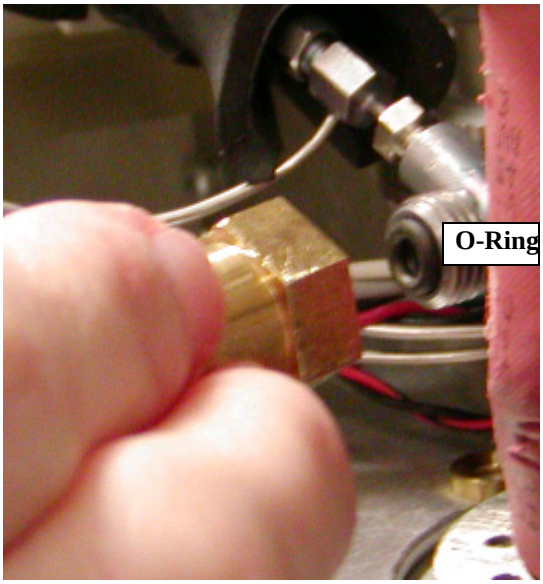


Figure 103

9. From the side of the instrument door take out the Trap Alignment tool. See the next photo.

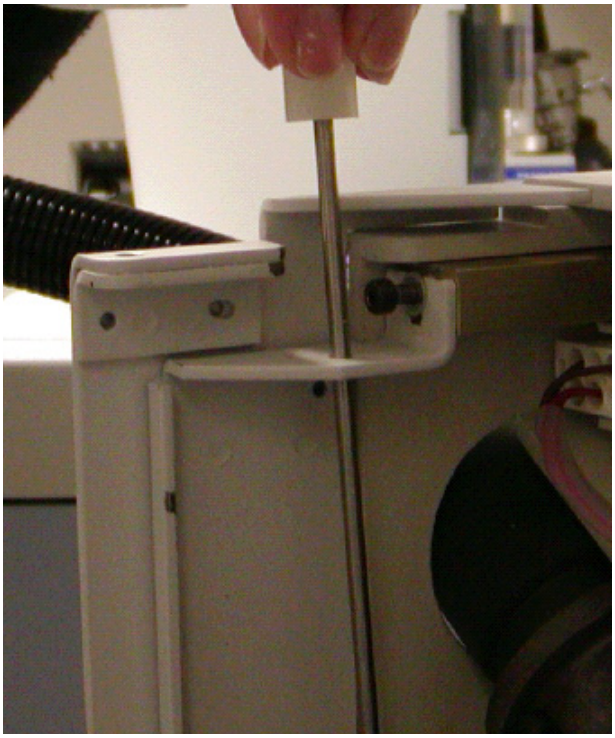


Figure 104

10. As shown in the following photo, use the tool to slide on and loosely screw on the brass fitting over the new O-ring. Do not over tighten. You will notice that as you push in the tool, the O-ring will offer a slight resistance. This is supposed to happen. Remove the trap alignment tool.

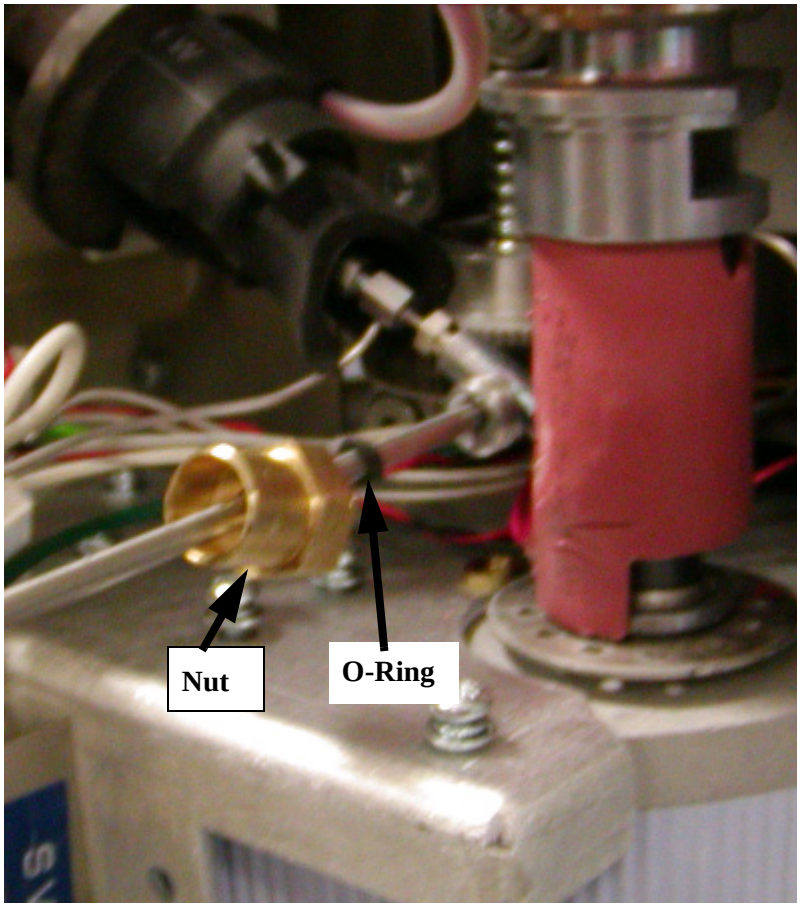


Figure 105

NOTE: If you are using only the TurboMatrix Headspace mode you do not need to install the trap. Use the brass button, shipped with the instrument, to seal the trap area from the analytical path.

11. Replace the housing, that is laying on the tray, and tighten the screws in the housing. Tighten the housing just enough that it is secure but you can still move it slightly.
12. Insert the Trap Alignment tool back into the housing.The tool will refit the O-ring pack into place.
13. With the tool pushed in there will be a gap between the housing and the tool. With the tool still in place tighten

the three screws. If the tool is not sliding in well, you may need to tighten the screws in the front and side of the housing.

NOTE: As you are pushing in the Trap Alignment tool, you should feel it move in slowly with only a slight resistance.

14. Use the plastic trap removal tool (P/N N6701077) to reinsert the trap.With the trap in the plastic tool, slowly twist the tool as you insert it.
15. Take the Trap Alignment tool out and use it to push the trap in. See the next photo.

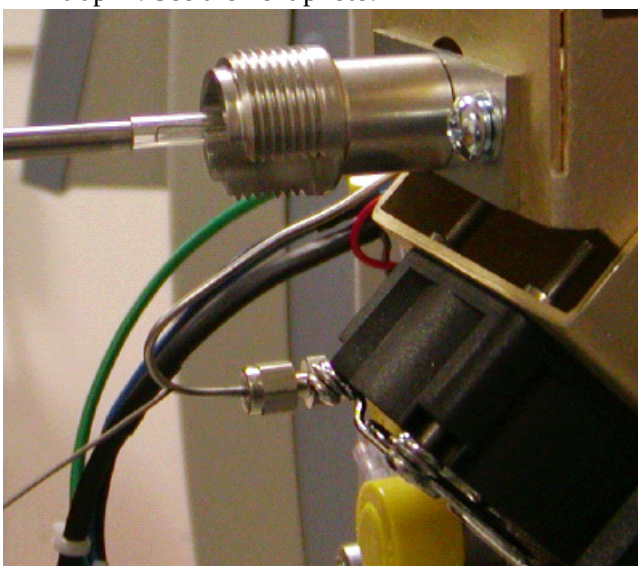


Figure 106

16. Carefully continue pushing in the trap with the Trap Alignment tool. Push in until it is about 1/2 mm flush with the front surface. See the next photo.

NOTE: If you cannot push in the trap enough to have it 1/2 mm flush with the front surface, the trap is not positioned properly. You will need to redo this procedure.

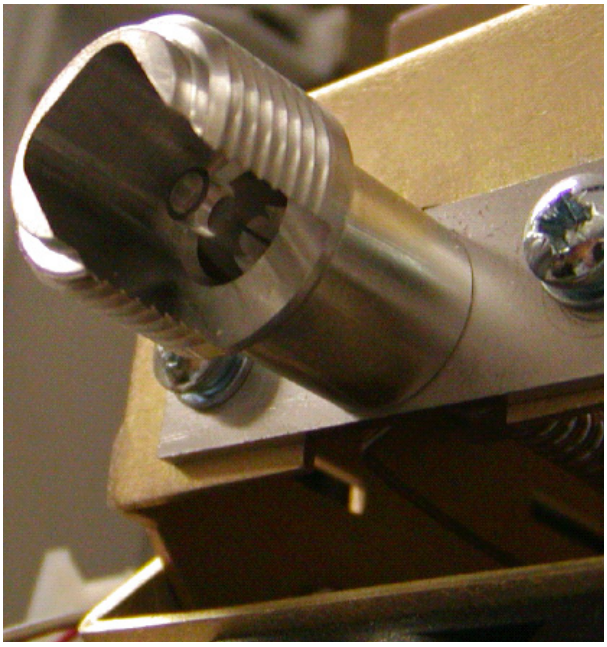


Figure 107

17. Tighten the back nut until it stops.
18. Return the Trap Alignment tool to its holder on the side of the instrument door.
19. Re-attach the Dry Purge Assembly and knurled nut.

Converting the HS 40/110 Trap to a TurboMatrix Headspace Mode

To convert the HS 40/110 trap to use for a standard headspace see the section later in this chapter *HS 40/110 Trap Maintenance; Removing and Replacing the Trap* and use the solid end trap (P/N N6701170) other than the trap that was shipped with the instrument.

Once you have done this, you must go to the touch screen into the Method and change the Operating Mode to **Constant**.

CAUTION	*Once you have converted your HS 40/110 trap to a TurboMatrix Headspace mode do **not** run the system in Trap mode. The plastic retainer will melt at the 400 °C temperatures for the trap methods. A melted plastic retainer will result in serious damage to the instrument.*

Leak Testing the Sample Injection System

If a leak is occurring and you cannot isolate the source, it is suggested that you separate the HS from the GC and leak test each instrument separately. Once each instrument is leak tight you can then connect them and test them together.

The automated leak test will leak test the sampling system. You must plug the end of the fused silica transfer line or if the fused silica line has been connected directly to the GC column then plug the outlet of the GC column.

To leak test the sample injection system:

NOTE: If you use a split/splitless injector, the split and purge outlet must be closed for the leak test. If you intend to leak test only the HS remove the fused silica line from the injector and plug the fused silica line with a septum.

1. Turn off all heated zones by setting the temperature to 0. Allow approximately 30 minutes for the needle assembly and transfer line to cool down.In the case of direct or on-column connections ensure that the flame is out and that your oven/injectors and detectors are at

room temperature.

2. Once the system has cooled, switch off the instrument.
3. Disconnect the instrument from the electrical supply.
4. Undo and remove the chromatographic column at the detector inlet.
5. Use a blanking plug to seal the detector column fittings. Seal capillary columns with a new, clean septum.
6. Switch the HS on. Open the Temperature tab and reduce the set points to ambient or lower.

NOTE: It may be necessary to touch some of the fittings if the leak test fails so set the temperature of the needle and transfer line to ambient or lower. If you enter a value of 0 the heaters are shut off.

7. Set the carrier pressure to 45 psi.
8. If using a headspace trap close the desorb slide in front of the instrument
9. When the instrument is in the **Standby** mode, open the **Tools** menu and select **Maintenance**. Then select **Leak Test**.
10. The HS sampling system is now a closed, pressurized system. The pressure, displayed on the PPC tab, must not drop by more than 1 psi over a period of 40 seconds.
11. If a leak is detected, check all of the connections with a helium Leak Hunter or concentrated ethanol and water solution. Once you have checked and tightened all of the connections, then run the leak test again.
12. Reduce the carrier pressure to the normal method pressure.
13. Unplug the fused silica line and ensure that there is no septum material blocking the fused silica line.

If you are leak testing the HS sampling system you should check the following connections first:

- O-rings in the upper and lower needle sealing elements.
- Transfer line connection to the needle unit.

- Column connection at the injector outlet (or to a transfer line in case of a direct connection).
- Sample vial closure (old septa, caps not crimped correctly)
- Trap fittings

If you are testing the whole chromatographic system, leakage may be occurring at the GC connections.

NOTE: Ensure the HS sampling system is leak tight before connecting the transfer line to the GC.

Refer to the GC manual to leak test the injector and detector connections. The following list provides further locations to test the GC connections:

- The connection of the heated transfer line with the GC injector (septum).
- Column connection at the injector outlet (or to a transfer line in case of a direct connection).

Leak Testing the HS 40/110 Trap

You must leak test all the connections for the GC as outlined in this procedure. You must also leak test the trap, the fittings that connect the trap to the transfer line and the fittings inside the HS 40/110 trap to determine that there are no helium leaks.

1. Let the GC cool down. Cool the headspace transfer line.
2. Undo and remove the chromatographic column at the detector inlet or inside the GC oven at the transfer line connection.
3. Use a blanking plug to seal the detector column fittings or the transfer line outlet. Seal capillary column or the transfer line with a new, clean septum.
4. Open the Headspace front door and locate the desorb outlet (the brass fitting). Close the desorb flow by sliding the fitting in (to open the fitting slide the fitting out).

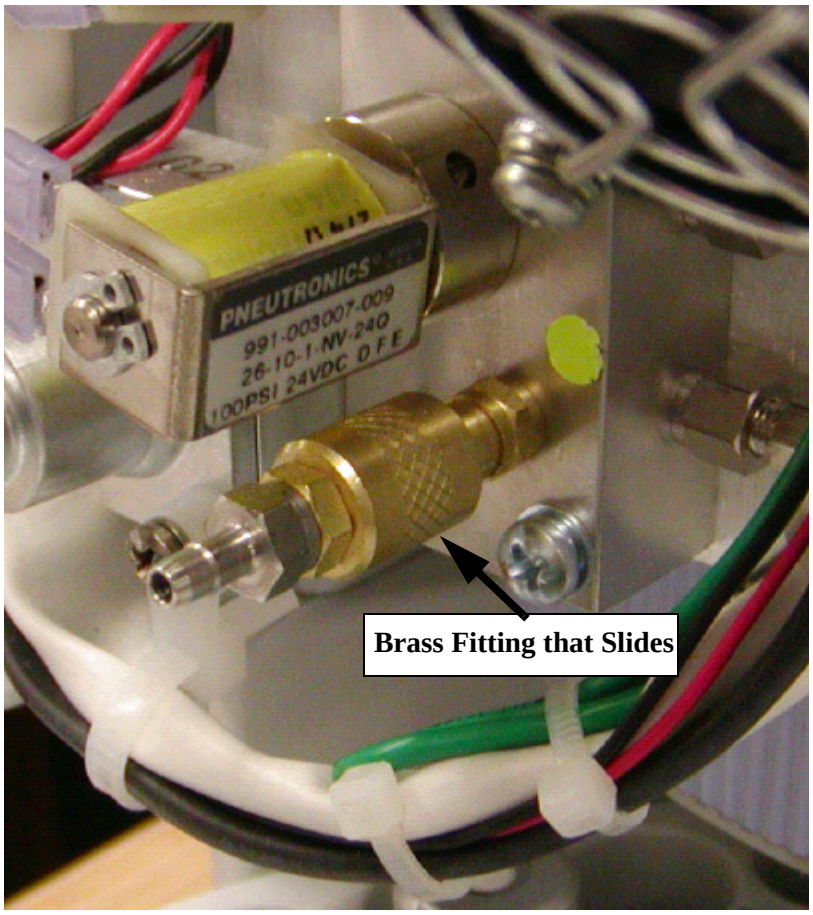


Figure 108

5. Set the carrier pressure to 45 psi. See *Setting the Carrier Gas* in the *Installation* chapter.
6. Open the Tools drop down menu and select **Maintenance** and then select **Leak Test**. If the leak test fails you will get the following screen (see the following figure). Go to step 8 for the procedure for a failed leak test.

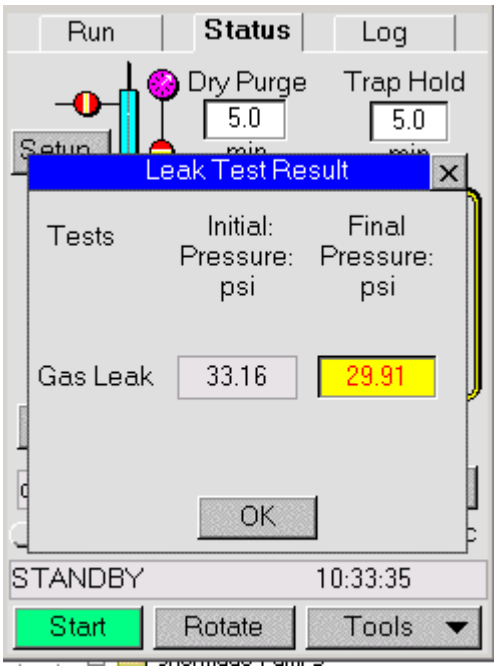


Figure 109

If the leak test passes you will get the following screen (see the following figure):

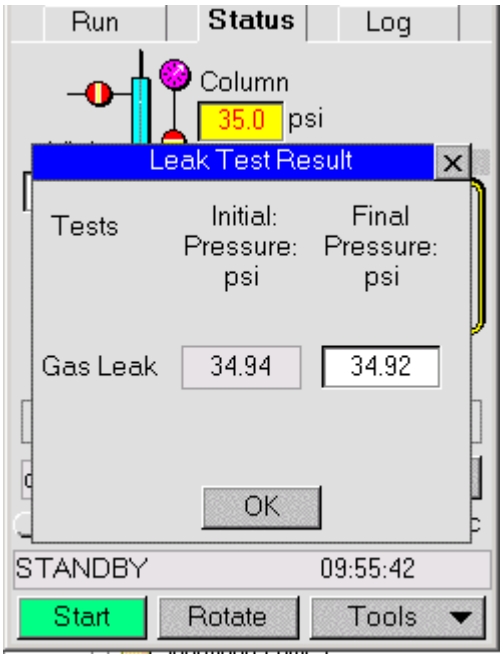


Figure 110

7. The HS sampling system is now a closed, pressurized system. The pressure, displayed on the PPC tab, must not drop by more than 1 psi over a period of 40 seconds.
8. If a leak is detected, check all of the connections with a helium Leak Hunter or concentrated ethanol and water solution. Once you have checked and no leaks are detected then run the leak test again.
9. Reduce the carrier pressure to the normal method pressure.
10. Because of the sensitivity of the HS trap make sure to trim the column where you plugged the end with a septum. Wear gloves when you trim the column.
11. Reopen the desorb (slide) flow path when finished, by pulling the brass fitting out.

If you are leak testing the HS sampling system and it has failed the leak test, you should check the following connections first:

- Headspace O-rings in the upper and lower needle sealing

elements.

- Transfer line connection to the needle unit.
- Leak check all nuts around the trap.
- Be sure the trap is not cracked or broken.
- Check flow valve connection.

NOTE: During shipping the valves may become loose and start to leak. If this happens, please go the next procedure, ***HS 40/ 110 Trap Valve Leak Test****.*

HS 40/110 Trap: Valve Leak Test

CAUTION	*When you run this test there will be no carrier gas going into the transfer line. In case of high flow rates, make sure that any hot zones (i.e. the GC oven, transfer line) are cooled to prevent damage to the instrument.*

NOTE: Use this procedure as a diagnostic tool only if you are having problems.

This leak test is limited to the plumbing area between the PPC module exit SV1a and the inlets of the solenoid valves SV1 and SV9. See following figure.

It does not involve the trap, the Sampling Head and Needle, and their plumbing. Nor does it involve the transfer line and the GC column.

As you can see in the following figure, all three solenoid valves, SV1a, SV1 and SV9 are closed. In this way this plumbing area is closed and pressurized.

As shown in the following figure, Isolation Flow to the transfer line and the GC column is shut off (SV8 is vented to atmosphere). If you are using high flow rates:

Be sure to protect the GC column and the detector by:

- If an MS detector is used in this channel ensure that the filament is switched off.
- If no carrier gas is supplied to the HS Transfer Line, cool it down if using a transfer line with a stationary phase.

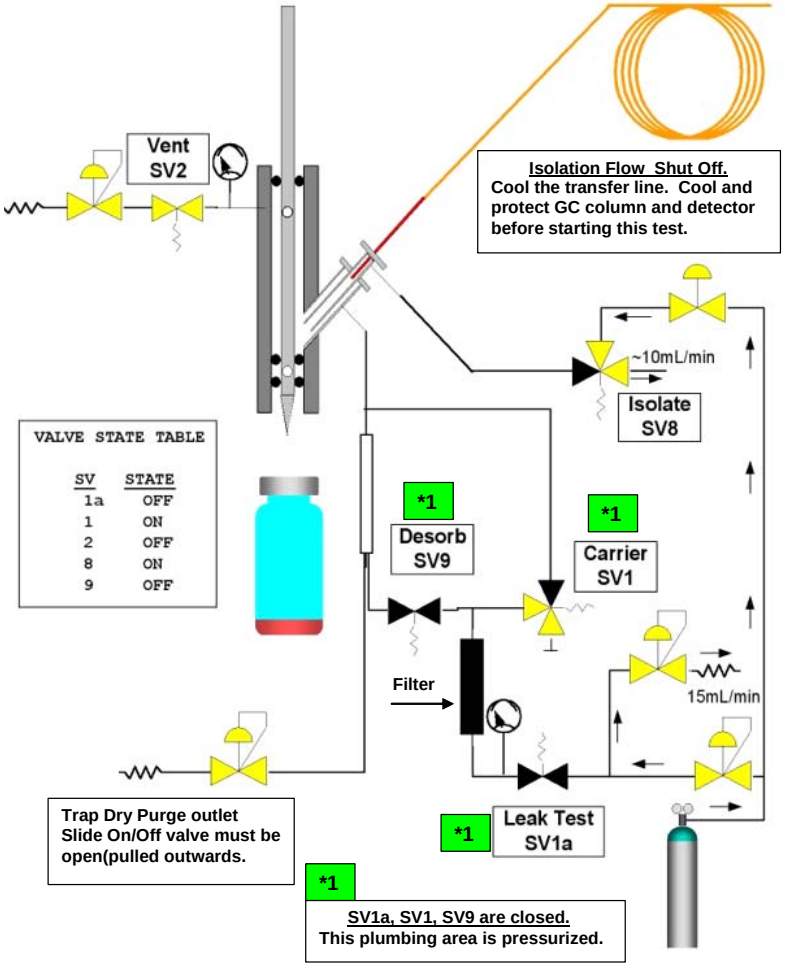


Figure 111 Valve Leak Test

During this Valve Leak Test the Needle Purge SV2 is open.

The slide On/Off valve at the Trap Dry Purge outlet must be open (knurled sleeve pulled all the way to the front).

To start a Valve Leak Test:

1. Set the carrier pressure at **45 psi**, in the Column field of the Option tab.
2. Open the Tools drop down menu, select **Maintenance** and then select **Valve Leak Test**.
3. Next, touch the **OK** button in **Start the Leak Test now?** window.

This part of the plumbing is now a closed, pressurized system. The pressure, displayed on the PPC tab, must not drop by more than 1 psi over a period of 40 seconds.

On test completion, the system displays the Leak Test Results screen.

4. If the first Valve Leak Test fails, repeat the test 3 more times. If these additional tests come out successfully/ pass, ignore the first one.
   However, if these tests fail, a leak is detected. Check all of the connections between SV1a and the inlets to SV1 and SV9 with a helium Leak Hunter or a concentrated ethanol and water (50:50) solution.

The most suspect parts in this area are SV1 or SV9 or the filter connections.

However, most of this plumbing area is not accessible to the operator. A PerkinElmer service engineer should be called in. For more details, see the Leak Troubleshooting section in the Troubleshooting chapter of this manual.

On completion of this check, return the system to Standby and set the Transfer line temperature to its method value.

Magazine Maintenance

Removing and Replacing the Magazine

The magazine on the HS 40 and the HS 110 is removable. You can maintain multiple magazines so that while one is in use, you can be

loading the other. The magazine on the HS-16 cannot be removed by the user.

To remove and replace the magazine:

1. Turn the HS off.
2. With one hand holding the magazine, loosen the magazine cap nut by turning it counter clockwise.
3. Remove the cap nut and then lift off the magazine.
4. Place the new magazine on the instrument and replace the magazine cap.
5. Turn on the HS. The instrument will initialize the motors. It will then return a **Ready** status if all of its systems have been initialized correctly.

NOTE: You can clean the magazine using warm soap and water. Do not immerse the magazine in water, simply use a damp cloth and wipe plastic. Do not use organic solvents to clean the magazine as you will damage the plastic.

Cleaning the Magazine

If dirt is allowed to build up on the magazine, the sample vials may stick and will not be loaded correctly. To prevent a build-up of contamination on the magazine, only load clean sample vials. Never use adhesive tape or any other type of adhesive materials on the vials.

The magazine should be cleaned every six months or more frequently if build-up is obvious.

With the HS disconnected from line power, clean each well of the magazine with a weak soap solution or suitable solvent using a cotton swab. Rinse with clean water using a fresh cotton swab and allow the magazine to dry.

CAUTION	*Do not use organic solvents to clean the magazine as it will damage the plastic.*

Adjusting the Hand Crimper

Adjusting the Stop Pin

An adjustable stop pin is located on the lower arm of the hand crimper.

To adjust the hand crimper:

1. Loosen the lock nut to allow adjustment of the pin.
2. If the crimper is not crimping firmly enough then shorten the stop pin by tightening it several turns until a satisfactory position is set.
3. If the crimper is crimping too tightly then lengthen the pin by loosening it several turns.
4. After you make the required adjustment to the crimper, seal a few test vials to ensure that the crimper is working correctly.

NOTE: An additional test to confirm good seals is to put several drops of a volatile solvent (i.e. acetone) into a vial. Seal it and, using tongs, immerse the vial into a beaker of boiling water. If no bubbling is seen around the crimper seal the crimper has been adjusted correctly.

5. Finally, retighten the lock nut.

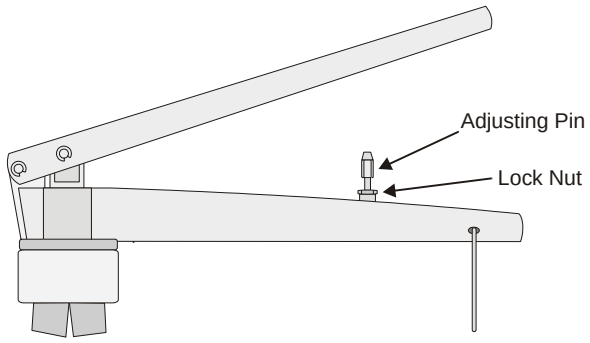


Figure 112 Hand Crimper Locking Nut

Adjusting the Crimp Plunger

If the stop pin cannot be adjusted further, but the hand crimper is still not crimping correctly, then you need to alter the position of the crimp plunger.

To adjust the crimp plunger:

1. Using circlip pliers, or round-nose pliers, remove the circlip from the securing pin on the crimp lever.

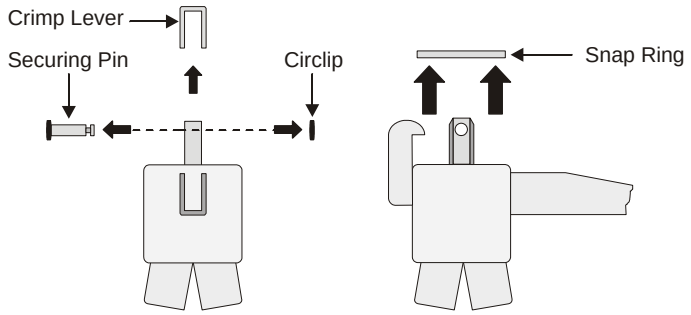


Figure 113 Removing the Circlip and Snap Ring

2. Pull out the pin.
3. Remove the lever.
4. Using the circlip pliers, or round-nose pliers, remove the snap ring from the top of the crimper.
5. Press the four jaws together and pull out the stamper assembly.
6. Clamp the flange of the stamper in a vice at the position shown in *Figure 114*.
7. Place a suitable metal rod or screwdriver through the hole on the plunger.
8. Screw the plunger up or down with enough force to break the seal. When the crimper is new, the stamper is sealed with a thread sealant to prevent inadvertent movement.

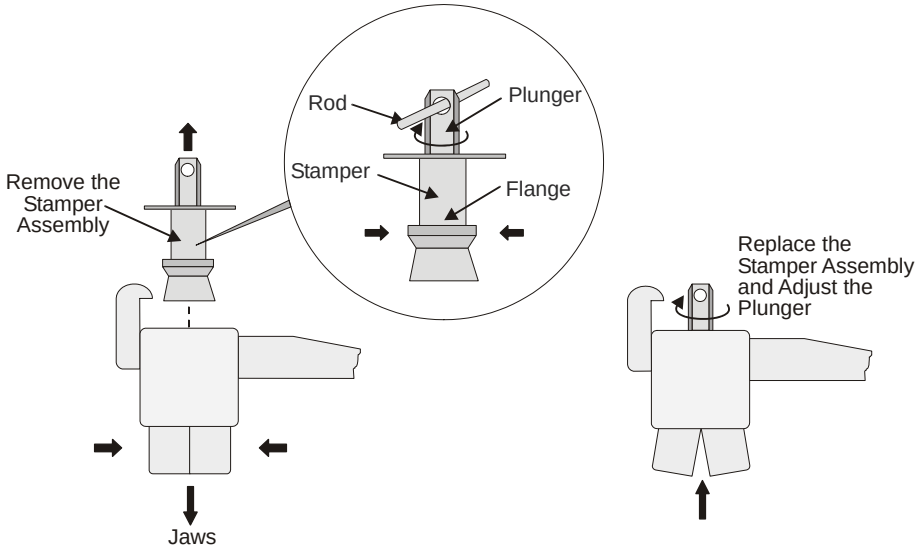


Figure 114 Stamper Assembly

9. Carefully place the stamper assembly back into the crimper.
10. Press against the stamper from below, using your thumb to keep it from rotating.
11. Screw the plunger up or down to obtain the required pressure. Screwing the plunger up increases the crimping pressure. Screwing the plunger down decreases the crimping pressure.
12. Replace the crimp lever and the pin, but not the snap ring or circlip.
13. Seal an empty vial with a septum, star spring and crimp cap and check the closure carefully to ensure that it has been sealed correctly.
14. Repeat steps 10 to 13 until a perfect seal is obtained.
15. Seal the plunger into the stamper with a thread sealant.
16. Reassemble the hand crimper.

Decapping the Vials

Some chemicals may be hazardous or may become hazardous after completion of an analysis. Do not store, handle, or work with any chemicals or hazardous materials unless you have received appropriate safety training and have read and understood all related Material Safety Data Sheets (MSDS). Use, store, and dispose of chemicals that you require for your analyses in accordance with the manufacturer's recommendations and local safety regulations. You must comply with all federal, state, and local laws related to chemical storage, handling, and disposal.

You must work under a suitable hood when handling and mixing certain chemicals. The room in which you work must have proper ventilation and a waste collection system. Always wear appropriate safety attire (full-length laboratory coat, protective glasses, gloves, etc.), as indicated on Material Safety Data Sheets.

Once the vials have cooled, you can decap them. The caps cannot be reused. The vials should only be re-used if they can be cleaned reliably and if the vial is not damaged in any way.

To decap the vials:

1. Hold the vial well away from your face. Depending on the nature of the sample, it may be necessary to decap the vials in a fume hood.
2. Grip the cap with the cap removal tool (P/N N9301270) and twist your wrist.
3. The cap will come off easily, if it has been crimped correctly.

If the materials being sampled are hazardous in any way, you must treat the collected samples, and the vials that contained them, as hazardous waste. Used vials and seals may contain small amounts of the substances that were analyzed and may thus constitute a chemical or biological hazard. Refer to your local safety regulations for proper disposal procedures.

Installing the Transfer Line Cap

The transfer line cap is used to protect the fused silica column when the transfer line is not connected to the GC.

Connect the cap to the transfer line once the heated transfer line has been removed from the GC. The cap simply screws into the knurled nut of the transfer line.

When you connect the transfer line to the GC remove the cap and store it in a safe place away from volatile organic compounds.

Zeroing the Carrier Gas PPC Module

The PPC module must be calibrated when you change the type of carrier gas being used.

To calibrate the module:

1. Open the Tools menu and select Preferences. Select the Config tab.
2. Use the drop-down box to select the type of carrier gas being used. You must use the same type and quality of carrier gas for the HS that you are using for your GC
3. Press Calibrate Sensor bar -> PPC Calibration bar, -> Zero.
4. Disconnect the carrier gas to the Carrier In port.
5. Press the Zero button. The PPC module sets its zero point.
6. Once you have completed the operation, the date will be entered for future reference.
7. Press OK to accept the settings and close the tab.
8. Reconnect the carrier gas to the carrier in port and set to 90 psi.

See PPC Tab in the Method Development chapter for details on configuring the PPC module and creating methods that utilize the PPC options.

HS 40/110 Trap Maintenance

The HS 40/110 trap differs from the other headspace samplers in that it also has a solid end trap (P/N N6701170). The following sections review the unique maintenance issues for the trap.

Removing and Replacing the Trap

After prolonged use, the packing in the trap may become contaminated, begin to decompose, or shift. This will cause the retentive properties of the trap to diminish, indicated by decreased output when a standard test sample is analyzed, or by skewed peaks. If in doubt, replace the trap with a new one, replacing all ferrules at the same time.

WARNING *Wear gloves when you are handling the trap. Handle the trap with great care since it is made from quartz and can easily break.*

NOTE: Before proceeding always have one or two spare traps on hand in case of breakage.

NOTE: Turn off the unit and Column Isolation will be automatically applied.

CAUTION ***Do not over tighten the fittings.*** *Only tighten the trap fittings enough to prevent leakage. The quartz tube is fragile.*

NOTE: When installing the trap in the instrument for the first time or you must take the trap assembly apart, you must do an alignment procedure. See the next procedure, ***Trap Breaks Inside the Trap Assembly,*** *for the steps on aligning the trap. If you are just removing an old trap and replacing it with a new trap you do* ***not*** *need to do an alignment procedure.*

To remove the trap:

1. Turn off and allow the system to cool.

WARNING

The vial oven and the trap enclosure may be very hot. Allow 20 -30 minutes for these parts to cool.

2. Remove the looks cover by opening it and taking the cover off the hinges by lifting it straight up. Turn off the HS40/110 trap or start the column isolation flow. This will enable gas flow to continue to the GC but the trap will be blocked off.
3. Remove the large thumb screw and put it in a secure location.
4. Pull out the dry purge assembly (P/N N6700112). See the following photo.

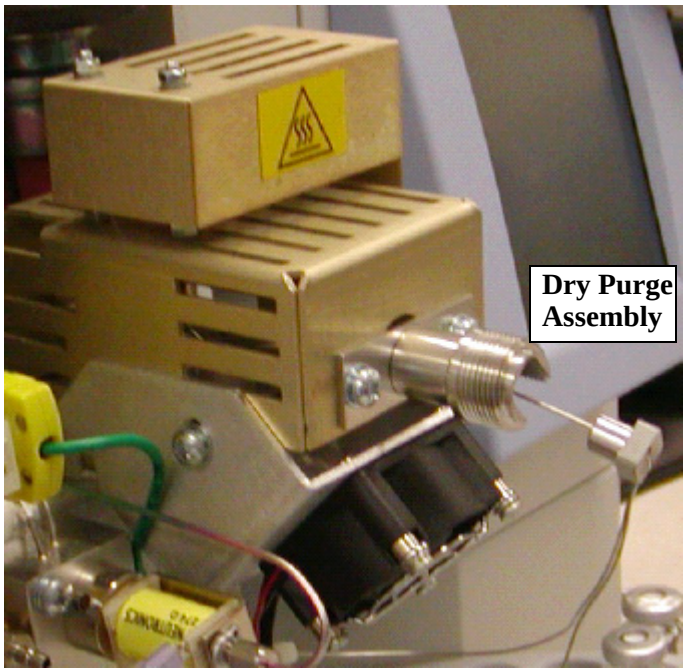


Figure 115

5. Carefully loosen the nut in the back of the trap assembly.Turn it 1/4 to 1/2 turn only since if you loosen

it too much the O-ring inside will be out of alignment and difficult to reinstall causing leaking. See the following figure.

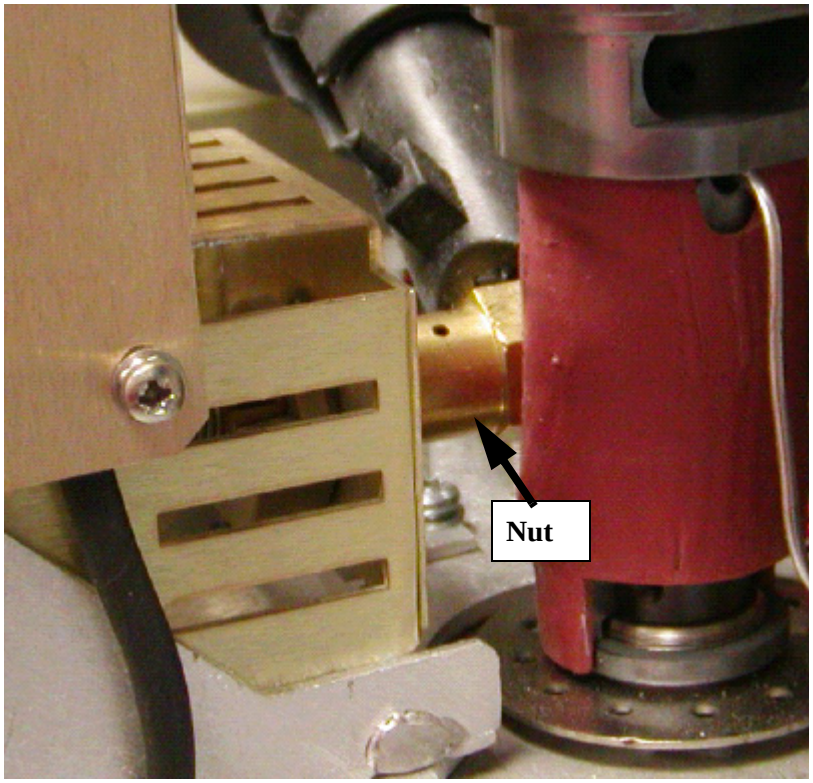


Figure 116

6. Use the trap removal tool (P/N N6701077) to carefully remove the trap. See the following photo.

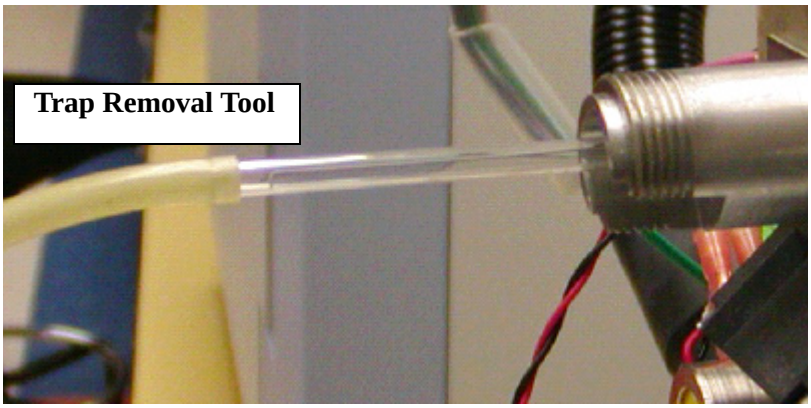


Figure 117

7. Wearing gloves, carefully remove the new trap from the box and insert it into the trap housing.

Figure 118

8. Put the ferrule on the trap (the tapered edge must face towards the front) at the front of the HS unit. See the following photo.

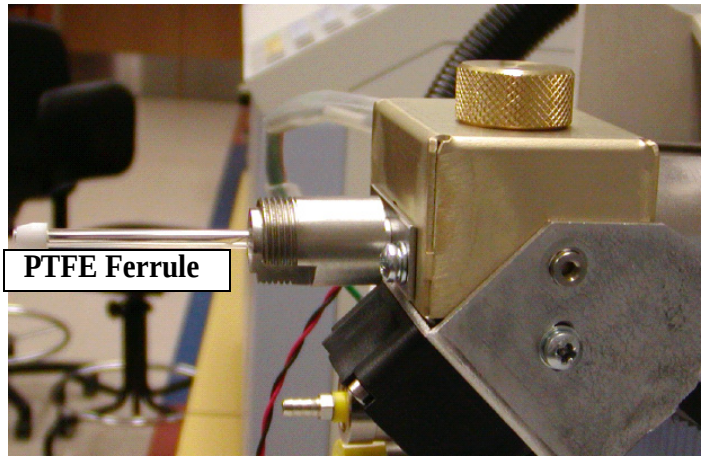


Figure 119

9. By hand gently push the new trap in as far as you can.

10. Use the Alignment Rod (P/N N6700122) to gently push the trap into the O-ring into the proper position. You will feel a small pop as the trap goes through the O-ring and is seated properly in the trap housing.
11. Retighten the back nut until it stops.
12. Inspect the dry purge assembly to see that it is not damaged. Feed the dry purge assembly into the trap until it stops.

NOTE: If the dry purge assembly is damaged see this chapter for information on replacing this assembly.

13. Reinstall the trap housing. Do not use any tools (only fingertighten) to tighten the thumb screw since it will damage the ferrule.
14. The trap must be conditioned (see the Routine Maintenance chapter for this procedure) before analytical use by establishing carrier gas flows and heating the trap several times to remove any volatile impurities from the trap packing.

If you heat the trap to high temperatures take care that the analytes do not degrade at these high temperature. For example, if the halogenated hydrocarbons are present in the sample, the temperature should not exceed 325 °C. When the trap is heated to 325 °C, trimethyl benzenes are released quantitatively. For higher boiling species it may be necessary to use a higher trap temperature.

Trap Breaks Inside the Trap Assembly

If the trap breaks inside the trap assembly you will need to contact your PerkinElmer Service engineer.

Replacing the Dry Purge Assembly

If you have noticed that the Dry Purge Assembly has been damaged you will need to replace it before you return it to the trap assembly.

1. Take the new dry purge assembly (P/N N6700112) out of its packing.
2. The dry purge assembly has two stainless steel lines which must be attached to the instrument correctly.

3. The longer stainless steel line goes to the bottom compression fitting (to the trap vent). See the following photo.
4. The shorter stainless steel line goes to the upper compression fitting (to the V9 valve). See the following photo.

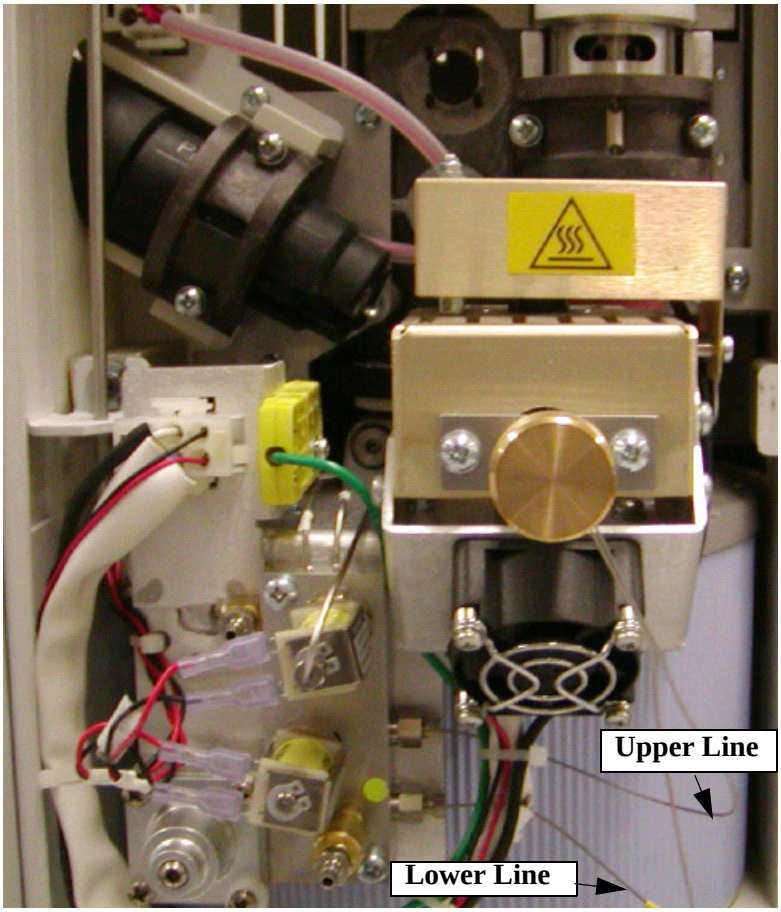


Figure 120

Trap Maintenance

Introduction for Conditioning the Trap

You will need to condition the trap if you have just installed a new trap or if you are encountering carry-over problems.

During the **Trap Clean** and the following procedure of **Trap Test** (to check if the trap is actually clean), no sample vial is involved.

The GC is also involved and a run is started only in **Trap Test**.

Before attempting a Trap Clean or Trap Test ensure that:

- There is adequate flow of carrier gas through the trap before a Trap heating cycle begins.
  First, in the **Option** tab choose **Trap Clean** from the **Operating Mode** drop down menu.
  Then, with a flowmeter, check flows at the **Needle Purge** outlet (12-18ml/min) and the **Trap Dry Purge** outlet (47-53ml/min). If not correct, re-adjust.
- Before starting the **Trap Test**, ensure that the GC has the proper GC method loaded and is **READY**.

Cleaning the Trap

The Trap Clean option allows you to clean the trap. See Figure 121.

CAUTION	*Be sure to stay below the minimum operating temperature of your installed trap adsorbent material.*

NOTE: The GC is not involved during the Trap Cleaning.

1. In the HS screen, select the **Option** tab.
2. To condition the trap, in the **Option** tab, select **Trap Clean** from the **Operating Mode** drop down menu.
3. Also, you can carry out the trap clean operation from the **Method Editor**. In the **Method Editor** screen, select the **Option** tab.
4. In the **Status** page, select the Temp button and set the trap Temperature to **280 ^{o}C** or higher, if required.

5. Select the **Timing** button on this **Status** page and set the **Trap Hold** time to **30 min**, or longer.
6. Touch **Start** to begin the trap conditioning procedure.

NOTE: If running in sequence, mode be sure to start the "active method" not the sequence. the sequence will activate the saved method.

During the Trap Hold 30 minutes, the vaporized Trap contaminants and moisture will elute, via the needle purge outlet or on to the column

During cleaning, the Isolation Flow of ˜10 ml/min will isolate the transfer line and analytical column from the Trap effluents. The GC is not involved in the trap cleaning. No GC run is started (it could even be off.

NOTE: It is recommended that you clean or install a new trap if you see carryover in your blanks run after this test.

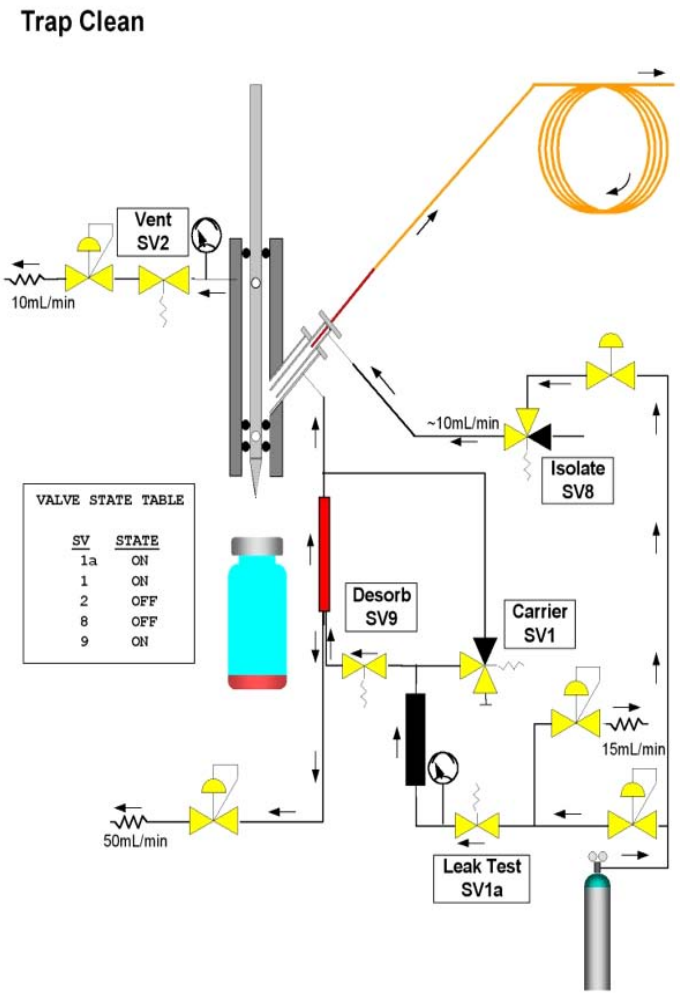


Figure 121 Cleaning the trap

Trap Test

After a Trap cleaning, the system should be checked with a blank run under the GC method to ensure that no ghost peaks or baseline disturbances are observed. This is the Trap Test procedure.

During the Trap Test, the system (HSTRAP+GC) simulates the Trap Desorption and Injection step of the HS Trap. At the same time, the GC runs its method cycle.

Monitoring of the baseline reveals whether the Trap cleaning is successful or that replacement of the Trap is necessary.

During this test, the Isolation Flow is shut-off, thus allowing, the trap desorption effluent to be transferred (injected) into the transfer line and the analytical column. The analytical head pressure is now supplied by the PPC module. See Figure 122 if you are running split setup. See Figure 123 if you are running a splitless setup.

- If the analytical requirements demand an HS Split operation, check the Outlet Split box on the OPTION tab. The VENT solenoid valve SV2 is kept open and a Split Flow of approximately 10 ml/min is used (Needle Purge outlet). See Figure 122.
- If HS splitless operation is used, SV2 is closed. See Figure 123.
- Ensure that:
  In the GC the correct method is setup and GC is READY.
  Dry Purge outlet flow is 50 ml/min.

To run a Trap Test:

1. On the HS screen, select the **Option** tab.
2. In the Option tab, select **Trap Test**.
3. On the Status page, select the **Temp** button and set the Trap Temperature to the value you have used for your standard method.
4. On the same Status page select the **Timing** button and set the TRAP HOLD time your typical method time (this time can vary from 5 to 45 minutes).
5. If you used Split operation, check the **Outlet Split** box (Option tab).
6. Touch the **START** to begin the TRAP TEST procedure.

NOTE: If running in sequence mode, be sure to start the "active method" not the sequence. the sequence will activate the saved method.

Monitoring of the GC baseline will show the condition of the Trap.

NOTE: Each Trap is shipped with a product sheet, "Air Monitoring

Trap Installation and Operating Instructions" (Part No. 0993-6724).

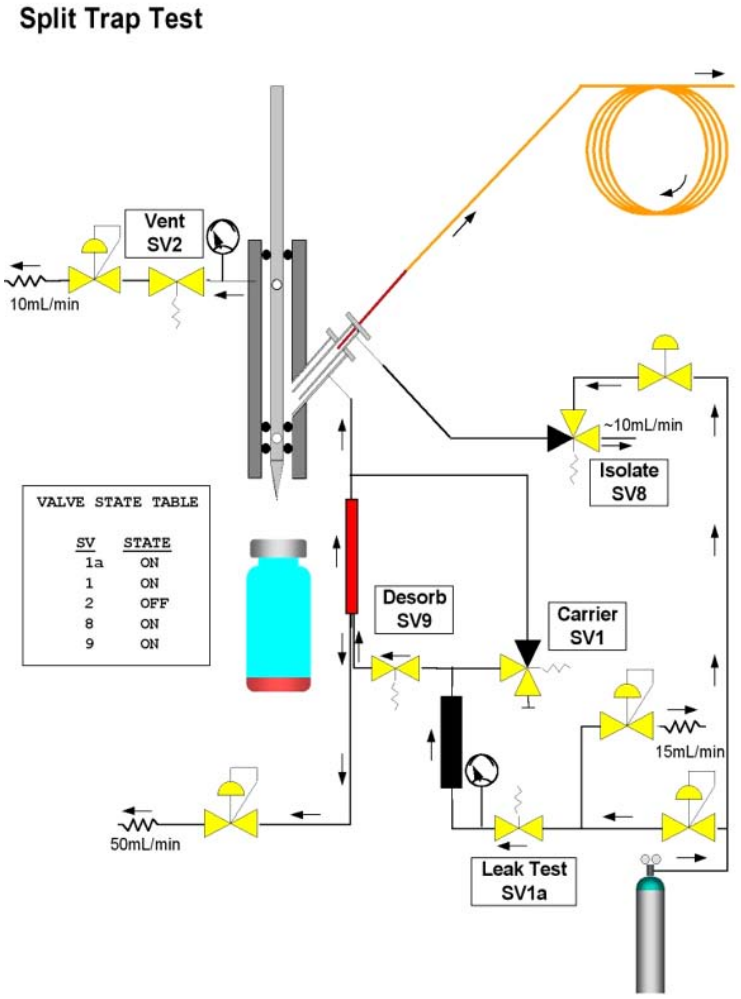


Figure 122 Split Trap Test

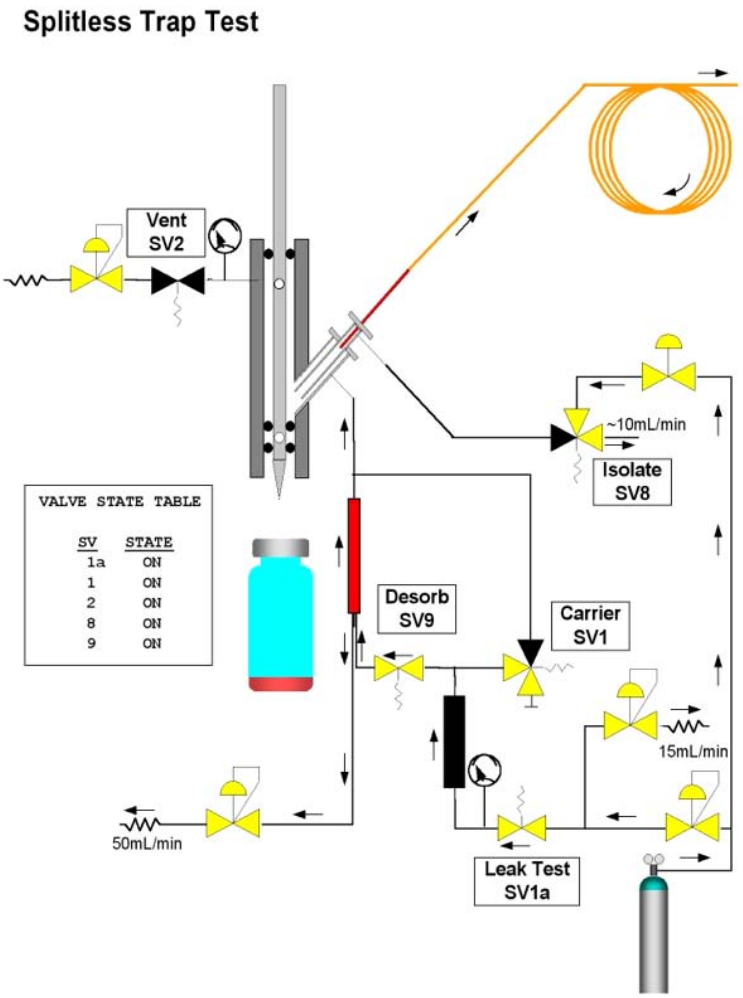


Figure 123 Splitless Trap Test

System Maintenance

System maintenance and a variety of part replacements can be carried out without switching the HS Trap unit off.

In the **Tools** drop down menu choose **Maintenance** and then touch the **Column Isolate** command. See the next screen.

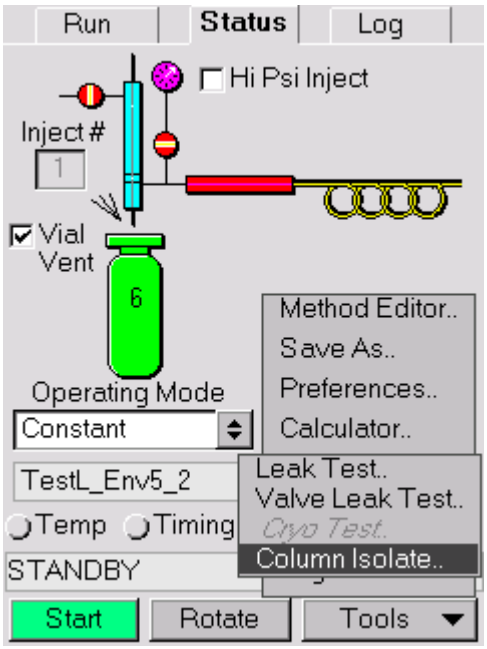


Figure 124

The following screen appears.

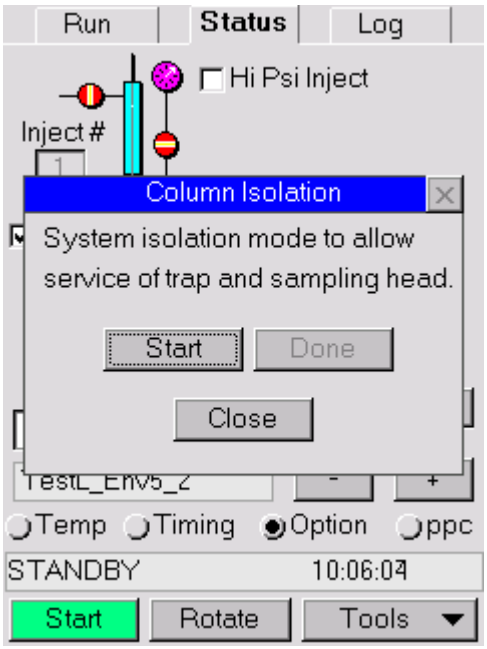


Figure 125

In Column Isolate (see figure below), the system supplies the Isolation Flow to isolate and protect the transfer line and the GC column. At the same time, it closes the SV1 and SV9 to stop the carrier gas to the Trap, the Sampling Head and the Needle.

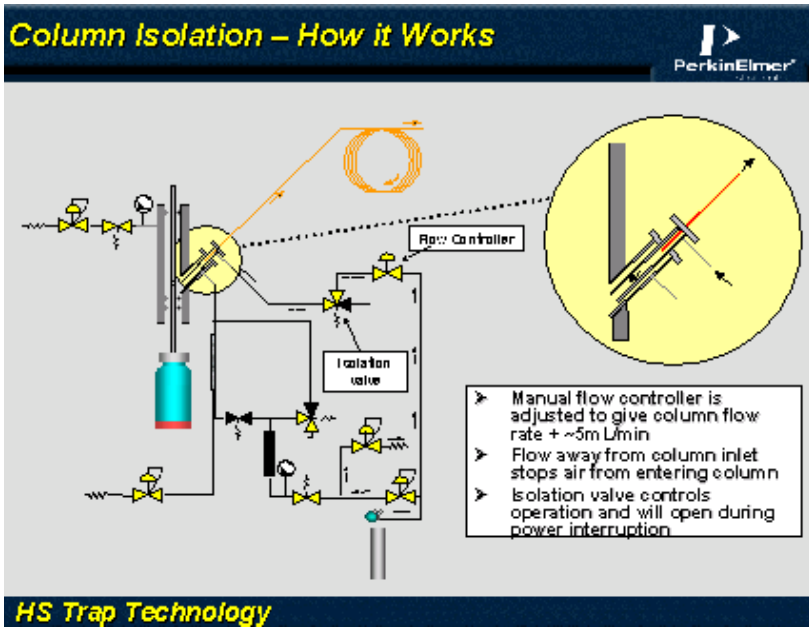


Figure 126 Column Isolation Flow

NOTE: Although PPC pressure is shut off, you will still measure a small flow, (5-9 ml/min), at the Needle Purge and Trap Dry Purge outlets. This is due to the Isolation Flow which, entering the Sampling Head, splits in to three branches, i.e. to the Transfer Line/GC column, to Needle Purge and to the trap and its Trap Dry Purge outlet.

If you have an MS detector, you will also observe an improvement in the vacuum, as the column flow is now considerably decreased.

See the following figure. This configuration will allow servicing and replacement of the trap, the needle and the upper and lower needle sealing o-rings.

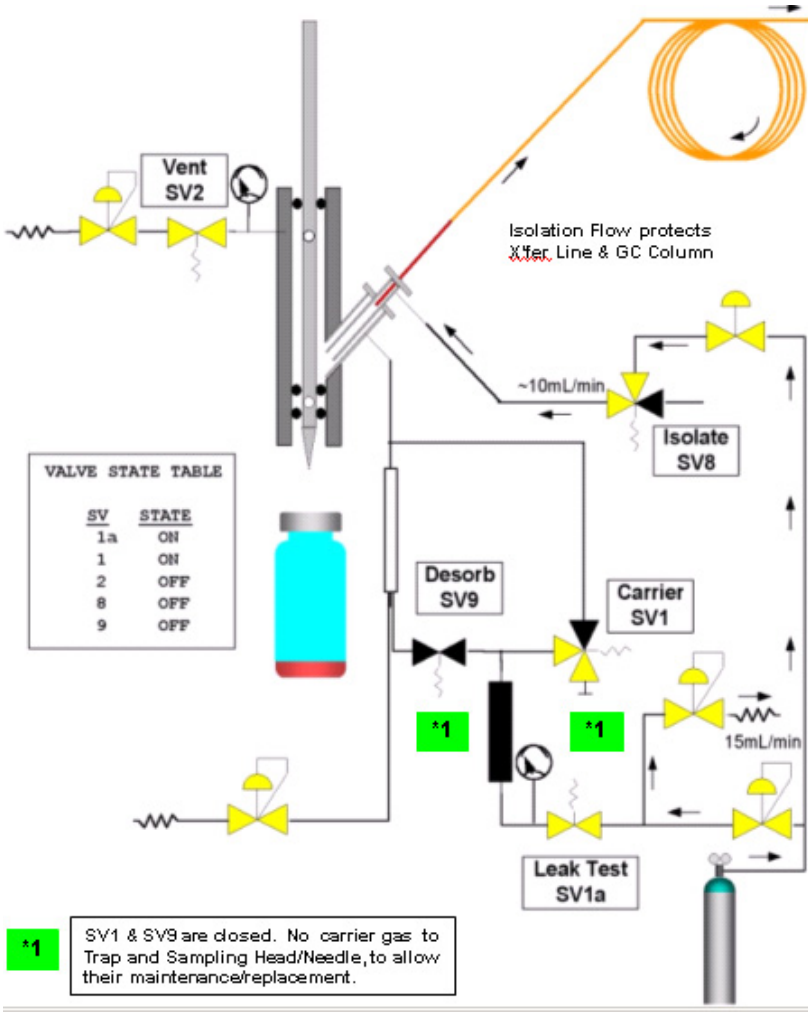


Figure 127

Before starting the maintenance, cool down the area you intend to work in (Oven, Needle, Trap).

Press the **Start** button to switch the system to the Column Isolate position and perform system maintenance.

On completion of the maintenance, press the **Done** button to return the system back to the Standby position.

Set the zones temperatures (Needle, Oven, Trap) to their method values.

Troubleshooting *7*

Status Messages

The following status messages are displayed during normal operation to indicate the current status of the instrument or the current analysis.

Status Messages	Description
Initializing	The instrument is testing all motors and internal systems at start up.
Standby	The instrument is ready and waiting for an analysis to begin.
Preparation..."	The HS is moving the first vial to the load position.
Equilibration."	The sample vial set points are being met.
GC Not Ready	The HS is ready to inject the sample or load a vial into the oven and has received a not ready signal from the GC.
Thermostat Time	The sample vial is being thermostatted
Pressurize Time	The sample vial is being pressurized
Ext Thermostat	GC oven temperature is not ready (the cycle time is probably not enough).
Inject Time	The sample is being injected onto the GC column.
Withdrawal Time	The needle is being retained in the vial after injection
Vent Time	The vial is being vented.
Analysis Time	This is the GC analysis time.
Finishing...	The HS is finishing up a vial sequence or returning the last vial.
Leak Test Time	The timer for the leak test
Error Condition	A fault condition is occurring. See *Status Messages* on this table and pages 314-315.
Economy Mode	The instrument is in economy mode.
Dry Purge Time	The trap is being purged with carrier gas to remove water.
Inject Standard	The internal standard is being added to the tube.

Sampling	The pressurized vial is being vented through the trap.
Trap Conditioning	The trap is being conditioned at top trap temperature (purged and vented).
Trap Test	The gas is flowing from the trap to the GC column.
Analyzing	An analysis is in progress
Dry Purging	The trap is being purged with helium out to vent.
Economy Mode	The instrument is in the economy mode. The heaters have been switched off and the carrier gas flow has been reduced to the minimum.
Fatal Error	A fatal error is a malfunction from which the instrument cannot recover without user intervention. The instrument may need to be switched off and then turned on again. If the fatal error reoccurs you will need to contact your PerkinElmer Service Representative.
Inject Standard	The internal standard is being added to the vial
Out Split Adjust	Adjust the outlet split flow. SV2 is open.
Primary Desorb	The tube oven is moved into position and the sample vial is heated for the duration of the desorb time.
Dry Purge	The trap is being purged with carrier gas to remove any air from it.
Ready	The instrument is ready to begin an analysis.
Fault	A fault is occurring.
Trap Cond Heat	The instrument is in trap condition mode and the trap is being heated to the set point.
Trap Cond Hold	The instrument is in trap condition mode and the trap is being held at the set temperature.
Trap Heat	The trap is being heated to its high temperature.
Trap Hold	The trap is being held at its high temperature.
Waiting for GC	The primary desorption has been completed and the HS 40/110 trap is waiting for a GC ready signal before the cold trap is heated.

Table 14 Instrument Status Messages for Headspace and HS 40/110 Trap.

TurboMatrix Headspace and the HS 40/110 Trap Instrument Fault Conditions

If there is more than one fault occurring at a time, the instrument will cycle through them and display them one at time until they are corrected. The following faults cause the instrument to stop immediately. All heated zones are turned off and a message is shown on the display localizing the error. Record the details of the failure before switching off the instrument.

Fatal error can only be remedied by switching the instrument off with the power switch, waiting thirty seconds and switching it on again. The instrument will then restart and attempt to initialize itself.

If the error recurs, please make note of the exact wording of the error message and contact your PerkinElmer service engineer.

Before you contact service, ensure you have the following information:

- What you were doing when the error occurred
- Any corrective action that you have taken
- The exact wording of the error message and any numerical error codes.
- Additionally you should have this manual at hand.

Fault: Pneumatic Psi Too Low

Cause: The supply of dry air used to drive the automated vial handling components has run out.

Action: Replace the tank of dry air. Open the tank and set the delivery pressure to 90 psig. See the *Gas Connections* section in the *Installation and Setup* Chapter for details.

Fault: Carrier Psi Too Low

Cause: The supply of carrier gas has run out.

Action: Replace the tank of carrier gas. Carrier gases require a minimum purity of 99.995%. Open the tank and set the delivery pressure to 90 psig. See the *Gas Connections* section in the *Installation and Setup* Chapter for details.

Fault: Magazine Motor Stalled

Cause: An obstruction has caused the magazine to halt rotation.

Action: Turn the HS off and disconnect the power cord from the AC mains. Remove the obstruction and reconnect the line cord. Turn on the HS. The instrument will initialize the motors. It will then return a Ready status if all of its systems have been initialized correctly. You must update the vial range on the Run tab to exclude the vials that have already been run and press the Start button to continue your analyses.

Cause: A mechanical problem has occurred.

Action: Contact your PerkinElmer service engineer.

Fault: Elevator Motor Stalled

Cause: An obstruction has caused the elevator to halt.

Action: Turn the HS off and disconnect the power cord from the AC mains. Remove the obstruction and reconnect the line cord. Turn on the HS. The instrument will initialize the motors. It will then return a Ready status if all of its systems have been initialized correctly. You must update the vial range on the Run tab to exclude the vials that have already been run and press the Start button to continue your analyses.

Cause: A mechanical problem has occurred.

Action: Contact your PerkinElmer service engineer.

Fault: Oven Motor Stalled

Cause: An obstruction has caused the oven to halt rotation.

Action: Turn the HS off and disconnect the power cord from the AC mains. Remove the obstruction and reconnect the line cord. Turn on the HS. The instrument will initialize the motors. It will then return a Ready status if all of its systems have been initialized correctly. You must update the vial range on the Run tab to exclude the vials that have already been run and press the

Start button to continue your analyses.

Cause: A mechanical problem has occurred.

Action: Contact your PerkinElmer service engineer.

Fault: Needle Motor Stalled

Cause: An obstruction has caused the needle to halt.

Action: Turn the HS off and disconnect the power cord from the AC mains. Remove the obstruction and reconnect the line cord. Turn on the HS. The instrument will initialize the motors. It will then return a Ready status if all of its systems have been initialized correctly. You must update the vial range on the Run tab to exclude the vials that have already been run and press the Start button to continue your analyses.

Cause: The o-ring seals have degraded.

Action: Replace the o-rings as outlined in the section on *Changing the O-rings* in the *Routine Maintenance* chapter.

Cause: The needle cannot penetrate the septa of the sample vial.

Action: You are using septa or caps that are not for use on automated headspace sampler. Use only PerkinElmer vials, caps and septa.

Cause: The needle is dirty.

Action: Abraded sealing material from the vial septa may stick to the needle and can cause the needle to seal incorrectly during pressurization and withdrawal. Clean the needle as outlined in the section on *Cleaning the Jet Needle* in the *Routine Maintenance* chapter.

Cause: The needle motor has overheated.

Action: The sampling head is not being cooled efficiently. Ensure the front access door is closed when the HS is in operation.

Action: Turn off the HS and allow 10 to 15 minutes for the driver motor to cool down. This fault may

be an indication that the motor is working too hard. Check the needle to ensure it is clean. Also check the o-ring and the seal assemblies. See the section on *Changing the Upper Needle Seal Assembly* in the *Routine Maintenance* chapter and *Changing the Lower Seal Assembly* in the *Routine Maintenance* chapter.

Cause: The needle seals have not been installed correctly.

Action: The needle seal assemblies have not been aligned correctly and the needle is bending. Remove the seal assemblies and ensure they are aligned correctly. See *Changing the Upper Needle Seal Assembly* in the *Routine Maintenance* chapter and *Changing the Lower Seal Assembly* in the *Routine Maintenance* chapter.

Action: An extra seal o-ring has been installed in the upper seal assembly. Remove the upper seal assembly and ensure that only the lower o-ring is installed.

Cause: The needle has not been installed correctly.

Action: Remove the needle and ensure it has been installed correctly. See *Removing and Replacing the Needle* in the *Routine Maintenance* chapter.

Cause: A mechanical problem has occurred.

Action: Contact your PerkinElmer service engineer.

Fault: Crane Motor Stalled

Cause: An obstruction has caused the crane to halt.

Action: Turn the HS off and disconnect the power cord from the AC mains. Remove the obstruction and reconnect the line cord. Turn on the HS. The instrument will initialize the motors. It will then return a Ready status if all of its systems have been initialized correctly. You must update the vial range on the Run tab to exclude the vials that have already been run and press the Start button to continue your analyses.

Cause: A mechanical problem has occurred.

Action: Contact your PerkinElmer service engineer.

Fault: Sensor Failure Zone 1, 2, 3 or 4

Cause: The sensor, for the vial oven in zone 1 has failed.

Action: Contact your PerkinElmer service engineer.

Cause: The sensor, for the needle, in zone 2 has failed.

Action: Contact your PerkinElmer service engineer.

Cause: The sensor, for the heated transfer line, in zone 3 has failed.

Action: Turn the HS off and disconnect the power cord from the AC mains. Ensure the electrical connector from the transfer line is securely connected to the HS connector. See the section on *Installing the Heated Transfer Line at the HS Needle Unit* in the *Installation and Setup* chapter. Reconnect the power cord and turn on the HS to see if the fault has cleared.

Action: Contact your PerkinElmer service engineer.

Cause: The sensor, for the cryofocusing accessory, or the sensor for the HS 40/110 trap HS 40/110 trap in zone 4 has failed.

Action: Contact your PerkinElmer service engineer.

Fault: Vial Missing

Cause: No vial was present when the Start button was pressed or an empty slot was encountered during automatic operation.

Action: Ensure all of the slots in the specified vial range contain vials. To check the vial range, open the Run tab. The methods and desired vial range will be displayed. Press Stop then rotate the magazine to gain access to the vial location and insert a sample vial. Press the Start button on the display to start analyses.

Cause: Your method specifies multiple analyses on a vial and you have removed the specified vial after the first analysis was started.

Action: Press Stop and then place the vial into the HS. Press Start to resume your analysis.

Cause: If there has been a power failure it is possible that a vial has been dropped by the crane arm.

Action: Turn the instrument off and disconnect the power cord from the AC mains. Remove the vial and replace it in the magazine.

Action: If the vial has fallen into the instrument, contact your PerkinElmer service engineer.

Fault: No Vials in First and/or Last Positions

Cause: The first and/or last vials from the specified range have not been placed on the magazine.

Action: Press Stop and rotate the magazine to gain access to the vial location and insert a sample vial and clear the fault. Press the Start button on the display to start the analyses.

Action: Press Stop. Open the Run tab and adjust the vial range so that only vials that have already been placed on the magazine are included in the vial range. Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Fault: Vial Load Failure

Cause: The HS could not load the vial from the magazine.

Action: The vial has not been capped correctly. Press Stop and rotate the magazine to gain access to the vial location. Recap the vial and place it on the magazine. Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Action: If the vial is stuck in the magazine, remove it and clean the outside of the vial. Ensure there is no adhesive material or residual sample on the outside of the vial before inserting it into the magazine. Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Action: Do not use vials, and seals that are not specifically identified for use with an automated headspace sampler. Using other types of vials may result in unreliable analysis data or damage to the instrument.

Action: If you are using the low volume (9mL) vials, you must install the low volume vial adapters (P/N N612-0110) in the magazine. If you are using HS 110, you can only use the low volume vials and adapters in the outer ring.

Action: The magazine is not rotating correctly. It either overshoots or does not reach the target position. Contact your PerkinElmer service engineer.

Fault: Vial Unload Failure

Cause: The HS could not replace the sampled vial on the magazine.

Action: If you have placed a new vial into the location, from which a vial was removed by the instrument, you must remove it. Press Stop and rotate the magazine to gain access to the magazine location and remove the vial. Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Cause: If there has been a power failure it is possible that a vial has slipped from the crane arm.

Action: Turn the HS off and disconnect the power cord from the AC mains. Remove the vial from the unload position. Turn on the HS. You may need to update the vial range on the Run tab to exclude the vials that have already been run. Press the Start button to continue your analyses.

Fault: Magazine Full

Cause: You have placed vials in the load and unload position of the HS 110 or in the unload position of the HS 40.

Action: On instruments with the 15-vial oven, it is

possible to turn the instrument off while vials are still in the oven. When the instrument is turned on again, it will attempt to unload the oven. You must provide an empty spot in the outside ring of the magazine so that the HS can unload the oven.

When you are loading the HS 40, you can place vials in all positions except for one. You must leave one spot open in order for the HS to unload any vials that may be present in the oven.

When you are loading the HS 110 you must leave the load and unload spots empty. You must also leave one further location on the outside ring empty for the HS to unload any vials that may be present in the oven. The HS will search the outer ring until it locates the empty position. To reduce the initialization time, leave location #1 empty. This is the first location that the instrument will look for the empty position.

Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Fault: Vial Jammed in Oven

Cause: The nut that holds the needle in place has loosened and the needle cannot be lifted out of the vial.

Action: Remove the needle. See the section on *The Sampling Needle* in the *Routine Maintenance* chapter for more details.

Cause: The vial is slightly eccentric or has material on the outside.

Action: Turn the HS oven power off. Allow time for the oven to cool. Turn the power on and check if the cooled vial is removed during initialization.

Cause: A mechanical problem has occurred.

Action: Contact your PerkinElmer service engineer.

Fault: Vial in Load Position

Cause: You have placed vials in the load position of the HS 110.

Action: When you are loading the HS 110 you must leave the load and unload spots empty. You must also leave one further location on the outside ring empty for the HS to unload any vials that may be present in the oven. The HS will search the outer ring until it locates the empty position. To reduce the initialization time, leave location #1 empty. This is the first location that the instrument will look for the empty position.

Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Fault: Vial in Unload Position

Cause: You have placed vials in the unload position of the HS 110 or the HS 40.

Action: On instruments with the 15-vial oven, it is possible to turn the instrument off while vials are still in the oven. When the instrument is turned on again, it will attempt to unload the oven. You must provide an empty spot in the outside ring of the magazine so that the HS can unload the oven.

When you are loading the HS 40, you can place vials in all positions except for one. You must leave one spot open in order for the HS to unload any vials that may be present in the oven.

When you are loading the HS 110 you must leave the load and unload spots empty. You must also leave one further location on the outside ring empty for the HS to unload any

vials that may be present in the oven. The HS will search the outer ring until it locates the empty position. To reduce the initialization time, leave location #1 empty. This is the first location that the instrument will look for the empty position.

Open the Preferences menu and select Reset. The instrument will reset itself and clear the fault. Press the Start button on the display to start the analyses.

Fault: Checksum error, to continue, press any key.

Cause: An instrument error has been detected during initialization.

Action: A message appears on the display indicating where the error was detected. Please take a note of the error message and contact your PerkinElmer service engineer.

ATTENTION: Carrier Gas Shut Off

There are cases where you are instructed to either switch off the Headspace or the HS 40/110 trap or/and shut-off the carrier gas supply to it.

However, in certain cases as

- Headspace units with direct or on-column connection configurations
- In HS 40/110 trap, the Isolation Flow

You should not switch the HS unit off (PPC) or the carrier gas supply to it, or, in an HS/TRAP unit, the carrier gas should not be shut-off as the Isolation Flow (that protects the analytical column) will be stopped.

Therefore, in cases as above, you should not attempt maintenance or troubleshooting in the Headspace or HS 40/110 trap unit, unless you have ensured the following in the GC:

1. If the detector is a flame one (FID, NPD, etc.), the flame should be off.
2. If the detector is an MS, the transfer line (if present)

should be cool. The ion source should be cool.

3. GC oven, injector and detector in the HS analytical column channel should be cool.

Following the above steps will protect you in case you are busy with the HS/TRAP maintenance or troubleshooting and you forget that the GC is still at a high temperature which will damage the column and possibly the transfer line if the carrier gas is cut off.

Troubleshooting Procedures

Problem:Leak test failed

Cause: The needle is coated with abraded sealing material.

Action: Abraded sealing material from the vial septa may stick to the needle and can cause the needle to seal incorrectly during pressurization and withdrawal. Clean the needle as outlined in the section on *Cleaning the Jet Needle* in the *Routine Maintenance* chapter.
Ensure that the oven temperature and the needle temperature do not exceed the septa maximum (see page 179).

Cause: The needle has not been installed correctly.

Action: Remove the needle and ensure it has been installed correctly. See *Removing and Replacing the Needle* in the *Routine Maintenance* chapter.

Cause: The needle seal assemblies have degraded.

Action: Open the upper and lower needle seal assemblies and replace the o-rings. Check the seals for signs of wear. Replace them if necessary. See Changing the O-Rings in the *Routine Maintenance* chapter.

Cause: The transfer line has not been installed correctly at the needle or at the GC injector.

Action: Check the connections at the sampling unit and ensure all nuts and ferrules have been installed correctly. Do not over-tighten any fittings.

NOTE: The adapter tube is a glass-lined stainless steel tube. Over-

tightening the fittings may result in damaging the tube or the fittings.

CAUTION	*Ensure that the transfer line does not touch the needle. This could cause bending and scratching.*

Action: Check the connections at the GC injector.

Action: Ensure both ends of the fused silica transfer line have been cut cleanly. See *Figure 13* in the *Installation* chapter.

Cause: The gas connections on the rear panels have not been made correctly.

Action: Check and tighten all gas fittings on the rear panel.

Problem: Cannot select another method on the Run tab.

Cause: Single method operation has been selected on the Preference's Run Tab.

Action: If you need to edit the method, you must disable single method operation. Refer to the section on *Preferences Tab* in the *Operation* chapter.

Problem: Cannot edit a method from the Run tab. The Method Editor is greyed out.

Cause: Single method operation has been selected on the Preference's Run Tab.

Action: If you need to edit the method, you must disable single method operation. Refer to the section on *Preferences Tab* in the *Operation* chapter.

Problem: Cannot enter an injection pressure for the high pressure injection on the PPC tab.

Cause: The high pressure option is not enabled on the Options tab.

Action: Switch to the Option tab and enable the High Pressure Injection option. A check mark must appear in the box. Open the PPC tab and press the injection pressure to enable it. Use the + and – keys to enter the desired value.

Problem: Cannot enter pre and post cryofocusing times on the Timing tab.

Cause: The cryofocusing accessory is not enabled on the Options tab.

Action: Switch to the option tab and enable the cryofocusing option. A check mark must appear in the box. Open the Timing tab and press the pre-cryofocusing to enable it. Use the + and – keys to enter the desired value.

Peak Broadening or Splitting

The chromatographic peaks represent the distribution of molecules in a band as it elutes from the column, the overall broadness being conveniently measured in terms of the width of the peak. A number of independent factors such as temperature and column retention processes, contribute to the dispersion of molecules in a band and band broadening. Classical chromatography theory considers that the separation process takes place by a succession of equilibrium steps, the more steps in a column the greater the column efficiency with less band broadening occurring.[1]

Peak broadening and splitting are undesirable and can lead to inaccurate quantitation or misidentification.

Cause: Activity in the transfer line can cause peak broadening

Action: If this occurs, the section of capillary column or the deactivated fused silica in the transfer line should be replaced as described in *Installing the Heated Transfer Line* in the *Installation and Setup* Chapter.

1. A. Braithwaite and F.J. Smith, Chromatographic Methods, Fifth Edition, (Glasgow, 1996), p27

Cause: A poor connection between the transfer line and the analytical column can cause peak broadening.

Action: Ensure that the connecting ends of the column and the transfer line should be cut cleanly using a wafer silica cutting tool. See Figure 10 in the Installation chapter of this manual.

Action: The union or connector assembly should be an inert, zero dead-volume fitting recommended for butt connected capillary, fused silica tubing.

Action: If you are using the zero dilution liner ensure that it has been installed correctly. See *Installing the Zero Dilution Liner* in the *Accessories* chapter. It is possible that during installation of the liner, pieces of fused silica have broken off the transfer line and dropped into the injector. Clean the injector as necessary

Cause: Activity of the analytical column itself can cause band broadening.

Action: Replace the GC capillary column with one that is more closely suited to your application. Refer to the GC manual for instructions.

Cause: The sample has overloaded the GC column.

Action: Use split sampling to reduce the sample volume sample that reaches the GC column. Adjust the injected split flow to deliver the correct sample volume onto the GC column.

Sample Carryover

Sample carry over is rare in HS and may be avoided with proper care and maintenance of you instrument. Contamination can also be avoided by ensuring that your method is suitable for the compounds that you are analyzing.

Cause: The needle or the transfer line is not hot enough and the sample is condensing.

Action: Set the temperature of the needle and the transfer line to at least 10°C higher than the vial oven.

Cause: The needle purge flow is below the acceptable range.

Action: Check the needle purge flow and ensure the flow rate at the purge vent is between 10-20 mL/min. If the flow is below 10 or greater than 20 mL/min, contact your PerkinElmer service engineer.

Cause: The GC injector is not being flushed completely.

Action: In split sampling, carrier gas continuously purges the split/splitless injector to avoid back diffusion and sample carryover. When operating with PPC control, a minimum flow of approximately 2 mL/min (split flow) should be supplied by the GC's pneumatic modules to purge the GC carrier gas lines.

Keeping this flow smaller than the septum purge flow ensures, that no dilution of the injected headspace sample takes place in the injector. A higher flow from the GC pneumatics can be applied to dilute the injected sample if necessary.

Cause: If your are using the direct connect configuration or an on-column connection and the injection time or the injection volume is too high, the column is being overloaded.

Action: Increase the flow of carrier gas through the transfer line, and then set the transfer line temperature to 210 °C. Allow the transfer line to purge overnight.

WARNING

Ensure that the column liquid phase can withstand a temperature of 210 °C. If not, then set the transfer line temperature to the maximum allowable for the selected liquid phase of column.

If you are observing a severe contamination problem, you should disconnect the transfer line from the GC.

WARNING

Make sure that the GC oven, detector and injector are at room temperature before you start working on the Headspace as instructed here.

Action: Reduce the injection time or volume to introduce a smaller sample.

Action: Reconfigure your transfer line connection for split injection sampling. Generally splitless sampling allows less flow through the sampling area. Reduced flow may lead to increased sample carry over and thus worse analyte reproducibility. This is due to less efficient sweeping of the needle area.

System Contamination for the Headspace and HS 40/110 Trap

System contamination can be avoided with proper care and maintenance of you instrument. Contamination can also be avoided by ensuring that your method is suitable for the compounds that you are analyzing.

When you are troubleshooting the HS-GC system, you should first eliminate the GC as the source of the contamination. Disconnect the HS from the GC. If the baseline signal drops to the normal detector background, the HS is most likely the source of contamination.

Cause: The carrier gas supply is a very common source of contamination in the headspace and the HS 40/110 trap.

Action: Contaminants can be introduced through the gas itself, cylinder regulators, gas lines or carrier gas filters. To establish whether or not this is the source of the problem, change your tank of carrier gas, the regulator and supply tubing. If the problem persists, contamination is likely entering from another source or the carrier gas is of poor quality.

Cause: The needle or the transfer line are not hot enough and the sample is condensing.

Action: Set the temperature of the needle and the transfer line to at least 10 °C higher than the vial oven.

Cause: The needle purge flow is below the acceptable range.

Action: Check the needle purge flow and ensure the flow rate at the purge vent is 15 ± 3 mL/min. See *Checking the Needle purge Gas Flow* in the Installation chapter of this manual. If the flow is below 10 or greater than 20 mL/min, contact your PerkinElmer service engineer.

Cause: The transfer line is contaminated

Action: Increase the flow of carrier gas through the transfer line set the transfer line temperature to its maximum 210 °C temperature. Allow the transfer line to purge overnight.

If you are using the on-column connection configuration, ensure that the column liquid phase material can withstand a temperature of 210 °C. If not, then set the transfer line temperature to the maximum allowable for the selected liquid phase.

If you are observing a severe contamination problem, you should disconnect the transfer line from the GC.

WARNING

Make sure that the GC oven, detector and injector are at room temperature before you start working on them.

Action: Replace the fused silica transfer line. See *Installing the Transfer Line Cap* in the *Routine Maintenance* chapter of this manual.

Action: When the transfer line is heated, there must always have flow through the transfer line. This

becomes more critical if you are using an on-column configuration that utilizes the GC column as the transfer line. When you are changing the supply tanks and if direct or on-column connection, you must power down the instrument and allow the transfer line to cool. Also insure that the GC oven injector/detector is cool and the MS filaments are off.

Action: If the transfer line is disconnected from the GC for any period of time protect the end of the fused silica tube with the transfer line cap (P/N B0510403). See *Installing the Heated Transfer Line* in the *Installation* chapter.

Action: The fused silica tube (transfer line) may need to be further de-activated for your application. Contact the PerkinElmer for further information.

Action: If you are using the direct or on-column connect configuration, ensure that the injection time or the injection volume is suitable for the column and does not overload the column.

Cause: The sampling head is contaminated.

Action: Increase the flow of carrier gas through the transfer line and set the transfer line temperature to 210 °C. Allow the transfer line to purge overnight.

If you are using the on-column connect configuration, ensure that the column liquid phase can withstand a temperature of 210 °C. If not, then set the transfer line temperature to the maximum allowable temperature for the selected liquid phase.

If you are observing a severe contamination problem, you should disconnect the transfer line from the GC and replace the transfer line.

Action: If purging does not reduce or eliminate the contamination, parts of the sampling head may need to be replaced. Contact your PerkinElmer

service engineer.

Cause: The fittings connecting the transfer line to the sampling head have degraded or they are contaminated.

Action: Disconnect the transfer line from the sampling head and replace all the nuts and ferrules. Also replace the deactivated tube adapter. Reconnect the transfer line to the sampling head as outlined in *Installing the Heated Transfer Line at the HS Needle Unit* in the *Installation and Setup* chapter. Increase the flow of carrier gas through the transfer line and set the transfer line temperature to 210°C. Allow the transfer line to purge overnight.

Poor Sample Recovery or Reduced Sensitivity

If you cannot obtain the desired detection limits, you must first eliminate the GC as the source of the problem. Inject a representative sample at the desired concentration directly into the GC and ensure that you can obtain the desired response. If you cannot obtained the desired response then refer to your GC user's manual or contact your PerkinElmer service engineer. If the GC is fine then you must troubleshoot the HS and the transfer line.

Cause: The needle or the transfer line are not hot enough and the sample is condensing.

Action: Set the temperature of the needle and the transfer line to at least 10°C higher than the thermostatting temperature.

Cause: The sample has not reached equilibrium before the sample injection was made.

Action: The pressurization time needs to be long enough to ensure homogeneity of the gas phase in the vial. The pressurization time should be at least 2 minutes for good reproducibility and 3 minutes for optimum reproducibility. For some applications where a short pressurization time is used to increase productivity, expect

deterioration in performance. You may see an increase in RSD from 1% to 3% to greater than 5% RSD. A pressure gauge (P/N B0501377) is available for measuring the sample vial pressure.

Cause: The fittings connecting the transfer line to the sampling head have degraded and are leaking.

Action: Disconnect the transfer line from the sampling head and replace all the nuts and ferrules. Also replace the deactivated tube adapter. Reconnect the transfer line and the sampling head, as outlined in *Installing the Heated Transfer Line at the HS Needle Unit* in the *Installation* chapter. Increase the flow of carrier gas through the transfer line and set the transfer line temperature to 210°C. Allow the transfer line to purge overnight.

Cause: There is leak in the sampling system.

Action: Disconnect the fused silica transfer line from the GC and ensure there is flow through the transfer line. Connect a flow meter to the end of the fused silica transfer line and then set the HS carrier pressure to 20 psi (138kPa), ensure there is a steady flow

Action: Perform a leak test as outlined in *Leak Testing the Sample Injection System* section in the *Installation* chapter.

Action: Connect the HS to the GC and leak test the GC/HS system. Check all of the connections with a helium Leak Hunter or concentrated ethanol and water solution. Once you have checked and tightened all of the connections, then run the leak test again.

Action: Heat the GC oven to the highest temperature used in the current method. Allow the GC oven to cool and then tighten the column connection fittings.

Action: Check the rear panel gas connections. Ensure the carrier gas is securely connected and that the

delivery pressure is set to 90 psig (620kPa).

Cause: The lower opening on the needle is plugged.

Action: Clean the needle. See *Cleaning the Jet Needle* in the *Routine Maintenance* chapter.

Action: Replace the needle. See *Removing and Replacing the Needle* in the *Routine Maintenance* chapter.

Cause: The needle seal assemblies have degraded.

Action: Open the upper and lower needle seal assemblies and replace the o-rings. Check the seals for signs of wear. Replace them if necessary. See *Changing the Upper Needle Seal Assembly* and *Changing the Lower Seal Assembly* in the *Routine Maintenance* chapter.

Cause: The needle has not been installed correctly.

Action: Ensure the needle holes are pointing towards the transfer line. See *Removing and Replacing the Needle* in the *Routine Maintenance* chapter.

Cause: The transfer line has not been installed correctly at the needle or at the GC injector.

Action: Check the connections at the sampling unit and ensure all nuts and ferrules have been installed correctly. Do not over-tighten any fittings.

NOTE: The deactivated tube adapter tube is a glass-lined stainless steel tube. Over-tightening the fittings may result in damaging the tube or the fittings.

Action: Check the connections at the GC injector.

Action: Ensure both ends of the fused silica transfer line have been cut cleanly. See Figure 10.

Cause: The thermostatting temperature is too high and the sample is degrading. Sample loss can also be attributed to polymerization, depolymerization, or decomposition.

Action: Ensure the thermostatting time and temperature are appropriate for your application. In the heated transfer line, the headspace gas is a

mixture of air with trace concentrations of the analytes. Setting a high temperature may cause sample decomposition by oxidation.[1] Set the thermostatting, needle and transfer line temperature accordingly.

Cause: The original samples have not been prepared correctly.

Action: Ensure that the sample was prepared correctly. i.e. if you are using a diluted liquid sample ensure that you have the correct dilution ratio.

Cause: The vials have not been sealed correctly.

Action: Ensure the caps have been assembled and crimped correctly. If you can turn the cap, after it has been crimped, it will leak. Remove the cap and dispose of it. Install another seal. See *Sealing the Vials* in the *Accessories* chapter. If the cap cannot be crimped correctly, adjust the hand crimper. See *Adjusting the Hand Crimper* in the *Routine Maintenance* chapter.

Action: If the pressure in the vial is excessive, the seal will begin to vent and result in sample loss. When you are creating methods, ensure that you set thermostatting times and temperatures that are suitable for both your sample and the solvent. See *Sealing the Vials* in the *Accessories* chapter.

Action: Unsafe high pressure in the vial during thermostatting may also be due to the use of a solvent with a boiling point that is too low for the application. Ensure that your solvent is suitable for you application. You can use the vial pressure gauge (P/N B0501377) to measure the vial pressure.

Cause: The sample is being absorbed by the septa sealing the sample vial.

Action: Butyl rubber septa (P/N B0159356, B0159357)

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 71

are known to absorb non-polar compounds.[1]. Use PTFE coated septa if your solvent or sample is non-polar.

Cause: The septa used to seal the vial is not suitable for the temperature requirements of your application.

Action: Ensure the temperature limit of the septa is within the range required for your analysis (see page 179).

With respect to the temperature limit of the septa, it is important to emphasize that it applies not only to the temperature of the vial, but also to the temperature of the instrument's needle used for pressurization and sample transfer, which is heated to prevent condensation. If, for example, the vial is at 80°C, but the needle is heated to 150°C, the hot needle may decompose the septum material, resulting in spurious peaks in the chromatogram and a leak around the needle. This may also occur with laminated septa, where one of the layers may have a lower temperature limit than the bulk material or vice versa.

Cause: The PPC on the GC has not been setup for operation with an HS.

Action: You can check by pressing **SYSTEM** from the GC keyboard, then keep pressing **ENTER** until you see **HEADSPACE Config. installed? YES,** if not, set it to **YES.**
On the Clarus GC touchscreen, select Tools->Configuration-> Injector icon and insure that the HS control box is checked.

Cause: The split flow has not been set correctly.

Action: If you have a split set-up, you will normally experience some dilution from the carrier added to the GC injector through the pneumatic modules in the GC. This flow needs to be higher

1. Bruno Kolb and Leslie S. Ettre, Static Headspace Gas Chromatography, Theory and Practice, (New York, 1997), p. 52

than zero in order to keep analytes and other contaminants from diffusing back into the pneumatic lines and contaminating the GC system.

Action: To reduce the dilution effect use the smallest available injector liner: 2mm for Split/Splitless injector (P/N N6121002) or zero dilution split injector (P/N N1011445 and P/N N1011446); 1 mm for PSS injector (P/N N6121006)

Action: Reduce the split flow in the GC method. On PPC instruments, this is the flow added to the GC injector by the PPC module. alternatively, you can use the zero dilution liner.

NOTE: Do not confuse this with the total flow through the split outlet or vent on the GC, which can be measured using a flow meter. The latter flow is comprised of the HS transfer line flow + the flow from the GC PPC module - Column flow.

Action: Turn off the split leak. Press **SYSTEM** from the GC keyboard, then keep pressing **ENTER** until it shows **SPLIT FLOW OFFSET 1, FIXED**.
On the Clarus GC touchscreen, select Tools->Configuration-> Injector icon and adjust the offset value to 1.00.

Action: Install the zero dilution interface at the GC injector. See *Installing the Zero Dilution Liner* in the *Accessories* chapter. This liner allows you to purge the injector with GC carrier gas without diluting the HS sample entering through the transfer line. this liner also eliminates peak tailing.

HS 40/110 Trap Only Troubleshooting

Cause: Excessive carryover.

Action: Perform a trap clean procedure (refer to the *Clean the Trap* in the *Routine Maintenance* chapter).You might need to clean the trap a few times to lower the excess carryover.

Action: The HS 40/110 trap is designed for low level volatile analysis therefore if you cannot resolve the excess carryover you may need to use a smaller sample size (lower concentration).

Cause: Blank sample contamination.

Action: Bake out the system at elevated temperatures. Make sure you are using PerkinElmer cleaned vials and PTFE Silicone septa. If the analysis requires extreme low levels, the vials and caps should be baked out overnight at 120 ºC.

Monitor Vial Integrity

This option is an excellent tool in HS or Trap operation. This tool can help reveal additional problems.

These problems are brought to light by the automatic, dynamic comparison of each vial's decay curve with that in the system memory from the Calibrate Decay Time procedure.

A couple of these problems are analyzed here.

Cause: Loss of Sensitivity - Pre-Injection Peaks

These two problems are attributed to and explained as follows:

Trap Dry Purge low flow: - If there are no leaks but the Trap Dry Purge flow is low (correct flow is 50 ml/min), during the Trap Load step the system will not have transferred the entire amount of the HS vapor to the Trap before the Decay time has elapsed. As a result, less analyte will be injected in the Desorption step. A bad pressure Decay curve is illustrated next.

This problem will result to a Loss of Sensitivity and, possibly, a poor peak repeatability.

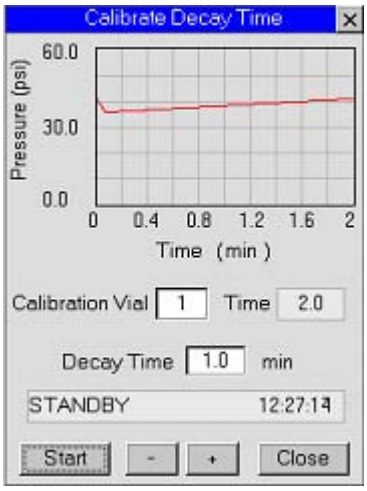


Figure 128

The straight line is a result of too low a Trap Dry Purge flow rate. The flat line is commonly caused by failing to open the trap Dry Purge vent (slide On/Off valve) after a gas leak test. This valve should be left in the ON position (pulled out, toward the operator). A similar result can be obtained with a very low Trap Dry Purge flow rate instead of the recommended 50 ml/min.

Isolation Flow is low: - A misadjusted column Isolation Flow will also decrease sensitivity and cause the Pre-Injection effect.

If the Isolation Flow is less than required, it will not isolate the HS transfer line-GC column from the vial-trap system. Instead, during the Trap Load step, the flow from the pressurized vial to the trap will also branch to the transfer line-GC column, carrying with it part of the HS vapor. This will result in an early peak (Pre-Injection) and the Desorption/Analysis peak will be smaller (loss of sensitivity) with poor repeatability as the trap HS vapor content is now less than that in a regular trap loading.

The next curve indicates a leak in the system or that the Isolation Flow is incorrectly adjusted, or both.

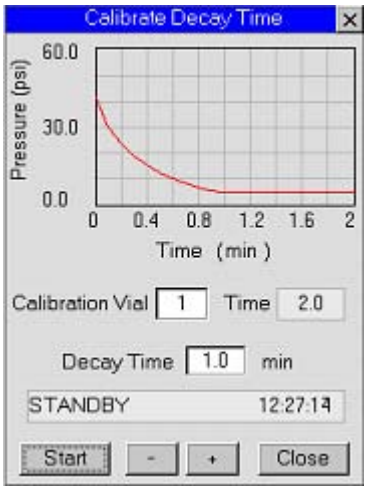


Figure 129

Action: A good rule here is to adjust the Isolation Flow to a value equal to the GC column flow plus 5-10 ml/min. A total value of 12-15 ml/min seem to be adequate for all types of columns and analytical conditions.

In the next figure the Pre-Injection effect is shown

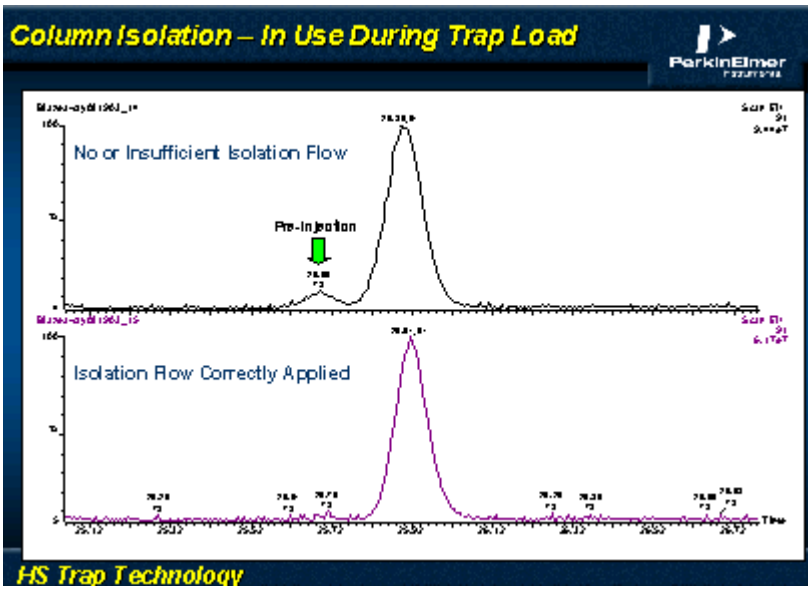


Figure 130

Troubleshooting the Leaks

It has been found that the quickest way to find leaks is to systematically section off the instrument to isolate the leaking area and then to reassemble that section identified as having the leak, one piece at a time until the one defective piece is identified and replaced.

Log Error Messages

Fast pressure decay:

Cause: Vial has a larger sample volume than the calibrated curve, vial is incorrectly capped or a leak has developed in the instrument.

Action:

1. Check sample volume, use a new carefully sealed vial, then check for system leaks.
2. Recalibrate vial decay and check obtained curve with

the provided guide

Slow pressure decay:

Cause: Vial has less sample than the calibrated curve or is at a higher pressure than the calibrated curve. SV9 valve could have failed or the slide valve (Dry Purge outlet) is closed.

Action: Recalibrate vial decay and check the new curve with the provided guide

1. Check slide valve (pulled forward is open).
2. If it is still failing call your PerkinElmer Service engineer to replace the SV9.

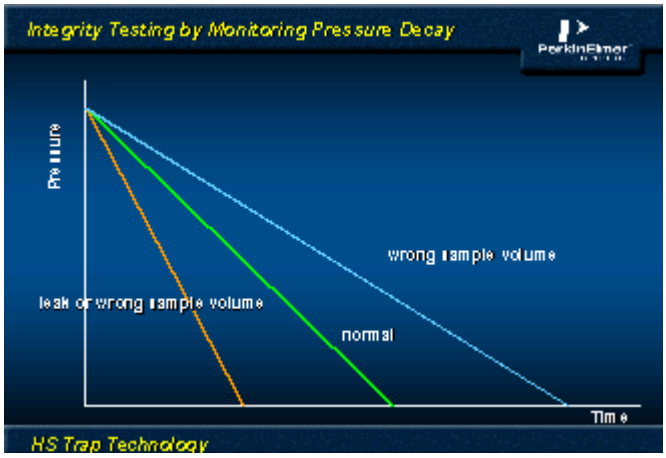


Figure 131 Pressure Decay in Vial with a Fixed Flow Venting

Extended therm: Trap

Cause: The trap was above the low trapping temperature when the sample should have been loaded onto the trap.

Action: Increase the GC cycle time to allow the instrument time to cool the trap down prior to the next injection. Alternatively if the user parameters allow, it may be possible to adjust the thermostat and trap times (desorb and hold) so that the trap is at the appropriate temperature

when required.

See the following figure; the Log tab vial 14.

GC not ready:

Cause: GC did not come ready prior to the trap Desorb step. The instrument will then wait until the GC becomes ready before the trap will fire.

Action: Increase the GC cycle time.

Figure 132

Action: Log Tab: Information on the vials already run

Status Messages

Error Messages	Description
TC @ MIN FAULT ZONE 4	Trap has gone below lowest allowed temperature. Call your PerkinElmer Service Representative.
TC @ MAX FAULT ZONE 4	Trap has exceeded highest allowed temperature. Call your PerkinElmer Service Representative.
HTR RATE FAIL ZONE 4	Trap rise error (rate of temperature change is inappropriate).Call your PerkinElmer Service Representative.
HEATER FAIL ZONE 4	Trap duty error (power is being applied to the heater but inappropriate results achieved). Call your PerkinElmer Service Representative.

The following status messages are displayed during normal operation to indicate the current status of the instrument or the current analysis.

If there is more than one fault occurring at a time, the instrument will cycle through them and display them one at time until they are corrected. The following faults cause the instrument to stop immediately. All heated zones are turned off and a message is shown on the display localizing the error. Record the details of the failure before switching off the instrument.

Fatal error can only be remedied by switching the instrument off with the power switch, waiting ten seconds and switching it on again. The instrument will then restart and attempt to initialize itself.

If the error recurs, please make note of the exact wording of the error message and contact your PerkinElmer service engineer.

Before you contact service, ensure you have the following information:

- What you were doing when the error occurred
- Any corrective action that you have taken
- The exact wording of the error message and any numerical error codes.
- Additionally you should have this manual at hand.

Status	Description
Analyzing	An analysis is in progress
Dry Purging	The trap is being purged with helium.
Economy Mode	The instrument is in the economy mode. The heaters have been switched off and the carrier gas flow has been reduced to the minimum.
Fatal Error	A fatal error is a malfunction from which the instrument cannot recover without user intervention. The instrument may need to be switched off and then turned on again. If the fatal error reoccurs you will need to contact your PerkinElmer Service Representative.
Ready	The instrument is ready to begin an analysis.
Fault	A fault is occurring.
Trap Cond Heat	The instrument is in trap condition mode and the trap is being heated to the set point.
Trap Cond Hold	The instrument is in trap condition mode and the trap is being held at the set temperature.
Trap Heat	The trap is being heated to its high temperature.
Trap Hold	The trap is being held at its high temperature.
Waiting for GC	The HS 40/110 trap is waiting for a GC ready signal before the cold trap is heated.

Table 15 Instrument Status Messages

Status Error Messages

Error Messages	Description
Slow pressure decay vial # x	Vial pressure decayed more slowly than expected, sample volume.
Fast pressure decay vial # x	Vial pressure decayed more quickly than expected, a leaky septum.
Bad pressure decay vial # x	Vial pressure decayed in an inappropriate fashion, but it is hard to qualify.

Table 16 TurboMatrix Headspace Leak Checking Error Messages

Error Messages	Description
Vial x pressure error	The vial standard headspace pressure is incorrect.

Table 17 Standard Headspace Leak Checking Error Messages

Log Error Messages

Error Messages	Description
Run terminated; vial integrity errors.	Run stopped due to missing vials and/or dynamic leak check failures. Three consecutive vials fail the Vial Integrity Test and the run is stopped.
Too many vial integrity errors.	Run stopped due to missing vials and/or dynamic leak check failures. Three consecutive vials fail the Vial Integrity Test and the run is stopped.

Table 18 Vial Integrity Log Error Message

Log Messages	Description
Extended therm: GC not ready	The instrument is not ready.
Extended therm: Cryo temp	Cryo temperature is not within tolerance of its setpoint for injection.
Extended therm: Trap temp	Trap temperature is not within tolerance of its setpoint for trap loading.
Extended therm: Heated zones	Heated zones, oven, needle, transfer line are not within tolerance of their setpoints.
Extended therm: Carrier Pressure	Insufficient carrier pressure.

Table 19 Extended Thermostatting Log Message

NOTE: The extended thermostatting is checked during the pre-injection phase (for standard headspace, that is before pressurization; for headspace, that is before trap load).

Timed Events Messages

Messages	Description
No events to delete	You tried to delete from an empty timed event list.
Read only	You can’t change events during a run.
Event list is full	You can’t add events to a full (32) timed event list.
Duplicate event	You can’t add an event at the same time as another existing event.
Event add error	An error in adding an event.

Table 20 Times Events Messages

Instrument Motor Messages

Messages	Description
Pneumatic PSI Too Low	You need shop air for internal standard. Check air supply for proper delivery pressure.
Magazine Motor Stalled	Check for jammed vial otherwise call PerkinElmer Service.
Elevator Motor Stalled	Check for jammed vial otherwise call PerkinElmer Service.
Oven Motor Stalled	Check for jammed vial otherwise call PerkinElmer Service.
Needle Motor Stalled	Check for jammed vial otherwise call PerkinElmer Service.
Vial Missing	Check for missing vial otherwise call PerkinElmer Service.

Table 21 Instrument Motor Messages

Appendices *8*

Appendix A

Customer Service

Contact PerkinElmer for columns, supplies, accessories, and replacement parts. PerkinElmer offers a full selection of high-quality chromatography data handling products and gas chromatography supplies and columns through the PerkinElmer essentials catalog.

The PerkinElmer Virtual Store is an exciting addition to the analytical instruments suite of interactive resources. The Virtual Store is easy to use and provides an electronic catalog with detailed product descriptions. With one click, orders can be sent via e-mail. Come and browse the store at www.perkinelmer.com.

Customer Service, Supplies, Accessories, and Replacement Parts	
Address:	PerkinElmer Instruments 710 Bridgeport Avenue Shelton, CT 06484-4794 U.S.A
Telephone (US and Canada only):	800-762-4000 8 a.m. to 5 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
Telephone (Worldwide):	Call your local PerkinElmer sales or service office or call PerkinElmer, in Shelton, CT. USA, 800-762-4000
Internet:	http://www.perkinelmer.com
e-mail:	essentials@perkinelmer.com
Applications Notes and Technical Bulletins	800-762-4000 and http://www.perkinelmer.com

Table 22 Contacting PerkinElmer

Appendix B

Warranty Exclusions and Limitations

The following consumable items are excluded from your instrument warranty agreement:

- Fused Silica Transfer Line
- Ferrules
- O-rings
- Filters
- Trap
- Fuses

WARNING

Any attempts to perform installation or maintenance operations that are not detailed in this manual are at the user's own risk.

If user-attempted service results in a visit by a PerkinElmer service engineer, the visit will not be covered by the instrument warranty.

Also excluded from your instrument warranty agreement is damaged caused by:

- corrosion by the sample
- overheating of the sample causing blockage of tubing
- undue stress on parts constructed of glass lined stainless steel tubing (deactivated tube)

Failure to observe the precautions described in this manual will not be covered by the warranty.

Appendix C

Supplies, Accessories and Replacement Parts

Sample Vials and Seals

The safety closures consists of a crimp cap, a star spring and a septum. See *Sample Vials* on page 175 for details.

Headspace Vial, 20 mm clear vials, approximately 22 mL. Caps and septa are not included	
Box of 1000 (crimped top sample vials)	B0104236
Box of 1000 (screw top sample vials)	N9306078
Butyl Rubber Septa for temperatures up to a maximum of 100 ºC; very weak interference peaks with FID; low permeability.	
Pack of 100 Seals	B0159356
Pack of 1000 Seals	B0159357
PTFE Coated Butyl Rubber Septa (Red) for temperatures up to a maximum of 100 ºC; very weak interference peaks with FID; lowest permeability.	
Pack of 100 Seals	B0104239
Pack of 1000 Seals	B0104240
Pack of 1000 Seals, pre-assembled - ready to use	B4000025
Aluminum Coated Silicone Septa for temperatures up to a maximum of 120 ºC; very weak interference peaks with FID; very low permeability.	
Pack of 100 Seals	B0104243
Pack of 1000 Seals	B0104244
Pack of 1000 Seals, pre-assembled - ready to use	B4000022
PTFE Coated Silicone Septa for temperatures up to maximum of 190 ºC; weak interference peaks with FID; very low permeability.	
Pack of 100 Seals	B0104241
Pack of 1000 Seals	B0104242
Pack of 1000 Seals, pre-assembled - ready to use	B4000028

Table 23 Sample Vials and Seals

Tools for Sample Preparation

Description	Part Number
Hand Crimper	N9302785
Cap Removing Tool	N9301270
Benchtop Crimper	N6621006
Vial Gauge	B0151737
Pressure gauge with needle for vials	B0501377
Gauge for checking 22 mL vial dimensions	B0151737

Table 24 Tools for Sample Preparation

Replacement Parts

Description	Part Number
Fuse, 5 x 20MM, 10 A @ 250V, TC	M0417002
Fuse, 5 x 20MM, 5 A @ 250V, TC	M0417038
Jet needle, stainless steel, (3 grooves)	B0510363
Jet needle Siltek®, platinum/iridium, (3 grooves)	B0510364
HS Needle Seal Assembly, without O-Rings	B0500833
O-Rings for Needle Seal Assembly; Pack of 10	B0198110
Deactivated fused silica transfer capillary,5 m, i.d. 0.25 mm	N9301356
Deactivated fused silica transfer capillary, 2.5 m, W. 0.32 mm	B0698537
Extended transfer line (1650 mm), heated	M0413532
Transfer Line, heated	M0413531
Headspace Starter Kit (see the following table)	B0505601

Table 25 Replacement Parts

Set of Injection vials (1000)	B0104236
Red PTFE coated Septa (200)	B0104239
Silicon Schall Coated Kit	B0104241
Silicon Septa Aluminium Coated Kit	B0104243
Hand crimper	N930785
Vial Pressure gauge	B0501377
2 needle Seal Assembly	B0500833
Butyl Rubber Septa (100)	B0159356
Needle O-rings (10)	B0198110
Static Headspace Gas Chromatography Theory and Practice by B. Kolb and L.S. Ettre	N1011210

Table 26 Contents of Headspace (Only) Starter Kit

Stainless Steel Jet Needle (silica steel treated-passivated)	N6700130
Cold trap, empty	M0413627
Needle seal assembly	B0500833
Transfer line tubing (ID= 0.32mm, L=5m)	N9301357
Transfer line f.s. tubing (ID= 0.25mm, L=5m)	N9301356
Needle seal O-rings	B0198110
Glass sample vials (1000)	B0104236
Caps, septa, star springs silicone/ PFTE septa kit	B0104242
Air monitoring trap (glass trap cartridge, filled)	M0413628
Assembly trap desorb tubes	N6700112
Alignment rod	N6700122
Trap removal tool	N6701077

Table 27 Contents of HS 40/110 Trap Spares Kit

Adapter Kits for Gas Chromatographs

Description	**Part Number**
HS adapter kit for HP 5890/6890/6850	B0505977
HS adapter kit for HP 5890/6890/6850 On-Column	B0507944
HS adapter kit for VARIAN 3400/3800 packed column	B0505978
HS adapter kit for VARIAN 3400/3800 capillary column	B0508598
HS adapter kit for FISIONS Series 8000/Trace 2000	B4000012
HS adapter kit for Thermo Trace	N6201030
HS adapter kit for SHIMADZU GC 9Ra/14A / 14B packed columns/17A	B0505889
HS adapter kit for SHIMADZU GC 16	B0508521
HS adapter kit for SHIMADZU GC 14A/B	B4000033
HS adapter kit for CARLO ERBA VEGA/ MEGA	B0506603
HS adapter kit for GERSTEL CIS	B0506539

Table 28 Adapter Kits for Other Gas Chromatographs

Appendix D

Reference Material

Headspace Gas Chromatography

- Hachenberg, H., Schmidt, A.R, *Gas Chromatographic Headspace Analysis,* Heyden & Son Ltd., (1977).
- Kolb, B., Auer, M., Pospisil, P, *Applications of Gas Chromatographic Headspace Analysis,* Applied Chromatography No. 20E, PerkinElmer (1978).
- Kolb, B., *Applied Headspace Gas Chromatography,* Heyden & Son Ltd., (1980).
- Kolb, B., Auer, M., Pospisil, *P., Multiple Headspace Extraction, a Procedure for Quantitative Analysis of Volatile Compounds in Solid Samples,* Applied Chromatography No. 35E, PerkinElmer (1981).
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- Kolb, B., Pospisil, *R, Applications of Gas Chromatographic Headspace Analysis,* Applied Chromatography No. 33E, PerkinElmer (1984).
- Kolb, B., *Analysis of Food Contaminants by Headspace Gas Chromatography,* in Gilbert, J.: Analysis of Food Contaminants, Elsevier Ltd., London - New York (1984).
- Kolb, B., Auer, M., Pospisil, *P, Quantitative Headspace Analysis of Solid Samples, a Classification of Various Sample Types,* Chromatographia 19, 113 (1984).
- Kolb, B., *Headspace Gaschromatographie mit Kapillar-Trennsdulen, Praktische instrumentelleAnalytik* LaborPraxis, Vogel, Wiirzburg (1986).

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- Bretherik, L., *Bretheriks Handbook of Reactive Chemical Hazards,* 4th ed., Butterworth & Co. Ltd, London 1990.
- Bretherik, L., *Hazards in the Chemical Laboratory,* 3rd ed., Royal Society of Chemistry, London, 1981.
- Furr, K., ed., CRC *Handbook of Laboratory Safety,* 3rd ed., The Chemical Rubber Co. Press, Florida, 1990.
- Data Sheets provided by chemical manufacturers, e.g.: USX Material Safety Data Sheets (MSDS); FRG, DIN-Sicherheitsdatenbldtter; GB, Hazard Data Sheets.
- *Prudent Practices for Handling Hazardous Chemicals in Laboratories,* National Research Council, National Academic Press, Washington D.C., USA, 1981.
- Sax, N., ed., *Dangerous Properties of Industrial Materials,* 7th ed., Van Nostrand Reinhold, New York, 1989.

Multiple Headspace Extraction

The theoretical principles of Multiple Headspace Extraction are presented in the following publications:

- Hachenberg, H., Schmidt, A.P, *Gas Chromatographic Headspace Analysis,* Heyden & Son Ltd., (1977).
- Kolb, B., Auer, M., Pospisil, P, *Applications of Gas Chromatographic Headspace Analysis,* Applied Chromatography No. 20E, PerkinElmer (1978).
- Kolb, B., *Applied Headspace Gas Chromatography,* Heyden & Son Ltd., (1980).
- Kolb, B., Auer, M., Pospisil, P, *Multiple Headspace Extraction, a Procedure for Quantitative Analysis of Volatile Compounds in Solid Samples,* Applied Chromatography No. 35E, PerkinElmer (1981).
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- Kolb, B., Pospisil, P., *Applications of Gas Chromatographic*

HeadspaceAnalysis, Applied Chromatography No. 33E, PerkinElmer (1984).

- Kolb, B., *Analysis of Food Contaminants by Headspace Gas Chromatography,* in Gilbert, J.: Analysis of Food Contaminants, Elsevier Ltd., London - New York (1984).
- Kolb, B., Auer, M., Pospisil, P, *Quantitative Headspace Analysis of Solid Samples, a Classification of Various Sample Types,* Chromatographia 19, 113 (1984).
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- Braithwaite, A. and Smith, F.J. *Chromatographic Methods, Fifth Edition,* Glasgow: Blackie Academic and Professional, an imprint of Chapman and Hall, 1996
- Kolb, Bruno and Ettre, Leslie S. *Static Headspace Gas Chromatography, Theory and Practice,* New York: Wylie-VCH, Inc. 1997

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