# Clarus 600/560 D MS Hardware Guide

#### **Release History**

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Any comments about the documentation for this product should be addressed to:

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# Warnings and 1 Safety Information



## **Conventions Used in this Manual**

Normal text is used to provide information and instructions.

**Bold** text refers to text that is displayed on the touch screen.

All eight digit numbers are PerkinElmer part numbers unless stated otherwise.

#### Notes, warnings and cautions

Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

**NOTE:** A note indicates additional, significant information that is provided with some procedures.

#### Terminology

Throughout the manual, the term 'mass spectrometer' or MS specifically refers to the Clarus MS; while for 'GC' Clarus GC is implied.

#### Caution

CAUTION

We use the term CAUTION to inform you about situations that could result in **serious damage to the instrument** or other equipment. Details about these circumstances are in a box like this one.

D	<b>Caution (Achtung)</b> Bedeutet, daß die genannte Anleitung genau befolgt werden muß, um einen <b>Geräteschaden</b> zu vermeiden.
DK	<b>Caution (Bemærk)</b> Dette betyder, at den nævnte vejledning skal overholdes nøje for at undgå en <b>beskadigelse af apparatet</b> .
E	<b>Caution (Advertencia)</b> Utilizamos el término <b>CAUTION</b> (ADVERTENCIA) para advertir sobre situaciones que pueden provocar <b>averías graves en este equipo</b> o en otros. En recuadros éste se proporciona información sobre este tipo de circunstancias.
F	<b>Caution (Attention)</b> Nous utilisons le terme <b>CAUTION</b> (ATTENTION) pour signaler les situations susceptibles de provoquer de <b>graves détériorations de</b> <b>l'instrument</b> ou d'autre matériel. Les détails sur ces circonstances figurent dans un encadré semblable à celui-ci.
	<i>Caution (Attenzione)</i> <i>Con il termine CAUTION (ATTENZIONE) vengono segnalate</i> <i>situazioni che potrebbero arrecare</i> <b>gravi danni allo strumento</b> o ad <i>altra apparecchiatura. Troverete informazioni su tali circostanze in un</i> <i>riquadro come questo.</i>
NL	<i>Caution (Opgelet)</i> Betekent dat de genoemde handleiding nauwkeurig moet worden opgevolgd, om <b>beschadiging van het instrument</b> te voorkomen.
P	<b>Caution (Atenção)</b> Significa que a instrução referida tem de ser respeitada para evitar a <b>danificação do aparelho</b> .
WARNING	<b>Warning</b> We use the term WARNING to inform you about situations that could result in <b>personal injury</b> to yourself or other persons. Details about these circumstances are in a box like this one.

<ul> <li>Warning (Advarsel) Betyder, at brugeren kan blive kvæstet, hvis anvisningen ikke overholdes.</li> <li>Warning (Peligro) Utilizamos el término WARNING (PELIGRO) para informarle sobre situaciones que pueden provocar daños personales a usted o a otras personas. En los recuadros como éste se proporciona información sobre este tipo de circunstancias.</li> <li>Warning (Danger) Nous utilisons la formule WARNING (DANGER) pour avertir des situations pouvant occasionner des dommages corporels à l'utilisateur ou à d'autres personnes. Les détails sur ces circonstances sont données dans un encadré semblable à celui-ci.</li> <li>Warning (Pericolo) Con il termine WARNING (PERICOLO) vengono segnalate situazioni che potrebbero provocare incidenti alle persone. Troverete informazioni su tali circostanze in un riquadro come questo.</li> <li>Warning (Waarschuwing) Batelent dat wanneer de amograda agneuiring vict in geht wordt</li> </ul>	D	<b>Warning (Warnung)</b> Bedeutet, daß es bei Nichtbeachten der genannten Anweisung zu einer <b>Verletzung</b> des Benutzers kommen kann
<ul> <li>Warning (Peligro)         <ul> <li>Utilizamos el término WARNING (PELIGRO) para informarle sobre situaciones que pueden provocar daños personales a usted o a otras personas. En los recuadros como éste se proporciona información sobre este tipo de circunstancias.</li> </ul> </li> <li>F Warning (Danger)         <ul> <li>Nous utilisons la formule WARNING (DANGER) pour avertir des situations pouvant occasionner des dommages corporels à l'utilisateur ou à d'autres personnes. Les détails sur ces circonstances sont données dans un encadré semblable à celui-ci.</li> <li>Warning (Pericolo)             <ul> <li>Con il termine WARNING (PERICOLO) vengono segnalate situazioni che potrebbero provocare incidenti alle persone. Troverete informazioni su tali circostanze in un riquadro come questo.</li> <li>Nu</li> <li>Warning (Waarschuwing)             Patekent dat wannear de genoamde agnuizing piet in geht wordt         </li> </ul></li></ul></li></ul>	DK	<b>Warning (Advarsel)</b> Betyder, at brugeren kan blive <b>kvæstet</b> , hvis anvisningen ikke overholdes.
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genomen, dit kan leiden tot <b>verwondingen</b> van de gebruiker.	NL	<b>Warning (Waarschuwing)</b> Betekent dat, wanneer de genoemde aanwijzing niet in acht wordt genomen, dit kan leiden tot <b>verwondingen</b> van de gebruiker.
P Warning (Aviso) Significa que a não observância da instrução referida poderá causar um ferimento ao usuário.	P	<b>Warning (Aviso)</b> Significa que a não observância da instrução referida poderá causar um <b>ferimento</b> ao usuário.

## **Customer Service**

This instrument is manufactured by:

PerkinElmer Inc. 710 Bridgeport Avenue Shelton, Connecticut 06484-4794 U.S.A.

Tel: 1 (800) 762-4000 Internet: http://www.perkinelmer.com

# Electromagnetic Compatibility (EMC)

## **Regulatory Information**

## United States (FCC)

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference at his own expense.

**NOTE:** Changes or modifications not expressly approved by PerkinElmer could cause the instrument to violate FCC (U.S. Federal Communications Commission) emission regulations, and because of this violation could void the user's authority to operate this equipment.

### Europe

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

The Clarus MS contains protective circuitry. Contact PerkinElmer Service before performing any AC line tests.

CAUTION

## **Electrical Symbols Used on ClarusMS**



Off position of the main power switch.

On position of the main power switch.



Warning: Risk of electric shock.



Warning: Hot surface.



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

## Label Location and Content



Figure 1 Front View of Clarus 600 MS



Front View of Clarus 560 D



Figure 2a Rear View of the Clarus 600 MS



Figure 2b Rear View of the Clarus 560 D MS

## **Clarus MS Safety Practices**

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

The Mass Spectrometer should be used in accordance with the instructions provided in the user's manuals and tutorial supplied with the instrument. If used otherwise, the protection provided by the instrument may be impaired.



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



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

## Generic Warnings

Before installing or operating the MS, read the following topics concerning hazards and potential hazards. Ensure that anyone involved with installation and/or operation of the MS is knowledgeable in both general safety practices for the laboratory and safety practices for this instrument. Get advice from your safety engineer, industrial hygienist, environmental engineer, or safety manager before you install or use this instrument.

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

## Moving the Clarus MS



Depending on the Clarus 600 MS GC pump option selected, the instrument weight will range from 46.8 kg (102 lb) to 49.9 kg (110 lb).

The Clarus 560 D MS is 48 kg (105 lb) in weight.

The mass spectrometer requires two people to safely lift it and should be lifted from the bottom. Use the following lifting posture to avoid back injury: With knees bent, simultaneously lift the instrument out of the carton as you end up in a standing position.



Connect the mass spectrometer to an AC line power outlet that has a protective ground connection. To ensure satisfactory and safe operation of the mass spectrometer, it is essential that the protective ground conductor (the green/yellow lead) of the line power cord is connected to a true electrical ground. Any interruption of the protective ground conductor, inside or outside the mass spectrometer, or disconnection of the protective ground terminal may impair the protection provided by the mass spectrometer.



Never operate the mass spectrometer with any covers or parts removed.



Do not make adjustments, replacements or repairs to the mass spectrometer except as described in this manual. Only a PerkinElmer Service Representative or similarly trained and authorized person should be permitted to service the mass spectrometer.

Ensure that the power cord is correctly wired and that the ground leads of all electrical units (for example, recorders, integrators) are connected together via the



circuit ground to earth. Use only three-prong outlets with common earth ground connections.

- Servicing of electrical components within the mass spectrometer should be performed only by a PerkinElmer Service Representative or similarly trained and authorized person.
- Servicing of the incoming AC power line components in your laboratory should be performed only by a licensed electrician.



*Electrical shock hazard. To prevent electrical shock, disconnect the power cord from the AC outlet before servicing.* 



Disconnect AC power cord from outlet before removing any cover or parts. Do not operate the instrument with any covers or parts removed.

Under no circumstances should circuit boards be removed or inserted unless the instrument is disconnected from line power.

## Decontamination and Cleaning

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.

## **Compressed Gases**



**Compressed Gases.** 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.

Avoid banging the valves, and ensure that the correct valves and gauges are installed. It is recommended that gas cylinders be stored and placed outside the laboratory and connected to the instrument through specially cleaned copper tubing. Take care not to kink or stress the gas tubing. For safety, cylinders must be firmly clamped in an upright position.



*Explosive hazard.* When using hydrogen, methane or isobutane, special care must be taken to avoid buildup of explosive gas mixtures either in the GC oven or the mass spectrometer vacuum manifold.

Ensure that all hydrogen line couplings are leak-free and do not allow hydrogen to vent within the oven.

## Ventilation



Hazardous vapors. When analyzing hazardous compounds, such as pesticides, or running in the chemical ionization (CI) mode, it is necessary to vent the mass spectrometer effluent from the forepump exhaust into a fume hood or charcoal trap.



**Toxic Gases-Fume Ventilation System.** Without adequate ventilation potentially toxic vapors can build up in the laboratory. Your laboratory **must** have reliable fume ventilation system before you use this instrument.

Adequate ventilation must be provided, particularly if a liquid nitrogen or carbon dioxide subambient accessory is in constant use. The area underneath the bench (around the forepump) should be well ventilated. An oil separation filter and charcoal trap should be installed at the outlet of the forepump exhaust to prevent contamination if fume hood venting is unavailable.

To ensure adequate cooling of the instrument electronics, do not obstruct the gap at the base of the Clarus MS/Clarus GC, and if practical, leave a minimum 6 inch clearance between each instrument in the system (for example, the ATD or HS 40XL). This does not include the Clarus MS/Clarus 600 GC as they are connected together.

## Heated Zones



**Risk of burns**. Never touch a heated mass spectrometer transfer line or a GC injector cap with unprotected (bare) fingers.

Heated zones should be treated with caution, for example, the transfer line, injector caps, and detectors. In addition, the detector cover may get hot, especially if flame ionization detectors are operated at high temperatures. As a general rule, allow heated zones to cool before attempting to work in the GC oven, on the transfer line, on an injector, around the detector areas or inside the mass spectrometer manifold. Cooling of the transfer line may require a wait of  $\frac{1}{2}$  to 1 hour.



## Using Hydrogen, Methane or Isobutane



## Using Ammonia Gas



Hazardous gas vapors. When using ammonia gas while running in the chemical ionization (CI) mode, it is necessary to vent the mass spectrometer effluent from the forepump exhaust into a fume hood or outside the building.

## Hazardous Chemicals



Hazardous chemicals. Before using samples, thoroughly familiarize yourself with all hazards and safe handling practices. Observe the manufacturer's recommendations for use, storage and disposal. These recommendations are normally provided in the Material Safety Data Sheets (MSDS) supplied with the solvents, chemicals, and pump oils.

Be aware that the chemicals that you use in conjunction with the mass spectrometer may be hazardous. 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). MSDSs give information on physical characteristics, precautions, first aid, spill clean up and disposal procedures. Familiarize yourself with the information and precautions contained in these documents before attempting to store, use or dispose of the reagents. 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.



When using toxic samples, the mechanical pump oil is toxic waste.



Some chemicals used with the mass spectrometer may be hazardous or may become hazardous after completion of an analysis. The responsible person (for example, the Lab Manager) must take the necessary precautions to ensure that operators and people in the surrounding workplace 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. Venting for fumes and disposal of waste must be in accordance with all national, state and local health and safety regulations and laws.



# Definitions in Warnings for Hazardous Chemicals

Responsible body:	Individual or group responsible for the use and maintenance of equipment, and for ensuring that operators are adequately trained. [per EN/IEC 61010-1].
Operator:	Person operating equipment for its intended purpose. [per EN/IEC 61010-1].
OSHA:	Occupational Safety and Health Administration (United States).
ACGIH:	American Conference of Governmental Industrial Hygienists.
COSHH:	Control of Substances Hazardous to Health (United Kingdom).

# Temperature, Humidity and Environment

## **Operating Conditions**

CAUTION	The Clarus MS is designed for indoor use only in a laboratory environment that is clean and is free of drafts, direct sunlight and vibration.
	Do not operate the mass spectrometer in a Cold Room or a refrigerated area. Clarus MS operates under the following conditions:
	Ambient temperature is 10 °C to 35 °C (50 °F and 95 °F) with a variability of less than $\pm 4$ °C ( $\pm 7$ °F).
CAUTION	The Clarus MS will operate safely between $5^{\circ}C$ and $40^{\circ}C$ (41 °F and 104 °F).
	If operating at ambient temperatures between 30°C and 35 °C, you will need the water-cooling option for the turbopump.
	Ambient relative humidity is 20 % to 80 % non-condensing.
	Operating altitude is in the range of 0 to 2000 m.



The mass spectrometer is not designed for operation in an explosive environment. The laboratory should be free of flammable, explosive, toxic, caustic, or corrosive vapors or gases and should be relatively free of dust.

**Pollution Degree:** 

Clarus MS will operate safely in environments that contain non-conductive foreign matter up to Pollution Degree 2 in EN/IEC 61010-1.

## **Storage Conditions**

The mass spectrometer may be stored under the following conditions:

- Ambient temperature is -20 °C to +60 °C (-4 to 140 °F).
- Ambient relative humidity is 20 to 80 %, non-condensing.
- Altitude is in the range 0 to 12000 m.
- The instrument is stored in an upright position.

# General Laboratory Safety

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

# 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.

# Pre-Installation Requirements

	Size	Weight
Clarus 600 MS	32 cm (13 in.) wide x 50 cm (20 in.) high x 77 cm (30 in.) deep	Depending on the pump option selected, the weight will range from 46.8 kg (102 lb) to 49.9 kg (110 lb).
Clarus 560 D MS	32 cm (13 in.) wide x 46 cm (18 in.) high x 77 cm (30 in.) deep	48 kg (105 lb)
Forepump	30.5 cm (12 in.) wide x 44 cm (17.3 in.) high x 72 cm (28.4 in.) deep.	25.9 kg (57 lb)
Clarus 600 GC	99 cm (39 in.) wide x 53 cm (21 in.) high x 82 cm (32 in.) deep	49 kg (108 lb)
Autosampler Tower	13 cm (5 in.) wide x 36 cm (14 in.) high x 24 cm (9.5 in.) deep	4.5 kg (10 lb)
Physical Configuration	Single unit for use on standard laboratory bench that can be interfaced to a computer and printer.	
Bench Space	The laboratory bench should be sturdy enough to support the full weight of the GC/MS as well as additional equipment (for example, computer and/or printer). Expect the total weight of the GC/MS and accessory equipment to weigh at least 159 kg (350 lb). Allow a minimum clearance of 15 cm (6 in.) on each side, 22.9 cm (9 in.) at the rear, and 137.2 cm (54 in.) at the top of the GC/MS. If this is not possible, install the GC/MS on a bench that has wheels. The bench requires an area underneath for the forepump. Do not position the Clarus 600 MS so that it is difficult to operate the AC power on/off switch on the lower left side of the instrument in case of a malfunction of the instrument. For the Clarus 650 D the AC power on/off switch on the lower left side of the instrument in the switch on the lower left side of the instrument in case of a malfunction of the instrument.	
Peripherals, Printers etc.	Allow at least 94 cm (36 in.) on either side of th additional equipment (for example, the compute	e instrument to accommodate er).

# Laboratory Space Requirements

# Environmental Requirements

Pollution Degree	This instrument will operate safely in environments that contain non-conductive foreign matter up to Pollution Degree 2 as defined in EN/IEC 61010-1.
Laboratory Environment	Install the GC/MS in an indoor laboratory environment that is clean and free of drafts and direct sunlight.
	The laboratory should be free of flammable, explosive, toxic, caustic or corrosive vapors or gases, and should be relatively free of dust.
	The ambient laboratory temperature should be between 10 $^{\circ}$ C and 30 $^{\circ}$ C (50 $^{\circ}$ F and 86 $^{\circ}$ F) for Clarus 600 C, 600 T, and 600 S systems unless the turbomolecular pump is water cooled, and between 10 $^{\circ}$ C and 35 $^{\circ}$ C (50 $^{\circ}$ F and 95 $^{\circ}$ F) for Clarus 600 D, or for Clarus 600 C, 600 T, and 600 S systems with water cooling.

# **Power Requirements**

Clarus MS	All electrical supplies must be smooth, clean, and free of line transients greater than 40 V peak to peak, and must meet and remain within the following tolerances: 120 VAC $\pm 10$ % @ 50/60 Hz $\pm 1$ % 1000 VA maximum 230 VAC $\pm 10$ % @ 50/60 Hz $\pm 1$ % 1000 VA maximum Add 100 VA for the computer and 108 VA for a printer
Clarus GC	All electrical supplies must be smooth, clean, and free of line transients greater than 40 V peak to peak, and must meet and remain within the following tolerances: For GC with slow heating rate as standard; 120 VAC $\pm$ 10% @ 50/60 Hz $\pm$ 1% @ 20 Amps, 2400 VA maximum 230 VAC $\pm$ 10% @ 50/60 Hz $\pm$ 1% @ 10 Amps, 2400 VA maximum For GC with optional oven heater for fast heating rate; 220 VAC $\pm$ 5% @ 50/60 Hz $\pm$ 1% @ 15 Amps, 3120 VA maximum 230 VAC $\pm$ 5% @ 50/60 Hz $\pm$ 1% @ 16 Amps, 3120 VA maximum 240 VAC $\pm$ 5% @ 50/60 Hz $\pm$ 1% @ 13 or 16 Amps, 3120 VA max Instruments and peripherals must not be connected to circuits with large inductive or large and frequent loads (for example, large motors, discharge lamps, photocopy systems, radio transmitters, etc.).

Power Outlets	<i>Clarus MS:</i> A minimum requirement of a power line separate from the GC at 15 amps or greater.
	<i>Clarus GC</i> : A minimum of one dedicated 120 VAC outlet at 20 A or one 230 VAC outlet at 10 A (minimum) is required for the Standard GC. When the optional oven heater is ordered, the outlets will be as indicated above.
	Additional equipment, such as computers and printers, should be connected per their specifications.

Gas Delivery Lines	Copper tubing that is free of grease, oil and organic material must always be used with the Clarus 600 MS on all gas lines, except ammonia reagent gas. Ammonia reagent gas requires stainless steel. Solvent-washed copper tubing must be used to avoid contamination of the Gas Chromatograph. Suitable solvents are acetone or dichloromethane (do not use if negative chemical ionization is planned) followed by methanol. Clean helium or nitrogen should be used to blow any residual solvent from the tubing. Cap all unused tubing. Care must be taken not to kink or overstress the gas delivery lines. Strain relief consisting of two one inch coils of tubing should be installed at every gas line connection.
Gas Cylinders	All gas cylinders should be firmly clamped to a suitable surface. Gas cylinders should be located outside of the laboratory whenever possible, and should always be stored and operated in a vertical position.
Hydrogen	Ensure that all hydrogen lines and connections are leak-free. When using a hydrogen tank, install an in-line hydrogen snubber (part number 00090038) between the tank regulator and the delivery tubing.
Ventilation	Always provide adequate ventilation. When analyzing hazardous compounds such as pesticides, it may be necessary to arrange to vent the mass spectrometer effluent from the forepump into a fume hood. To prevent contamination if a fume hood is unavailable, an oil separation filter should be installed at the outlet of the forepump vented to a fume hood or an oil mist separator (Alcatel 68316) with a charcoal trap (Koby KA1). An acceptable alternative is to attach a <sup>1</sup> / <sub>2</sub> inch Tygon tube and vent to a hood. Pump oil vapor is considered toxic and must be vented properly.

### Clarus 600/560 D MS Hardware Guide

## **Gas Requirements**

Carrier gases used with the mass spectrometer require a minimum purity of 99.999%. Gas cylinders should be located outside of the laboratory whenever possible, and should always be stored and operated in the vertical position.

CAUTION	For all gases delivered to the mass spectrometer, always use copper tubing that is free of grease, oil, and organic material. If in doubt about the
	condition of your tubing, clean it before use.

#### Gases

GC/MS Carrier Gases:	Minimum purity of 99.999% for helium or hydrogen. Carrier gas tubing should be ultra-clean.
Helium	A number 1A (200 ft <sup>3</sup> ) gas cylinder should be used for all carrier gases with a high-purity, stainless-steel diaphragm, two-stage regulator. Filter through a moisture filter and/or hydrocarbon trap and de-oxo filter designed for MS.
	Gas delivery pressure to the GC should be $70 - 100$ psi (483 - 689 kPa). Do not exceed 100 psi (689 kPa) on the carrier gas inlet.
Reagent Gases:	Minimum purity of 99.999% for methane, minimum purity of 99.98% for isobutane, minimum purity of 99.998% for ammonia. Carrier gas tubing should be ultra-clean.
	The gas delivery pressure required is 15 psi (103 kPa) to the bulkhead fitting (1/8 in. Swagelok) on the mass spectrometer.
Ammonia	If ammonia is used for chemical ionization, all fittings and tubing must be stainless steel to avoid corrosion. A single-stage regulator is required for ammonia, rated for corrosive service. Also, the forepump must be vented to a fume hood or outside the building.
Methane and Isobutane	A high-purity, stainless-steel diaphragm, two-stage regulator is required for methane and isobutane with a final delivery pressure of 15 psi (103 kPa). Clean tubing must be used. It must be solvent-washed and nitrogen-dried. The bulkhead connector at the rear of the instrument is a 1/8 in. Swagelok fitting.
	The use of commercial gas purifiers for reagent gas is recommended.
### Computer and System Software Requirements

To ensure that your system performs at the expected high level, your computer must be configured to the minimum capabilities indicated below.

These requirements may be updated as the requirements for TurboMass software and/or Microsoft Windows XP SP2 are changed. Consult the latest Product Description List for current requirements.

**NOTE:** This guide does not cover the installation and configuration of your computer. If you have purchased a complete system from PerkinElmer, the computer will be configured by your Service Engineer during product installation.

### **PC Requirements**

The TurboMass software is installed at PerkinElmer prior to shipment and tested using the following minimum PC system specifications. If you need to reinstall the software, verify that the PC meets the following minimum requirements:

- Dell OptiPlex 745, GX620, GC270, or GX 280
- Intel<sup>®</sup> Pentium processor
- 512 MB of Random Access Memory (RAM)
- High Color (16 bit) at 1024 x 768 SVGA
- Hard disk with 2.0 GB free space
- 8x speed CD-ROM drive
- 1 RS-232 port
- 2 RJ-45 10/100Base-T ports
- A keyboard and PS/2®-style mouse

#### **Operating System**

Windows XP SP2

#### Software

TurboMass Software.

## Instrument Firmware Versions

Internal dotLINK

#### **Printers**

•

- HP LaserJet 4200 Printer Series (HP 4200, 4210, and 4250)
- HP DeskJet 5650 Color InkJet Printer

**NOTE:** Using any printers other than the ones recommended above may not correctly display the Communiqué reports.

# **Pre-Installation Checklist**

MODEL:

\_\_\_\_\_ DATE: \_\_\_\_\_

CUSTOMER:

SPO#:

Requirements	ОК	Needs Prior To Installation
Customer Responsibility		
Lab Space Requirements		
Power Requirements		
Gas Requirements		
Environmental Requirements		
Safety Requirements		
Preparation of Samples (Customer Responsibility)		
Computer Configuration		
Customer Experience		

Clarus 600/560 D MS Hardware Guide

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# Introduction **2**



## Preface

The Clarus MS is a benchtop mass spectrometer designed with the user in mind. The small profile of combination Gas Chromatograph and Mass Spectrometer (GC/MS) allows it to fit on a standard six foot long laboratory bench. Sophisticated software controls the GC/MS from a Windows XP SP2 computer.

#### System Overview

The system consists of:

- Clarus GC.
- Clarus MS.
- Computer.
- TurboMass Software.
- Foreline Pump.

#### Summary of this Guide

Thoroughly read and understand the Safety and Regulatory Information chapter before using the mass spectrometer.

Chapter 1:	Warnings and Safety Information						
	Contains all of the safety information and topics to comply with EN/IEC 61010.						
Chapter 2:	Introduction						
	Provides an instrument overview and the references to related documentation.						
Chapter 3:	About the Clarus 600/560 D System						
	Describes each of the components in the system and includes a list of items to check before using the instrument.						
Chapter 4:	Maintenance						
	Contains preventive and routine maintenance procedures that typical users can perform.						
Chapter 5:	Troubleshooting						
	Provides helpful troubleshooting tips and a table to help you identify and solve typical problems.						

#### **Related Documentation**

The Clarus 600/560 D family of manuals includes the following:

• *Clarus 600/560 D MS Tutorial* (part number 09936769): The tutorial provides a step-by-step guide to performing a number of tasks using the instruments and software.

- *Software User's Guide* (part number 09936767): A comprehensive manual describing the functionality of each part of the TurboMass software. It describes the keys and fields on each screen.
- *Clarus 600/560 D MS Hardware Manual* (part number 09936768): Contains the required safety and regulatory information required for EN/IEC 61010. It contains an overview of mass spectrometry and of each component in this system; a pre-operational checklist, typical user maintenance and a troubleshooting guide.
- *Service Manual (not included)*: Contains information for trained service engineers to completely service the Clarus.

#### About Part Numbers Listed in this Manual

The part numbers listed in this manual are available from PerkinElmer's catalog service.

#### Supplies, Accessories and Replacement Parts

Supplies, accessories, and replacement parts can be ordered directly from PerkinElmer's catalog service. PerkinElmer offers a full selection of high-quality chromatography data handling products and gas chromatography supplies and columns through *e*-essentials.

To place an order for supplies and many replacement parts, request a free *e*-essentials catalog, or ask for information:

Telephone:

- U.S. only: Call toll free 1-800-762-4000, 8:30 a.m. to 7 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- Worldwide: Call your local PerkinElmer sales or service office or call PerkinElmer, Shelton, CT USA

Internet: <u>http://www.perkinelmer.com</u>

Clarus 600 MS User's Guide



# About the Clarus 600/560 D System **3**



#### About the Clarus 600/560 D System

The Clarus 600/560 D mass spectrometer is a compact benchtop instrument that produces positive identification and quantitation of compounds separated by the Clarus 600/560 D GC. Even if the compounds coelute, the mass spectrometer can still positively identify and quantitate each compound. Clarus 600 MS is designed to run analyses that best identify your sample by using the electron impact (EI) or chemical ionization (CI) mode. The Clarus 560 D MS runs analyses that identify your sample by using the electron impact (EI) and the electron impact (EI) mode.

The Clarus 600/560 D system is controlled by a PC using TurboMass Software. The application runs in a Microsoft Windows XP SP2 operating environment. The software user interface contains color graphics and provides full user interaction with either the keyboard or the mouse. TurboMass completely controls the GC/MS system from tuning and data acquisition (scanning or selected ion recording mode), through quantifying your results. Complete operating instructions of all TurboMass controls are in the *TurboMass Software Guide* (part number 0993-6767), supplied with the system.

A high-performance, research-grade analytical quadrupole mass analyzer with a quadrupole prefilter assembly transmits only those ions having your selected mass-to-charge ratio. The prefilter rod set improves sensitivity and protects the analytical quadrupole rods from contaminating ion deposits. Ions emerging from the quadrupole mass analyzer are converted to photons and detected by the photomultiplier detector system. The low noise photomultiplier typically operates with a gain of 10<sup>5</sup>.



Figure 3 Clarus MS with Clarus GC

The system consists of two major components: Clarus MS and the Clarus GC. Brief descriptions of each major component follow.

# Clarus 600/560 D GC

The Clarus 600/560 D Gas Chromatograph is a dual-channel, temperatureprogrammable gas chromatograph (GC). It is available in many configurations, such as with or without, an autosampler, programmable pneumatic control (PPC), and a variety of injector/detector combinations to provide you with total GC flexibility. The Clarus 600/560 D GC is microprocessor controlled, where you enter the operating parameters and view the prompting text and monitor instrument functions on a large full-color touch screen display.



Figure 4 Clarus 600/560 D GC

*The Programmed Pneumatic Control (PPC) Version of the Clarus 600/560 D GC* is used where the carrier gas and detector gases are monitored and controlled by the microprocessor, thereby producing a fully automated system that is capable of managing all pneumatic functions within the gas chromatograph.

#### Clarus 600 MS Hardware Guide

The Clarus 600/560 D GC can store up to five GC methods. Methods can be generated, copied, deleted, edited, set up, and printed. These methods are normally developed and stored on the TurboMass data system. The automatic liquid autosampler can run up to 15 injections per vial from as many as 82 vials and one priority vial using one or two autosampler programs (if not under TurboMass control). In the latter case, a different GC method can be used by each program if desired.

PPC provides real-time digital readouts to simplify setting carrier gas pressures and flows.



Figure 5 Clarus 600 Mass Spectrometer

# GC Interface (Transfer Line)

The detector end of a capillary GC column in the Clarus 600/560 D GC oven is inserted through a temperature-controlled transfer line and optimally positioned so that the column end is flush with the inner wall of the EI or CI ion source. The transfer line is temperature controlled by Clarus MS and has a 350 °C upper limit. If the Clarus 600 GC detects improper operation (for example, no carrier gas) and goes into an alarm condition, it will turn off the temperature to the transfer line.

**CAUTION** Do not use metal capillary columns in the transfer line. They may electrically short-out the source.



Figure 6 The transfer line

# **Reference Gas Inlet**

The reference gas inlet system consists of a glass bulb filled with heptacosa (FC43) connected to tubing which directs it to the ion source. You can switch the reference gas solenoid valve on and off and also purge the reference gas lines from the Tune screen.

Figure 7 and 8 shows the components or assemblies that comprise the Clarus 600/560 D MS with the manual vent switch (Clarus 600 D and Clarus 560 D) or the CI adjustment valve (Clarus 600 C) configuration (see the following two pages).



Figure 7 Components of the Clarus 600/560 D MS and a detail of the ion optics path (manual vent valve for diffusion pump configuration)

#### Clarus 600 MS Hardware Guide



Figure 8 Components of the Clarus 600 MS and a detail of the ion optics path (CI needle valve for large turbo pump configuration)

# Ion Optics Path

Ion Source	The ion source consists of a removable EI or CI inner source and a fixed outer source for the Clarus 600. The Clarus 560 D uses an EI source only. In the EI source, molecules exit the column where they are bombarded by electrons from the filament and ionized into positive and negative ions as well as neutral species. The positive electron trap attracts the negative ions and electrons to the repeller that directs the positive ions out of the inner source through focusing lens to the mass analyzer. Those remaining molecules and neutral fragments are pumped away by the vacuum. Heaters in the outer source raise the source temperature high enough to prevent sample molecules from condensing in the source and minimize any contamination.
Mass Analyzer	The mass analyzer element of this high performance quadrupole mass spectrometer is a finely machined assembly that has been precisely aligned using specialized equipment. <i>Under no</i> <i>circumstances</i> should the main analyzer rod set assembly ever be dismantled. The mass spectrometer is fitted with a quadrupole prefilter
	assembly that is designed to protect the main analyzer by intercepting the majority of any contamination. As a consequence, the main analyzer should never require cleaning.
	On occasion, it may be necessary to remove the prefilter rods for cleaning. The need to clean these rods is usually indicated by poor peak shape or loss of resolution, although other more likely causes, such as source contamination, should be eliminated first. It is necessary to remove the inner and outer ion source assembly before the prefilter assembly can be removed.
Detector	The detector consists of a conversion dynode, phosphor plate, and photo-multiplier tube. The detector works by accelerating positive or negative sample ions onto a dynode surface that emits electrons. The electrons are then accelerated to strike a phosphor, which produces photons of light that are amplified by the photomultiplier and collected as the signal.

#### Clarus 600 MS Hardware Guide

Photomultiplier	The photomultiplier consists of a photosensitive surface and electron multiplier sealed in a glass tube. The light strikes the front window, electrons are emitted and accelerated onto the first dynode of the electron multiplier and avalanche down the chain of dynodes. The multiplier is sealed in its own permanent vacuum chamber (glass tube) and cannot be contaminated. However, contamination on the front window will raise the noise level and lower the sensitivity.
Electronics	The Clarus 600/560 D MS electronics consist of a port in the PC, an embedded processor & digital I/O board, analog board (GC/MS), backplane board, PMT electrometer board, and high voltage and low voltage power supply boards. The embedded processor controls all aspects of instrument and data acquisition.
	Ions exiting from the quadrupole are accelerated into a cup- shaped dynode where they strike the inner surface. Electrons are emitted into an electric field, which extracts them from the conversion dynode and passes them onto the phosphor. The phosphor is held at a higher positive potential than the dynode. Light is emitted when the electrons strike the phosphor. The resulting optical signal is detected by the photomultiplier (PMT).

# Vacuum System

The source, ion optics, analyzer, and detector are fitted inside a cast aluminum chamber. Vacuum is applied to the chamber using a rotary pump and a turbomolecular pump. The vacuum is monitored through a wide range gauge. The rotary pump sits on the floor and a turbomolecular high vacuum pump or an aircooled oil diffusion pump (Clarus 600 D) is mounted under the ion optics chamber:

#### **Rotary Pump**

The Clarus 600 MS has a 3  $\text{m}^3$ /hr computer controlled mechanical pump. The turbomolecular or diffusion pump is backed by this direct drive rotary pump. The rotary vane pump rests on the lab floor and may be positioned beneath the instrument. Care should be taken to avoid mechanically coupling vibrations from this pump to the mass spectrometer. Operation and maintenance details about these pumps can be found in the manuals provided with the pump.

The rotary vane pump (also called the forepump) provides the first level of vacuum to approximately  $2 \times 10^{-3}$  Torr. The pump has a switchable dual voltage.



#### Figure 9 The rotary (fore) pump

Connect the rotary pump exhaust to a line vented to the atmosphere outside the laboratory or use an appropriate exhaust line filter.

The AC line cord for the rotary vane pump must be plugged into the<br/>designated receptacle on the back of the Clarus 600 MS. The pump is<br/>controlled by the TurboMass software.

Connecting the vacuum hose to the exhaust connection of the rotary pump will severely contaminate the Clarus 600 MS.

#### Vacuum Pump Options

The Clarus 600 MS offers three different vacuum pump capacities. The tubomolecular and diffusion pump options are designed to fit your applications, performance and budgetary needs. The Clarus 560 D only utilizes a diffusion pump.

#### Turbomolecular Pump

Clarus 600 MS has two turbomolecular pump options in three configurations. Turbomolecular pumps are high-speed turbines which transport the sample and carrier gas molecules away from the mass spectrometer.

Clarus 600S - The 75 L/sec turbomolecular pump supports Electron Ionization operation (EI) and has optional water cooling.

Clarus 600 T – All of the functions and options of the 600 S with a 255 L/sec turbomolecular pump for higher column flow rates, pump-down time under three minutes, and lower detection limits

Clarus 600 C - All of the functions and options of the 600 T with positive and negative Chemical Ionization (CI) operation.

#### **Diffusion Pump**

Clarus 600 D and the 560 D MS has an air-cooled oil diffusion pump. This pump is only available for Electrical Ionization (EI) operation.

Pump fluid is heated in the base of the pump to produce a vapor which passes through the interior of the jet assembly and emerges from the jets as high-velocity vapor streams. These streams entrain eluting compounds and carrier gas, condense on the cooled pump body wall, and drain into the base of the pump for recirculation. The entrained compounds are transferred to the forepump.

The diffusion pump system has a manual vent switch. This manual vacuum venting is controlled by a push button toggle switch and a pump temperature sensing switch. When you push the button in, the vent is opened. When the button is up (not pushed in), the vent is closed. See the following illustration.

The mass spectrometer's vacuum system is controlled from the Tune page. Be sure that this is done in accordance with the information provided in your *Mass Spectrometry Hardware Guide*. The following procedures describe a **Turbomolecular** Pump system and a **Diffusion Pump** system.

System Description	System Diagram
Vacuum System Off.	R
Backing pump turned on.	
High vacuum pump is on.	
Proper operating conditions reached.	R
Fault with high vacuum pump.	
When vacuum system off has been initiated	

# Diffusion Pump Operating States

System Description	System Diagram				
When system has cooled and diffusion pump is turned off.	R				
When backing pump is turned off.					
When vacuum leak is detected.					
When vacuum gauge failure is detected.					
When vacuum gauge failure is detected on start up.					
High vacuum pump is on.	Display count down timer.				
When system has cooled and diffusion pump is turned off.	Display count down timer.				

# Diffusion Pump Operating States Continued

#### Turbomolecular Pump

#### Pumping Down a Turbomolecular Pump Vacuum System

Select Pump/Vacuum System On from the Options menu on the Tune page.

The menu name will change from **Pump/Vacuum System On** to **Vent/Vacuum System Off**, and the system will begin its pump-down sequence. Once **OPERATE** is enabled, it remains enabled unless the **Vent/Vacuum System Off** command is given.

#### Venting the Vacuum System (Turbomolecular Pump)

- 1. Cool the source and inlet to below  $100 \,^{\circ}$ C.
- 2. Select **Vent/Vacuum System Off** from the **Options** menu on the Tune page, and confirm that you want to vent the system.

#### **Diffusion Pump**

#### Pumping Down a Diffusion Pump Vacuum System

Select Pump/Vacuum System On from the Options menu on the Tune page.

The menu name will change from **Pump/Vacuum System On** to **Vent/Vacuum System Off**, the roughing pump turns on and waits for the system to achieve a minimum vacuum level of **3.7**  $\times$  10<sup>-1</sup> Torr. Once that vacuum level has been achieved, a relay turns on the diffusion pump heater and a countdown timer starts. A typical vacuum level will stay constant until the count down timer reaches 10 minutes, the vacuum drops quickly to 1  $\times$  10<sup>-4</sup> Torr, and continue to 4  $\times$  10<sup>-5</sup> Torr before the countdown timer ends.

When the timer reaches 5 minutes, the software will enable **OPERATE**. If you attempt to use the system prior to achieving a safe operating vacuum ( $5 \times 10^{-5}$  Torr), a warning message will appear.

# CAUTION The software will not prohibit the use of the system prior to reaching the desired vacuum. The software will monitor the vacuum gauge pressure to determine when the system has reached the proper operating vacuum ( $5 \times 10^{5}$ Torr).

Once **OPERATE** is enabled, it remains enabled unless the **Vent/Vacuum System Off** command is given.

#### Venting the Diffusion Pump Vacuum System

- 1. Cool the transfer line and source to under  $100 \,^{\circ}$ C.
- 2. Select **Vent/Vacuum System Off** from the **Options** menu on the Tune page, and confirm that you want to vent the system.

The system will start its automatic venting sequence. The software monitors the temperature of the source and the inlet. When the temperatures of both of the source and the inlet are less than 100  $^{\circ}$ C, the software turns off the diffusion pump heater and starts a 20 minute countdown timer.

During the cooling down period, the countdown timer will display the time remaining in minutes and seconds. When the count down timer reaches 0, the backing pump turns off.

Once the backing pump has been turned off, the software will display a message indicating that the system is now cool enough to vent. The message also reminds you to turn off the carrier gas.

3. Vent the mass spectrometer by **pressing the push-button** behind the front door of the mass spectrometer. It will lock in the pressed-in position and turn red indicating the vent valve is open.

**CAUTION** If you try to vent a hot diffusion system, oxidation of the pump oil may occur and cause oil to enter the analyzer tub which will damage the mass spectrometer.

#### Clarus 600 MS Hardware Guide



The depressed push button vent switch lights when venting is allowed. Before pressing **Pump/Vacuum System On**, make sure the vent switch is closed (the button is out and the light is off)

Never vent when:

- The diffusion pump is hot
- During the 20 minute cool-down period

Always check that the front panel vent button is out and the lamp is off when starting to pump the system down.

- **NOTE:** The vent valve will operate if the vent switch is pushed in (on) before the diffusion pump becomes hot. This includes the first few minutes of **Pump/Vacuum System On**. Venting during this period may cause a vacuum fault to occur and risk back streaming the diffusion oil into the analyzer. It is a good reminder to leave the instrument front door open whenever the push button switch is pushed in (on).
- **NOTE:** When the diffusion pump is hot, the vent switch is deactivated and will not light when pushed in (on). Since the vent switch may be left in the depressed position at any time, you should be careful to avoid closing the instrument door and forgetting that the push button switch is pushed in. (When the diffusion pump cools, the pushed in push button switch will light and automatically vent the system).

#### Vacuum Gauge

The single wide range vacuum gauge monitors the system pressure from atmosphere down to  $10^{-9}$  Torr using a combined Pirani/Inverted Magnetron ionization sensor.

Normal operating pressure with 1 mL/min helium for the 255 L/sec turbomolecular pump is between  $9x10^{-6}$  Torr and  $2x10^{-5}$  Torr after pump-down and ion source bakeout. The 75 L/sec turbomolecular and the diffusion pumps will operate at somewhat higher pressures, typically below  $4x10^{-5}$  Torr.

# TurboMass Software

TurboMass software is the user interface of the Clarus system. The following screens show some examples of how you can control Clarus. Interaction is via the mouse and keyboard using menu-driven commands. Printing, file management and other routine procedures are performed using the appropriate Microsoft Windows modules.

# **Top Level Screen**

This screen contains the GC/MS status, sample list, sequence queue, and provides you with access to all other functions.

TurboMass - DEFAU	JLT - De	fault.SPL												
<u>File E</u> dit <u>S</u> amples <u>R</u> un	<u>V</u> iew (	Quantify ⊆onfigu	ure <u>G</u> C <u>T</u> ools	Help										
1000		<b>-</b>	· 🔁 🔁	51 🔤 🔟 🧉	<u>?</u>		3 🖻 🛍	3 <u>3<sub>+∞</sub> 3</u> + <del>q</del> }=		¥ 👗		<u>=</u> =1	8	
GC		File Name	MS Method	GC Method	Vial #	Injector	Sample ID	File	Text		Condition	IS	Quantify Method	Calib
General Status General Status General Status GC Status MS Operate Pressures Flament		Piel vanne Piel vanne Default01	MS Method	CE Method DEFAULT	*   Vial #   1	Injector A	Sample ID	9 <u>3</u>	Text		International In		≦∆ [ Quantify Method	Calt
							_							>
	Index	Acqui Des	cription	Status					Index	Proc	Description		Status	
Ready							Inst	rument Not Pre	sent		0:0	Shutdown	Enabled	

### Tune Page

The Tune Page allows you to tune the mass spectrometer, control the gases, set the GC interface temperature, and monitor the instrument vacuum pressure.



# Analytical Column

The analytical column inside the Clarus GC oven provides the sample separation. Make sure you select the proper column for your analysis. PerkinElmer offers a wide range of columns in the *Gas Chromatography Column Catalog*. The TurboMass Tutorial provides additional column selection tips.

There are several things to consider when choosing an analytical capillary column:

- 1. Know the *types of samples* you will be analyzing. Are they volatile, semi-volatile, pesticides, solvents, etc?
- 2. Select a *stationary phase* based on polarity of the sample. A very general rule in column selection is that like dissolves like. Column polarity has the greatest effect on how the column separates the compounds of interest as the sample interacts with the stationary phase. There are different degrees of polarity from non-polar to very polar. When compounds are separated primarily on their boiling points the phase is considered to be non-polar. Polar phases typically separate compounds based on the chemical interactions between the sample components and the stationary phase.
- 3. The *inside diameter* of the capillary column has an effect on the column's resolving power and its capacity or concentration range. In general, the larger the inside diameter of the column, the larger the sample capacity. However, the larger the inside diameter, the higher the flow necessary to achieve good performance.
- 4. The next parameter is the *phase or film thickness*. Film thickness will primarily affect the retentive character and the capacity of the column. Increasing the film thickness will cause an increase in the retention of the compounds being analyzed. Thick film columns are primarily used for extremely volatile compounds. The thicker phases will retain components longer, allowing them to interact longer with the stationary phase, thereby increasing the separation of closely eluting compounds.
- 5. The last variable to consider is *column length*. The effect of column length on a separation becomes less important as column length increases. Resolution is a function of the square root of the column length. An example of this relationship is that, if you want to double the separation between two peaks without changing the stationary phase, inside diameter, film thickness, or GC conditions, it would take a four-fold increase in the column length. A 30 meter column is the most common length and is usually sufficient for analyzing most samples. Typically, users doing environmental EPA type analysis will use a 30 meter column for semi-volatile compounds and 60 to 105 meter columns for volatile compounds.

# **Pre-Operational Checklist**

This checklist provides you with a list of items to check to make sure everything are in working order before you begin to use Clarus.

Item					
Are the gases connected to the GC?					
Is the proper column connected?					
Is the proper liner installed in the injector?					
Are your samples prepared?					
Is there a GC method?					
Is the GC split vent open?					
Is the proper mass spectrometer vacuum achieved?					
Is the system leak-free?					
Is there a mass spectrometer method?					
Are the autosampler wash vials filled with solvent?					
Are you using the proper column flow or pressure?					
Did you check the air/water spectrum on the TurboMass Tune screen?					
Is the injector hot and set to the proper temperature?					
Is the transfer line hot and set to the proper temperature?					
Is the source hot and set to the proper temperature?					

Clarus 600 MS Hardware Guide

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# Maintenance **4**



## Overview



High electrical voltage is present inside the mass spectrometer. To prevent the risk of electrical shock or injury from high voltage, unplug the AC line cord from the AC outlet and wait at least one minute before opening or removing an instrument panel.



Disconnect AC power cord from outlet before removing any cover or parts. Do **not** operate the instrument with any covers or parts removed.



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.

Cleanliness and care are of critical importance whenever internal assemblies are removed from the instrument.

- Always prepare a clear, clean work area.
- Make sure that any required tools or spare parts are close at hand.
- Obtain small containers to store screws, washers, spacers etc.
- Never touch any internal source parts with your bare fingers.

- Use tweezers and pliers whenever possible.
- If nylon or cotton gloves are used, prevent leaving fibers in sensitive areas. **NEVER** use rubber gloves.
- Before reassembling and replacing dismantled components, inspect O-rings and other vacuum seals for damage. If you in doubt, replace the O-rings and vacuum seals with new ones.

If a fault occurs soon after repairing or disturbing a particular part of the system, ensure that this part has been correctly refitted and/or adjusted and that any adjacent components have not been inadvertently disturbed.



Many of the procedures described in this chapter involve removing potentially toxic contamination deposits using flammable or caustic agents. Anyone performing these operations should be aware of the inherent risks and should take the necessary precautions.

## Typical Overall Maintenance Schedule

Performing maintenance tasks on a routine basis can reduce the overall costs of operation. If a fault occurs, you can correct it with minimum difficulty.

Advanced maintenance should be performed by a skilled person capable of removing complicated mechanical assemblies. For example, an untrained individual should not attempt to remove the manifold but may be able to perform basic maintenance such as draining and filling the forepump.

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

Factory trained service personnel can assist in any advanced training needs. All tasks should be logged into a logbook to keep a record of any problems or trends.

## Daily

- Make sure all system components are in working order.
- Check and ensure that there are gas supplies to the GC and to the mass spectrometer.
- Check the air/water spectrum. Perform leak checking if necessary.
- Enter information into a logbook.

### Weekly

- Check the tune and mass calibration. Tune if necessary.
- Check the forepump oil level and color. Replace if necessary.
- If equipped with a water chiller, check the water level and temperature.
- If using CI, at the end of the day gas ballast the forepump lightly for 20 minutes.

## Monthly

- Clean the fan filters on the rear of Clarus MS.
- Check the reference gas vial. Refill if necessary.

## **Every Six Months**

- Replace the forepump oil.
- Check the inner source. Clean if necessary.
- Check the analyzer prequadrupole. Clean if necessary.

## Yearly

- Check lens 1, lens 2, and analyzer prequadrupole. Clean if dirty.
- Check and clean the forepump inlet filter, gas ballast control, and the motor fan cover and enclosure.

## Leak Checking

Checking for leaks is actually checking the integrity of the vacuum system. You observe masses 4 (helium), 18 (water), 28 (nitrogen), and 32 (oxygen).

To leak-check the system, follow this procedure:

- 1. Ensure that all connections are made to the mass spectrometer.
- Set the GC split flow to 50 mL/min by pressing the PSSI injector icon on the Clarus GC touch screen. On the next screen, press the split flow setpoint and use the up and down arrow or keypad 
   Image: Complexity of the capillary injector is in position 1 and you selected split flow in the PPC configuration software, the following screen is displayed.

Method 5		8	ő	м 🖏
A-PSSI	Oven	A-F	ID [	Events
. 🔊			🔽 He	ater Off
	∏	OFF	°C	<u>Lara</u>
25	°C terr	ıp-init.		Program
🐴 🧿 Car	rier Gas —			
0.0	psi	2.0	nsi	Research 1
0.00	cm/sec pre	issinit.		Brogrom
Fr 😐 Spli	t			
total flow	0	50	mL/m	
ratio	0	low		
Not Ready	r			Mar 4
Start	<b>•</b>			Tools 🔻

The total flow (split vent + septum purge + column) is displayed in the lower left of the screen. The split vent flow setpoint is displayed in the setpoint box in the lower right.

3. Start the TurboMass software by clicking on the Windows **Start** button at the bottom left of the screen and selects **TurboMass** under the *Programs/TurboMass/TurboMass* path, or double-click on the TurboMass icon if it is on your Windows desktop.

The initial TurboMass window is displayed.

TurboMass - DEFA	ULT - De	fault.SPL										
Elle Edit Samples Bur	i ⊻iew g	Quantify ⊆c	onfigure <u>G</u> C <u>I</u> ools	Help								
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	< (1)											>
	Index	Acqui	Description	Status				Index	Proc	Description	Ratus	
Ready						Ins	trument Not Pre	sent		0:0	Shutdown Enabled	

4. Display the Tune dialog by clicking 66

The Tune dialog is displayed.

- 5. Make sure the Tune parameters are similar to the values displayed.
- **NOTE:** The inlet and source temperatures should be less than 100°C <u>only</u> when venting the system.

TunePage - c:\turbomass\default.pro\acqudb\default.ipr												
	1											
Ele Source Disponitor   Vocum System	₩ 1 ₩ 2 ₩ 3 ₩ 4	Mass 69 131 219 502	Spar 4 4 4 4		Gain 1 3 3 30		Tor	" ) €-7				
GC Interface	0.0%	000	88 ×1	0.0%	131.0	3	0.0%	219.0	3	0.0%	502.0	30 x20
-Souce Parameter Detector Frage 0 10 10 10 10 10 10 10 10 10 10 10 10 1												
- MS Palandes -												
	7.0	69.0	71	9.0	131.0	13:	7.0	219.0	221	0.0	502.0	50
Acquire										Press fo	( Operate	
Ready					Ver	xed			Stat	ndby		

6. Click **Press for Operate** next to the red indicator box.

The indicator box color changes to green and the name of the button changes to **Press for Standby**.



7. Select **Pump** from the **Options** menu.

This starts the forepump and the turbomolecular pump or diffusion pump depending on which pump you have installed. Watch the vacuum gauge readout and allow time for the gauge to achieve  $4 \times 10^{-5}$  Torr.

8. Observe the displayed masses.

Mass 4 (helium) should be much larger than mass 18 (water), which should be larger than mass 28 (nitrogen), which should be about 4 times larger than mass 32 (oxygen).

- 9. If the nitrogen and oxygen are larger it indicates an air leak, which could damage the filament. Immediately click **Press for Standby** to turn off the filament.
- 10. If a leak exists, locate and fix it.

Typical areas to check for leaks are around fittings and areas under vacuum.

## Tuning Clarus 600/560 D MS

After determining that no leaks exist and before acquiring data, you may need to check the mass spectrometer tuning conditions and, if necessary, modify one or more of the tuning parameters. Clarus MS can be tuned either manually or automatically from the Tune window.

The left side of the page holds the tuning parameters for a selected region of the mass spectrometer. You can change the region by selecting an item from the Window menu, or by pressing one of the buttons on the bottom-left of the Tune page.



The panel in the top right of the Tune page displays the tune peak information and instrument pressure information.

The tune peak display is located on the right side of the screen and allows you to view up to four masses. The corresponding check boxes located above the peaks allow you to control each peak display. Any one of the tune peaks can be zoomed so that it occupies the entire tune peak area. When a tune peak has been zoomed, the controls for the mass and span for that peak are displayed at the top of the display window. This enables you to display the pressure information while having control over the peak display.

To display the Tune page:

1. While displaying TurboMass sample list screen click 60.

The Tune page is displayed.

Vacuum System	Status Vecuum DK		Mass 69 131 219 502	Span 4 4 4 4 4 4	G 3 3 30	ain		To					
		100.016	69.0	8		131.0	20	50.0%	210.0	2	6.0%	602.0	8
GC Interface (nlet Line Temperature	26 25												
Source Parameters	[-70 [70 []												
Isap Emission Bepeller				-		-		-	-			1	
Lens 1	52 30			-									
Source Temp (C)	26 25												
Filament Current	3.67												
Source Current	-256			-				-					
MS Parameters		-1	-			A							
JM Res	10.6												
HM Res	12.4		- 61	-				-		-			
log Energy	0.7												
Ion Energy Rang	25												
Marchine D.O.	200									- N. 1	A CONTRACTOR OF		

2. Turn on the filament and high voltages by clicking **Press for Operate** at the bottom right of the window.

The indicator box turns green to indicate that it is on.

3. Select UltraTune/Custom (AutoTune) from the Options menu, then click Start.

You will hear a click when the reference gas solenoid valve opens and AutoTune begins. Upon completion, the message **AutoTune completed successfully** is displayed.

4. Click **OK**.

#### Clarus 600 MS Hardware Guide

- 5. Select **Reference Gas On** from the **Gas** menu to remove the check mark ( $\checkmark$ ), or click **iff** to set it in the **up** position.
- 6. Save this new Tune of the instrument by selecting Save As from the File menu and entering a name for this tune in the File name field.A way to keep track of the tunes is to use dates for the file names.
- 7. Click Save.

Your mass spectrometer is now Tuned.

To ensure proper operation, check the mass calibration.

## Preparing Clarus 600/560 D MS for Hardware Maintenance

To prepare Clarus MS for hardware maintenance, there are several steps that are common and precursory to all maintenance procedures. They are as follows:

#### Turn off the Operate Mode

- 1. Display the **Tune** window.
- 2. If the **Press for Standby** button is green (indicating an operating instrument), switch it off by clicking on it.



#### Cool the Transfer Line, GC Column Oven, and the Source



The transfer line, GC oven, and source are **HOT**. Touching them can cause serious burns. To prevent personal injury, wait until the oven and transfer line reach the lower setpoint temperature before touching them. Only grab and hold the source by its handle.

1. On the left side of the Tune page enter **20** in the **Inlet Line Temperature** to cool the GC Interface (transfer line).

**CAUTION** If the Clarus GC is off, then the PPC control is off and no gas is flowing through the system.

- 2. Open the GC oven door to cool the column oven.
- 3. On the **Tune** page, set the source temperature by entering **20** in the **Source Temp** field.
- 4. Allow the transfer line, column oven, and source to cool before touching them.



*Remember, the transfer line was heated and it may take at least 10 to 20 minutes to cool.* 



#### Vent the System

1. Once both the Inlet Line and Source temperatures have dropped below 100 °C, select **Vent/Vacuum System Off** from the **Options** menu.

The Vent Pump dialog appears. All pumps are turned off.

- 2. Click **OK**.
- 3. Observe the Vacuum Pressure Gauges status on the Tune window.

The gauge goes to **ZERO** after the turbopump reaches 50 % speed and the vent valve is opened.

- **NOTE:** *Mass Spectrometer venting may take several minutes depending on the helium flow and/or vent gas flow into the manifold.* 
  - 4. The system is now vented to atmosphere (or optional dry gas).

#### Turn off the GC Carrier Gas



Set all temperatures to ambient. Once the GC column oven, source, and transfer line are cool you may then turn off the GC carrier gas.

## Changing a Column

**CAUTION** To ensure that the mass spectrometer remains contamination free, wear powder-free, lint-free gloves while performing this procedure.

## Tools and Items Required

- New column.
- Column wafer scribe (Part No. N930-1376).
- Two high-temperature septa.
- Two <sup>1</sup>/<sub>4</sub>-inch open-end wrenches
- Lint-free, powder-free PVC gloves (Part No. N621-2495).
- 1/16-inch graphite/Vespel ferrules (0.8 mm i.d., Part No. 0992-0107, 0.25 mm i.d., Part No. 0992-0104 or 0.325 mm i.d., Part No. 0992-0105).

## Removing a Column

- 1. Prepare the mass spectrometer for hardware maintenance (see *Preparing Clarus 600/560 D MS for Hardware* Maintenance on page 85).
- 2. Using a <sup>1</sup>/<sub>4</sub>-inch open-end wrench, loosen the 1/16-inch column nut attached to the injector fitting and slide the column tubing completely from the injector and column nut.

3. Using a <sup>1</sup>/<sub>4</sub>-inch open-end wrench, loosen the 1/16-inch column nut on the transfer line. Slide the column tubing completely from the transfer line and column nut. Remove the column from the GC oven.



#### Figure 10 Removing a column

4. Open the Clarus 600 MS access door, loosen and remove the two black thumbscrews, hold the source by the handle, and pull it out.

**CAUTION** To prevent contamination, only hold the source by its handle. Never touch the part of the source that comes in contact with ions with your bare fingers.

#### Clarus 600 MS Hardware Guide

5. Place the source on a clean surface. Preferably place the handle end on a flat surface so that the source stands in an upright position.



**Figure 11 Removing the source** 

## Connecting the New Column to the Split/Splitless Injector

This procedure describes how to connect a column to a capillary injector. For procedures to connect a column to a PSS or POC injector refer to "Installing a Capillary Column" in Chapter 6 of the *Clarus GC User's Manual* (Part Number 0993-6780).

*This injector has a fragile 1/16-inch fitting. To preserve the integrity of the fitting:* 

#### • *Carefully tighten the nut on the fitting.*

- Do not cross-thread or overtighten the nut on the fitting.
- Allow the injector to cool before connecting a nut.
- 1. Unwind 20 cm (8 inches) from one end of the column.

CAUTION



Figure 12 Unwinding 20 cm from the injector end

#### Clarus 600 MS Hardware Guide

2. Insert a septum, 1/16-inch column nut (part number 09903392), and 1/16-inch graphite ferrule (0.8-mm i.d. Part Number 0992-0141, or 0.5-mm i.d. Part Number 0990-3700) over one end of the column as shown in Figure 13.



#### Figure 13 Inserting the nut and ferrule on the column

**NOTE:** Verify that the tapered end of the ferrule is facing towards the nut as shown above.

3. Cut about 1 cm (3/8 inch) from the column end using a wafer scribe (part number N930-1376, pkg. of 10 scribes). Break off the tubing at the score mark so that the break is clean and square. Examine the cut with a magnifying glass and compare it to the examples shown in Figure 14.



Figure 14 Good cut and bad cuts

4. Locate the capillary injector fitting inside the oven.



#### Figure 15 Location of the capillary injector in the oven

5. Position the septum on the column as shown in the following table:

Injector Type	Column Insertion Dept (measured from the back of the nut to the end of the column)
CAP	4.44 to 5.1 cm (1 <sup>3</sup> / <sub>4</sub> to 2 inches)
PSS	3.8 to 4.44 cm (1 <sup>1</sup> / <sub>2</sub> to 1 <sup>3</sup> / <sub>4</sub> inches)
POC	Flush with the septum end of the injector

- 6. Insert the column into the capillary injector fitting.
- 7. Hand-tighten the column nut <sup>1</sup>/<sub>4</sub> turn past fingertight.
- 8. Using two ¼-inch wrenches tighten the column nut only until the column cannot be pulled out of the fitting.

**CAUTION** Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.

## Connecting a New Column to Clarus 600 MS

1. Uncoil 50 cm (20 inches) from the new column. Place the column on the column hanger in the oven.



2. Slide the septum, 1/16-inch column nut and 1/16-inch graphite/Vespel ferrule over the end of the column.



Figure 17 Inserting a septum, column nut and ferrule on a column

3. Slide the septum, column nut, and ferrule along the column to the position shown below. Score and break 1.0 cm off the end of the column. Wipe the column with a methanol dampened lab tissue (for example, a Kimwipe).

#### Maintenance



#### Figure 18 Preparing to install the column in the transfer line

- 4. Carefully insert the end of the column through the transfer line and into the mass spectrometer source until it is positioned midway between the hole and the wall on the right side of the source. See Figure 19.
- 5. Hold the column in this position as you slide the column nut and ferrule to the transfer line and tighten the nut fingertight. Slide the septum until it is flush against the rear of the column nut. See Figure 20.

This marks the position of the column end in the source.

**NOTE:** Finger tight means just tight enough to hold the column in place so that you are still able to move the column slightly if necessary to reposition it.



Figure 19 Positioning the column end in the source assembly



## Figure 20 Marking the column position in the source

6. Pull the column back until the distance between the back of the nut and septum is about 10 cm.

CAUTION

To prevent breaking the end off the column, you need to pull the column back to reinstall the source in the mass spectrometer.

#### Clarus 600 MS Hardware Guide



Figure 21 Pulling the column back to reinstall the source

7. Position the inner source so it aligns with the guide pin, gently insert the source into the mass spectrometer, and secure it in place with the two black thumbscrews. Tighten the black thumbscrews until they are fingertight.



Figure 22 Reinstalling the source

8. Carefully slide the column into the transfer line until the septum is flush to the back of the column nut. To make a leak-free seal, use a <sup>1</sup>/<sub>4</sub>-inch wrench to tighten the column nut <sup>1</sup>/<sub>4</sub> turn past fingertight.



Figure 23 Repositioning the column in the source

#### **Checking for Leaks**

1. Start the carrier gas flow and leak-check the fittings for leaks.

CAUTION

To ensure maximum sensitivity, if this is a new column condition the column to its maximum accepted operating temperature, even if the column manufacturer claims that it is preconditioned.

2. Start the vacuum by selecting **Pump** from the **Options** menu on the Tune window. Monitor the vacuum and search for leaks if necessary.

Refer to the vacuum leak-checking procedure described in *Leak Checking* on page 79.

3. Once you have verified that no leaks exist, set the transfer line temperature to its original value and close the GC oven door to heat the column.

## Refilling the Reference Gas Vial

It is time to refill the reference gas vial when you lose reference gas peaks intensity and you do not see a liquid in the reference gas vial, or any time the mass spectrometer is vented, and the liquid in the vial level appears low.

**NOTE:** You should check liquid level in the reference gas vial any time you need to remove the mass spectrometer cover and vent the instrument. Using a tool such as a dental mirror will help you observe the liquid level in the Reference Gas Vial.

## Items Required

- Lint-free, powder-free PVC or polypropylene gloves (Part No. N621-2495). •
- Pasteur Pipette or 50 µL syringe. •
- Heptacosa (FC43) (Part No. N621-2407). •

To refill the reference gas vial, follow this procedure:



Make sure to vent the instrument and turn the power off.

1. Ensure that the solenoid have been switched off.

2. With the power off, unplug the source connector for more room to work. Use a 5/32 inch Allen wrench to remove the two allen nuts from the handle. See following photo.



3. With the handle off remove the reference gas vial assembly and bracket out. See the following figure.



Figure 24 Removing the reference gas vial from the mass spectrometer



The toxicity of the FC-43 calibrant is uncertain. Take appropriate precautions to avoid getting the calibrant on your skin or in your eyes.

- 4. Loosen the knurled fitting behind the gas vial by ½ turn, and pull out the vial. A black O-ring may remain in the fitting.
- 5. Using a pipette or syringe, add 25 to 50  $\mu$ L but no more than 50  $\mu$ L of Heptacosa (FC43). See the following figure. Fill the bulb.

Never add more than  $50 \,\mu$ L.



#### Figure 25 Filling the reference gas vial

- 6. Re-insert the reference gas vial into the mass spectrometer. Make sure the O-ring is still present and the tapered end of the ferrule faces the mass spectrometer.
- 7. Tighten the knurled nut with your fingers until fingertight.
- 8. Return the bracket to the proper position and use a 5/32 inch Allen wrench to retighten the two Allen nuts to the bracket.
- 9. Replace the top cover and pump the system down to the proper vacuum.
- 10. From the Gas Menu select the **Reference Gas Option** from the drop down menu. Leave the Reference Gas Valve open for 60 minutes with **Operate off** to pump to pump out the gas from the bulb before tuning the mass spectrometer.
- **NOTE:** On the Tune Page the Gas drop down the **Pump Out Reference Gas** choice no longer works. If you try to have both the **Reference Gas On** and the **Pump Out Reference Gas** options selected you will get an error message. To avoid an error message make sure to only check **Reference Gas On**.

## Inner Source Maintenance

Inner source maintenance consists of cleaning those components that contact ions. For example,

- EI source: the ionization chamber, repeller, trap, ion exit plate, and ion entry area.
- CI source: the ionization chamber, ion entry area, and ion exit plate.

**CAUTION** *Never clean the filament. If the filament is open, replace it by following the procedure in this chapter.* 

#### Items and Tools Required

- Ultrasonic bath.
- Aluminum foil or lint-free disposable cloth squares.
- Aluminum oxide powder.
- Wooden stick cotton swabs.
- 2 mm open-end wrench (for EI source).
- Tweezers.
- Powder-free, lint-free gloves (Part No. N621-2495).
- Small flat-blade screwdriver.
- Reagent Grade acetone.
- Reagent Grade methanol.
- De-ionized Water
- Clean 100 mL glass beaker.
- 6000 Grade Micro Mesh (Part No. N930-3420).
- 8000 Grade Micro Mesh (Part No. N930-3421).
- 600 grit aluminum oxide in DI Water with a few drops of methanol to make a paste

## Removing the Inner Source

To remove the inner source, follow this procedure:

**CAUTION** Before performing source maintenance, always prepare the mass spectrometer by following the instructions in Preparing Clarus 600/560 D MS for Hardware Maintenance on page 85.

- 1. Prepare the mass spectrometer for maintenance as described in *Preparing Clarus 600/560 D MS for Hardware Maintenance* on page 85.
- 2. Open the GC oven door and locate the Clarus MS transfer line.
- 3. Using a 9/16-inch wrench, loosen the <sup>1</sup>/<sub>4</sub>-inch nut on the transfer line.
- 4. Pull the inner transfer line tube back 25 mm (1 inch).



Figure 26 Pulling back the transfer line

- 5. Loosen the two thumbscrews on the inner source, grab it by its handle, pull it out of the mass spectrometer and set it on a clean surface.
- 6. Close the mass spectrometer access door.
- 7. Put on a pair of powder-free, lint-free gloves (Part No. N621-2495).



Figure 27 Removing the inner source

## El Inner Source Maintenance

To gain access to the parts on the EI inner source that need cleaning, follow this procedure.

NOTE: For this procedure use the Inner Source Rebuild Kit Part No. E640-0043.

**CAUTION** To ensure that the mass spectrometer remains contamination free, wear lint-free, powder-free gloves (Part No. N621-2495) while performing this procedure.

#### Disassembling

- 1. Prepare a clean, uncluttered work area and place a square of aluminum foil with the shiny side up. Obtain some clean, small containers (for example, small beakers) to store the screws and small parts as you remove them.
- 2. Using a small flat-blade screwdriver, loosen the screw that secures the old filament assembly to the source. Use tweezers to remove the screw and washer. Pull out the filament assembly.



Figure 28 Removing the filament assembly

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- 3. Using a small flat-blade screwdriver, loosen and remove the four screws on the ion chamber cover plate and remove the cover plate.
- 4. Using a 2 mm open-end wrench, loosen and remove the nut and washer that secure the repeller. Then remove the repeller.
- 5. Using a 2 mm open-end wrench, loosen and remove the nut and washer that secure the ion trap. Then remove the ion trap.



Figure 29 Removing the ion chamber cover plate and repeller
#### Cleaning

- **NOTE:** You can do the following cleaning method of aluminum oxide paste or use the 6000 or 8000 grade micro mesh to polish the flat surfaces of the parts. For either cleaning method, the final step you **must** sonicate the parts in an ultrasonic bath of methanol for at least five minutes. Dry off the parts using lint-free tissue and/or clean compressed Nitrogen gas to prevent solvents from drying on these parts and leaving a residue
  - 1. Mix together aluminum oxide and de-ionized water and a few drops of methanol to make a watery paste.
  - 2. Dip a wooden-stick cotton swab in the solution and clean the darkened areas on the source. Work quickly to prevent the mixture from drying on the surface. Place the cleaned components in de-ionized water prior to rinsing to prevent drying.

### Rinsing

1. Add 50 mL of acetone to a 100 mL beaker, insert the source assembly, repeller, ion trap, ion chamber plate, and sonicate in an ultrasonic bath for ten minutes.

**CAUTION** Do not allow the acetone and methanol to touch the O-ring on the source.

- 2. Carefully drain the acetone.
- 3. Add 50 mL of methanol to another 100 mL beaker, insert the source assembly, repeller, ion trap, ion chamber plate, and sonicate in an ultrasonic bath for at least ten minutes.
- 4. Carefully drain the methanol.



### Figure 30 Rinsing the aluminum oxide from the source

- 5. Dry off the repeller, ion trap, source assembly and ion chamber plate using lintfree tissue and/or clean compressed Nitrogen gas to prevent solvents from drying on these parts and leaving a residue.
- 6. Take the parts amd wrap them in a clean, lint-free cloth and bake them in the GC oven at about 80 °C for about fifteen minutes.

### Reassembling

1. Insert the ion trap and repeller through the spacer and insulator. Then insert it into the source.

#### Maintenance



### Figure 31 Inserting the ion trap and repeller into the inner source

- 2. Invert the source and insert a crumpled laboratory wipe into the repeller and ion trap.
- 3. Using tweezers, install the remaining ceramic pieces, spacers and nut first on the ion trap and then on the repeller.
- 4. Tighten each nut with a 2 mm open-end wrench.

5. Replace the filament assembly.



Figure 32 Installing the ceramics, contacts, washers and nuts on the repeller and ion trap



Figure 33 Replacing the filament assembly

### **CI Inner Source Maintenance**

The supplied CI inner source assembly can be used for both negative CI and positive CI. The maintenance procedures for the CI source are very similar to those of the EI; however, the CI does not have a repeller or ion trap. It does have a smaller exit aperture to ensure that the sample ions properly react with the CI gas.

**CAUTION** To ensure that the mass spectrometer remains contamination free, wear lint-free, powder-free gloves (Part No. N621-2495) while performing this procedure.

To gain access to the parts on the CI inner source that need cleaning, follow this procedure.

### Disassembling

- 1. Prepare a clean, uncluttered work area and place a square of aluminum foil with the shiny side up. Obtain some small containers (for example, small beakers) to store the screws and small parts as you remove them.
- 2. Using a small flat-blade screwdriver, loosen the screw that secures the old filament assembly to the source. Use tweezers to remove the screw and washer. Pull out the filament assembly.



Figure 34 Removing the filament assembly

3. Using a small flat-blade screwdriver, loosen and remove the four screws on the ion chamber cover plate and ion aperture plate, then remove the plates.

Maintenance



# Figure 35 Removing the ion chamber and aperture plates and cleaning the areas shown

#### Cleaning

**NOTE:** You can do the following cleaning method of aluminum oxide paste or use the 6000 or 8000 grade micro mesh to polish the flat surfaces of the parts. For either cleaning method, the final step you **must** sonicate the parts in an ultrasonic bath of methanol for at least five minutes. Dry off the parts using lint-free tissue and/or clean compressed Nitrogen gas to prevent solvents from drying on these parts and leaving a residue

- 1. Mix together aluminum oxide and de-ionized water and a few drops of methanol to make a watery paste.
- 2. Dip a wooden-stick cotton swab in the solution and scrub the darkened areas on the source. Work quickly to prevent the mixture from drying on the surface.

### **Rinsing and Reassembling**

1. Add 50 mL of acetone to a 100 mL beaker, insert the source assembly, ion aperture plate, ion chamber plate, and sonicate in an ultrasonic bath for ten minutes.

```
CAUTION Do not allow the acetone and methanol to touch the O-ring on the source.
```

- 2. Carefully drain the acetone.
- 3. Add 50 mL of methanol to another 100 mL beaker, insert the source assembly, ion aperture plate, ion chamber plate, and sonicate in an ultrasonic bath for at least ten minutes.
- 4. Carefully drain the methanol.



50 mL sonocation solution in 100 mL Beaker

Figure 36 Rinsing the aluminum oxide from the source

- 5. Dry off source assembly and plates using lint-free tissue and/or clean compressed Nitrogen gas to prevent solvents from drying on these parts and leaving a residue.
- 6. Take the parts amd wrap them in a clean, lint-free cloth and bake them in the GC oven at about 80 °C for about fifteen minutes.
- 7. Reassemble the ion aperture plate and ion chamber plate on the source.
- 8. Replace the filament assembly.



Figure 37 Reassembling the CI inner source



Figure 38 Replacing the filament assembly

### **Reinstalling the Source**

1. Position the source so it aligns with the guide pin, gently insert the source into the mass spectrometer, and secure it in place with the two black thumbscrews. Tighten the black thumbscrews until they are fingertight. Do not overtighten the thumbscrews.



Figure 39 Reinstalling the source in the mass spectrometer

- 2. Insert the inner transfer line tube back in the outer transfer line.
- 3. Tighten the ¼ inch transfer line nut fingertight.

To make a leak-free seal use a 9/16 inch wrench to tighten the  $\frac{1}{4}$  inch nut an additional 1/8 turn. Tighten it enough to make a leak-free seal but do not overtighten the nut.



### **Figure 40 Reconnecting the transfer line**

### **Checking for Leaks**

- 1. Start the carrier gas flowing and leak-check the fittings for leaks.
- 2. Start the vacuum by selecting **Pump/Vacuum System On** from the **Options** menu on the Tune window. Monitor the vacuum and search for leaks if necessary. Refer to the leak checking procedure described in *Leak Checking* on page 79.
- 3. Once you have verified that no leaks exist, set the transfer line temperature to its original value, and close the GC oven door to heat the column.

## Replacing a Filament

**CAUTION** Make sure you are wearing powder-free PVC gloves (Part No. N621-2495), and that you wipe each part with a methanol dampened Kimwipe.

### Items and Tools Required

- Filament assembly (Part No. E6400209).
- Tweezers.
- 0.8 mm hex wrench.
- Methanol.
- Powder-free, lint-free gloves (Part No. N621-2495).
- Small flat-blade screwdriver.
- Aluminum foil or lint-free disposable cloth squares.

To replace a filament, follow this procedure.

- 1. Prepare a clean, uncluttered work area and place a square of aluminum foil with the shiny side up.
- 2. Remove the EI source by following the procedure *Removing the Inner Source* as described on page 105.

**CAUTION** To ensure that the mass spectrometer remains contamination free, make sure you are wearing powder-free, lint-free gloves (Part No. N621-2495), and that all tools have been cleaned with a methanoldampened laboratory wipe.

- 3. Using a small flat-blade screwdriver, loosen the screw that secures the defective filament assembly to the source. Use tweezers to remove the screw and washer.
- 4. Pull out the defective filament assembly.



Figure 41 Removing the defective filament assembly

5. Using a 0.8 mm hex wrench, loosen the two screws that secure the contacts to the filament assembly leads. Remove the two contacts.



### Figure 42 Removing contacts from the defective filament assembly

- 6. Position the new filament assembly with the filament side up.
- 7. Insert the contacts on the new filament assembly leads so that they are flush with the white ceramic and positioned with the hex screw side facing up.
- 8. Secure the contacts to the filament assembly by tightening the hex screws with a 0.8 mm hex wrench. Tighten firmly but do not overtighten.



Figure 43 Installing contacts on the new filament assembly

- 9. Position the new filament assembly so that it faces the source and the white ceramic rests on the tab.
- 10. Ensure that the filament coil is aligned with the entrance hole on the inner source assembly.

11. Using a small flat-blade screwdriver, secure the filament assembly in place with the screw and washer that you previously removed.



#### Figure 44 Installing a new filament assembly on the source

12. Install the source assembly back into the mass spectrometer by following the procedure described in *Reinstalling the Source* on page 119.

# Replacing the Head Amplifier

To replace the Photomultiplier head amplifier (Part Number E649-9032), follow this procedure and refer to Figure 45:



The mass spectrometer contains high voltage. To prevent the risk of shock, unplug the line cord from the AC outlet and wait at least one minute before opening or removing any instrument cover or panel.

- 1. Vent the vacuum from the mass spectrometer.
- 2. Turn off the mass spectrometer.
- 3. Unplug the AC line cords from the AC outlets.
- 4. Remove the four screws securing the rear panel and remove the rear panel.
- 5. Remove the two screws on the left side of the top panel and remove the top panel.
- 6. Remove the head amplifier board cover.
- 7. Remove the cable connector to the board.
- 8. Pull off head amplifier board, removing the screw with the ground wire on it.
- 9. Install a new board, making sure that you do not over-tighten the screw with the ground wire.

### **NOTE:** *Pins on the photomultiplier are key for proper alignment.*

10. Replace the photomultiplier head amplifier board cover, cable connector, replace the panels, turn on the mass spectrometer, and pump-down the system to the proper vacuum.



11. Go to the Head Amplifier electronic adjustment procedure.

Figure 45 Replacing the head amplifier

# Mass Analyzer Maintenance

The analyzer element of any high performance quadrupole mass spectrometer is, of necessity, a finely machined assembly that has been precisely aligned using specialized equipment. **Under no circumstances** should you ever disassemble the main mass analyzer assembly.

The mass spectrometer is fitted with prequads that act as a prefilter assembly designed to protect the analytical quads by intercepting the majority of any contamination. As a consequence, the analytical quads should never, under normal working conditions, require cleaning. Occasionally, it may be necessary to remove the prefilter rods for cleaning. The need to clean these rods is usually indicated by poor peak shape or loss of resolution, although other more likely causes, such as source contamination, should be eliminated first.

### Items and Tools Required

- 4 mm hex wrench.
- 5 mm hex wrench.
- Lint-free, powder-free PVC gloves (Part No. N621-2495).
- Wooden stick cotton swabs.
- Deionized Water
- 6000 Grade Micro Mesh (Part No. N930-3420).
- 8000 Grade Micro Mesh (Part No. N930-3421).
- 600 grit aluminum oxide in DI Water with a few drops of methanol to make a paste
- Small flat-blade screwdriver.
- Long flat-blade screwdriver.
- Aluminum foil.
- Acetone.
- Methanol.
- Tweezers.

### **Cleaning Materials**

When cleaning internal components it is important to maintain the quality of the surface finish. Deep scratches or pits can cause loss of performance. Where no specific cleaning procedure is provided, you should use fine abrasives to remove dirt from metal components. Recommended abrasives are:

- 6000 Grade Micro Mesh (Part No. N930-3420).
- 8000 Grade Micro Mesh (Part No. N930-3421).
- 600 grit aluminum oxide in DI Water with a few drops of methanol to make a paste

After cleaning with abrasives, it is necessary to wash all metal components in suitable solvents to remove all traces of grease, oil and, if micro-mesh is used, rubber. The recommended procedure is to swill or sonicate the components in a clean beaker of methanol for at least ten minutes and subsequently to blot them dry with lint-free tissue. Recommended solvents are:

After the components are reassembled, they should be blown with oil-free nitrogen to remove dust particles.

### Removing the Ion Optics Assembly

To remove the EI inner source, follow this procedure:

B	efore performing source maintenance, always prepare the mass
CAUTION sp	ectrometer by following the instructions in Preparing Clarus 600/560
D	MS for
H	ardware Maintenance on page 85.
	I O

- 1. Prepare the mass spectrometer for maintenance as described in *Preparing Clarus 600/560 D MS for Hardware Maintenance* on page 85.
- 2. Open the GC oven door and locate the mass spectrometer transfer line.
- 3. Using a 9/16-inch wrench, loosen the <sup>1</sup>/<sub>4</sub>-inch nut on the transfer line.



*Risk of burns. Never touch a heated mass spectrometer transfer line or a GC injector cap with unprotected (bare) fingers.* 

4. Pull the inner transfer line tube back 25 mm (1 inch).



### Figure 46 Pulling back the transfer line

5. Loosen the two thumbscrews on the inner source, grab it by its handle, gently pull it out of the mass spectrometer, and set it on a clean surface.

6. Close the mass spectrometer access door.



Figure 47 Removing the inner source

### **Removing the Mass Spectrometer Cover Panels**



The mass spectrometer contains high voltage. To prevent the risk of shock, unplug the line cord from the AC outlet and wait at least one minute before opening or removing any instrument cover or panel.

- 1. Turn off the Clarus GC and Clarus MS.
- 2. Unplug the AC line cords from the AC outlets.
- 3. Remove the four screws securing the rear panel, remove the rear panel, and lay it on the bench. If necessary, disconnect the fan wires.
- 4. When reconnecting the fan wires, make sure the fan blows the air into the mass spectrometer.

### Clarus 600 MS Hardware Guide

5. Remove the two screws on the left side of the top panel and remove the top panel.



Figure 48 Removing the mass spectrometer panels

### Removing the Photomultiplier Tube

- 1. Unplug the cable from the head amplifier board.
- 2. Using a long flat-blade screwdriver, loosen the two captured screws securing the photomultiplier tube (PMT) amplifier cover, and remove the cover.



### Figure 49 Removing the PMT amplifier cover

- 3. Using a 4 mm hex wrench, remove the screw securing the PMT amplifier board-grounding strap.
- 4. Carefully remove the amplifier board from the PMT by pulling it straight back.

- 5. Using a 4 mm hex wrench, remove the screws securing the PMT flange to the vacuum chamber.
- 6. Put on a pair of powder-free, lint-free gloves.
- 7. Carefully remove the PMT. Cover it with a laboratory wipe and place it in a safe, dark place.
- 8. Insert a crumpled laboratory wipe into the PMT hole in the vacuum chamber to prevent particulates from entering the vacuum chamber.



Figure 50 Removing the head amplifier board and the PMT

### Removing the Ion Optics Assembly



Make sure to vent the instrument and turn the power off and unplug the mass spectrometer from the AC power source.

- 1. Disconnect the cables connected to the ion optics assembly and place them to the side so they will not interfere with the removal of the ion optics assembly. See the following figure.
- 2. Disconnect the Reference and CI gas lines from the top of the ion optics assembly. See the following figure.



Figure 51 Disconnecting cables and tubing from the ion optics assembly

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3. With the power off , unplug the source connector for more room to work. Use a 5/32 inch Allen wrench to remove the two allen nuts from the handle. See following photo.



4. With the handle off remove the reference gas vial assembly and bracket out and move it out to the side of the instrument. See the following photo.



- 5. Remove the four hex head bolts loosely holding the ion optics assembly to the vacuum chamber. See the following figure.
- 6. Grabbing the ion optics assembly by the two handles, carefully lift the ion optics assembly straight up and away. See the following figure.



Figure 52 Lifting the ion optics assembly from the vacuum manifold

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- 7. Place the ion optics assembly on a clean work surface, with the RF box down and the mass analyzer facing up (see Figure 3).
- 8. Cover the open vacuum chamber with aluminum foil to prevent particulates from contaminating the chamber.



### Figure 53 Positioning the ion optics for maintenance

### **Cleaning the Prequads**

When operating under normal circumstances, you may not have to remove the prequads from the ion optics assembly. To clean the prequads, follow this procedure.

1. Using a very fine abrasive paper (8000 grade) gently clean the ion burns off of the prefilters.

- 2. Wipe the prequads with a methanol dampened laboratory wipe.
- 3. Blow dry with helium or dry nitrogen.

### Replacing an Outer Source Thermocouple

#### Items and Tools Required

- Thermocouple (Part No. E640-0213).
- Lint-free, powder-free PVC gloves (Part No. N621-2495).
- Small flat-blade screwdriver.
- Small adjustable wrench.

To replace the thermocouple, follow this procedure and refer to the following figure:

**CAUTION** To ensure that the mass spectrometer remains contamination free, wear lint-free, powder-free gloves while performing this procedure.

- 1. Using a small flat-blade screwdriver, remove the screw and the thermocouple.
- 2. Loosen and remove the nut.
- 3. Remove all wires from the contact pins.
- 4. Pull all the wires down through the feed connector
- 5. Thread the new wires up through the feed connector and connect all the wires on the contact pins. Tighten the nuts.
- 6. Secure the new thermocouple with the screw.



Figure 54 Replacing the thermocouple

### Removing the Outer Source from the Ion Optics

To remove the outer source from the ion optics, follow this procedure (use Outer Source Rebuild Kit, Part No.E640-0042):



Figure 55 Removing the outer source from the ion optics assembly

### **Replacing Outer Source Heaters**

### Items and Tools Required

- Two Cartridge heaters (Part No. E640-0202).
- Tweezers.
- Lint-free, powder-free PVC gloves (Part No. N621-2495).
- Small flat-blade screwdriver.

To replace the outer source heaters, follow this procedure:

- 1. Position the outer source base plate on a clean work area so that the clamp plate faces up.
- 2. Using a flat blade screwdriver, loosen the three screws that connect to the three pillars.

**CAUTION** The four ceramic rods (Part Number E640-1319) are very fragile. Use extreme care when removing the support plate and lens.

- 3. Carefully lift the clamp plate (Part Number E640-1322) straight up and put it aside.
- 4. Carefully lift and remove the contact support plate from the four ceramic rods.
- 5. Remove the pillar.
- 6. Unplug the outer source heaters from the quadrupole heater.
- 7. Using a flat-blade screwdriver, loosen the outer source heater setscrews.
- 8. Slide the old heaters out of the outer source block and insert the new heaters (Part Number E640-0202).
- 9. Tighten the setscrews 1/8 turn past fingertight.

Do not overtighten the setscrews.

- 10. Connect the heater wires together and plug the quadrupole heater wire into the remaining connector.
- 11. Reassemble the outer source.

Maintenance



Figure 56 Replacing heaters in the outer source

### Replacing the Quadrupole Heater

The quadrupole heater is connected in series with the two outer source heaters.

### Items and Tools Required

- Cartridge heater (Part No. E640-0202).
- Small flat-blade screwdriver.
- Lint-free, powder-free PVC gloves (Part No. N621-2495).

To replace the quadrupole heater, follow this procedure:

- 1. Unplug the quadrupole heater wire from the outer source heater wires.
- 2. Loosen the setscrew that secures the quadrupole heater to the standoff.
- 3. Slide the heater out, discard it, and slide the new heater into the standoff.
- 4. Secure the heater in place by tightening the setscrew 1/8 turn past fingertight. Do not overtighten the setscrew.
- 5. Plug the quadrupole heater wire into the outer source heater wire connector.


Figure 57 Removing the quadrupole heater

## **Cleaning the Outer Source Lens**

#### Items and Tools Required

- 600 grit aluminum oxide.
- Tweezers.
- Methanol.
- Laboratory wipes.
- Acetone.
- Lint-free, powder-free PVC gloves (Part No. N621-2495).
- Small flat-blade screwdriver.
- Wooden stick cotton swabs.

To clean the outer source, follow this procedure:

- 1. Position the outer source on a clean work area so that the side with the support plate is facing up.
- 2. Using a flat-blade screwdriver, loosen the three screws that connect to the three pillars.
- 3. Carefully lift the support plate straight up and put it aside.
- 4. Carefully remove the Lens 2, then Lens 1 focus plates.

#### Cleaning

- 1. Mix together aluminum oxide and methanol to make a watery paste.
- 2. Dip a wooden-stick cotton swab in the solution and clean the darkened areas on the source. Work quickly to prevent the mixture from drying on the surface.
- 3. Remove the residual aluminum oxide by sonication in a beaker of methanol for ten minutes.
- 4. Blow dry with helium or dry nitrogen.
- 5. Reassemble the outer source.



Figure 58 Removing the lenses from the outer source for cleaning

#### Reassembling the Ion Optics Assembly

The following drawing is an exploded view of the outer source showing all parts with their part numbers.



Figure 59 Exploded view of the outer source

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- 1. Place the assembled outer source on the ion optics assembly and secure it in place with the two screws.
- 2. Reattach the tubing to the outer source.



2. Reattach the tubes to the outer source assembly.



#### Figure 60 Reinstalling the outer source

3. Using a flat-blade screwdriver, reconnect the thermocouple to the outer source.

4. Reconnect the wires to the outer source as shown in the following illustration.



Figure 61 Reconnecting the wires to the outer source

### Reassembling the Clarus 600/560 D MS

To reassemble the mass spectrometer, follow this procedure:

- 1. Remove the aluminum foil covering the vacuum manifold.
- 2. Hold the ion optics assembly by its handles and align the guide pins with the holes in the vacuum manifold.
- 3. Gently lower the ion optics assembly until it is seated on the vacuum manifold.
- 4. Replace the four screws and tighten fingertight.
- 5. Remove the laboratory wipe from the PMT hole and reinstall the PMT assembly. Observe the orientation of the PMT tube as shown in the detail.
- 6. Insert the end of the head amplifier ground wire through one of the screws for the PMT assembly. Insert this screw into the hole in the 2 o'clock position and insert the other screw in the other hole. Tighten both screws fingertight. Using a 4 mm hex wrench, tighten both screws <sup>1</sup>/<sub>4</sub> turn past fingertight.
- 7. Plug the head amplifier board onto the pins on the PMT.

#### Maintenance



#### Figure 62 Reinstalling the PMT

- 8. Reinstall the PMT head amplifier cover and secure it in place with the two captured screws.
- 9. Replug all the cables and reconnect the tubing.
- 10. Reinstall the top and rear panels.
- 11. Reinstall the source.

## Vacuum System Maintenance

Vacuum system maintenance consists of the following:

- Checking the forepump to ensure the oil is at the proper level.
- Adding oil to the forepump reservoir.
- Replacing forepump oil.
- Replacing foreline trap pellets.

Complete pump instructions are in the instruction manual supplied with the pump.

## Maintanenace of the Turbomolecular and Diffusion Pump

You should never service the turbomolecular and diffusion pumps. Call your PerkinElmer Service Representative for the maintenance and any problems you may have with these pumps.

## Checking the Forepump Oil Level

1. Locate the oil level indicator window on the forepump.



Figure 63 Location of the forepump oil viewing window

- 2. Determine if the oil level is between the Max Oil Level and Min Oil Level marks next to the window.
- If the oil level is closer to the Min Oil Level mark, add oil. Use Edwards 45 oil (Part No. 0992-3492, 1 liter).
- If it is near the scheduled six-month service, drain and refill the pump with clean oil.
- If the oil is contaminated (indicated by a darkened color), try gas ballasting and if that does not help, drain and refill the pump with clean oil.

## Adding Oil to the Forepump Reservoir

**CAUTION** *Vent the mass spectrometer before opening the plug to add oil.* 

- 1. Unscrew and remove one of the filler plugs on the top of the pump.
- 2. Locate the bottle of pump oil supplied with the pump and add oil until it reaches the MAX mark on the top of the sight glass. Do not overfill.
- 3. Replace the oil filler plug by tightening it until it is fingertight. Do not overtighten the oil filler plug.
- 4. After restarting the pump and allowing it to run for a few minutes, recheck the oil level. If the oil level is below the MAX mark, repeat the above procedure by adding more oil until it reaches the MAX mark.

## Decontaminating the Oil

The pump oil should be clear. If the oil is cloudy or discolored, it is contaminated with residual sample vapors.

- 1. Observe the oil in the oil sight glass.
- 2. Turn the mode selector fully counterclockwise to select the High Throughput mode and set the gas ballast control to the low flow (position I).
- 3. Run the pump until the oil appears clear.

## Replacing the Oil

- 1. Warm the oil by running the pump for at least 10 minutes, and then switch off the vacuum system.
- 2. Unplug the pump from the AC outlet and disconnect it from your vacuum system.
- 3. Remove one of the oil filler plugs.



Figure 64 Draining forepump oil

4. Place the pump on a table. Place a drain container under the drain plug. Raise the end of the pump opposite the drain plug by putting a block under it.



If you were running toxic samples, the oil is contaminated as toxic waste. Handle and dispose of waste oil appropriately.

- Remove the drain plug and allow the oil to drain into the container.
   If the pump oil was contaminated, pour clean oil into the filler hole and allow it to drain until the oil appears clear.
- 6. Replace the drain plug, remove the block and reconnect the vacuum system.
- 7. Add oil until it reaches the MAX mark on the top of the sight glass. Do not overfill.
- 8. Replace the oil filler plug by tightening it until it is fingertight. Do not overtighten the oil filler plug.
- 9. After restarting the pump and allowing it to run for a few minutes, recheck the oil level. If the oil level is below the MAX mark, repeat the above procedure by adding more oil until it reaches the MAX mark.

Part No.

## **Inline Gas Purifiers**

The inline gas purifier lets you change the trap without introducing contaminants into your system. This eliminates the need to flush the system. The trap contains oxygen, moisture and hydrogen adsorbents and is packed and purged under helium.

Color changes in the glass indicating trap will indicated when filter needs to be replaced.

The click on connector fitting has a spring loaded needle valve, which seals when the trap is removed and only opens when the new trap is connected and locked into position. When the click on connectors are installed into the gas line here is no need to loosen or tighten any fittings, the new trap will just click in.

•	
Description	
Indicating Glass Tr	tiple Gas specific (He)

#### **Replacement Traps**

Indicating Glass Triple Gas specific (He) Oxygen/Moisture/Hydrocarbons	N09306107
Indicating Glass Triple Gas specific (He) Oxygen/Moisture/Hydrocarbons with 1/8" Brass Connector (2)	N09306114
Indicating Glass Triple Gas specific (He) Oxygen/Moisture/Hydrocarbons with 1/8" Steel Connector (2)	N09306116

#### **Click On Connectors**

Description	Part No.
1/8" Brass Connector (2)	N09306119
1/8" Steel Connector (2)	N09306120
Stain steel Connector (for connecting two click on traps)	N09306121

Refer to the installation instructions that accompany your new in line gas purifier trap for detailed installation and operating instructions.

## Changing from EI to CI Mode

Changing modes consists of the following:

- Connecting the CI gas.
- Changing the source and instrument control mode.
- Leak-checking.
- Setting-up CI.

## Connecting the CI Gas



*Hazardous gas vapors.* When using ammonia gas when running in the chemical ionization (CI) mode, it is necessary to vent the mass spectrometer effluent from the forepump exhaust into a fume hood or outside the building.



#### **Recommended Gases**

Reagent gases used in chemical ionization (CI) are methane with a minimum purity of 99.999%, isobutene with a minimum purity of 99.98% and ammonia with a minimum purity of 99.998%. Carrier gas tubing should be ultra-clean.

Methane and isobutene require a gas delivery pressure of 15 psi (104 kPa) to the bulkhead fitting on the back of the mass spectrometer. A two-stage stainless steel diaphragm, high purity regulator is. A single-stage stainless steel diaphragm, high purity, rated for corrosive service is required for ammonia. Clean tubing must be used. It must be solvent-washed and nitrogen-dried. The bulkhead connector at the rear of the instrument is a 1/8 inch Swagelok fitting.

To prepare Clarus 600 MS for CI:

- **NOTE:** *Make sure to purge the CI line before you attach it to the rear of the mass spectrometer.* 
  - 1. Obtain the CI gas cylinder for your analysis.
  - 2. Connect the gas line to the CI Gas connector on the rear of the mass spectrometer.
  - 3. Ensure that the mass spectrometer is at the proper vacuum level.
  - 4. Turn on the CI gas and set the delivery pressure to 15 psi (104 kPa).
  - 5. Leak-check all connections.



#### Figure 65 CI Gas connection on the rear panel of the mass spectrometer

## Changing to Cl

To change from the EI to the CI mode:

- 1. Remove the EI inner source by following the procedure described in earlier in this chapter, *Removing the Inner Source*.
- 2. Install the CI source by following the procedure described in on page 105. Properly cover and protect the EI source and put it in a safe place.
- 3. Select **Cl+** from the **lon Mode** menu. The CI+ window appears.

PD	Status Vacuum OK			4 18 28 32	4		16 256 256 256		6-	2		
GC Interface Inter Temperatu	** ( <del>91</del>  200	0.0	•	40		25 -10 -	10.0	8	0.0%	29.0	92.0	
Source Parameters Electron Energy Source Emission Lens 1 Lens 2 Source Temp (C)	1         30           1-1395         200           1-11.0         5.0           4.8         50.0           140         150											
Filament Current MS Parameters LM Res												
Ion Energy Ion Energy Ramp Multiplier (V)								_	_			

- 4. Select **Pump** from the **Options** menu. This starts the forepump and the turbomolecular pump.
- 5. In the Vacuum Pressure Gauges area of the window, observe the Pirani gauge time line and the Penning gauge time line. Wait about 5 minutes until the vacuum gauge achieves about 2.5 x 10<sup>-5</sup>.

## Leak Checking

Before running in the CI mode, confirm that the column is properly installed and the system is leak-free. The best way to check this is by running CI without the reagent gas.

To leak-check the system:

1. Display the Tune page.



2. Select **CI+** from the **Ion Mode** menu.

3. Click **Press for Operate** and observe the air/water masses.



The CI source running in the CI mode without reagent gas to produce an EI emission similar to the EI mode but with reduced sensitivity. You will leak-check your system this way.

If mass 28 is larger than mass 18, you have a leak. Determine the source of the leak and correct it. For example, leak-check all fittings and connections.

## Setting-Up CI

After verifying that no leaks exist, you can proceed to set up the CI mode for an analysis.

#### Setting the Parameter Values

1. Display the following CI window:

💆 TunePage - c:\ci.pro\acque	lb\ci+.ipr														<b>a</b> 🔀
File Ion Mode Calibration Gas C	ptores Help  P3  =  ≡  Max Max  1	2													
CI+ Source Diagnostics	Status Vocuum OK	2 2 2	1 2 3	Mass 4 18 28		Span 4 4		Gain 16 256 256		F					
	1		4	4.0		4		10.0	×	9.2	20.0	×	-	72.0	8
GC Interface         [piet Line Temperature [3]]           Source Parameters         []           Bichton Energy         []           Gource Emission         [-1255]           Lener 1         [-110]           Lener 2         [-4.0]           Source Temp (C)         [148]															
Flammer Countext [0.11 MS Parameters LM Res [125] HM Res [125] Ion Crengy Rang [1.0] Multiplier (V) ISS															
Brase .			3.0	4.0	5.0	6.	3.0	18.0	20	3.0	28.0	30	3.0 Press	32.0 for Operate	3
Ready								Vac	uum OK			5.4	ndby		-

2. Set the values as shown above.

The following table describes the CI parameters to check:

Parameter:	CI+ Values and Comments:
Electron energy	30 eV
Emission	Should be below 200 $\mu$ A, although 200 to 300 $\mu$ A is acceptable. (Above 200 mA may cause hydrocarbon "cracking" patterns with methane and isobutane.) Emission measures the real emission current, i.e. the source current from the source block, there is no trap "source current" in CI.
Lens 1 and 2	The tuning of these lenses may be different from the optimum values set for EI, since the source pressure is much higher in CI.
Multiplier	200V to 600V
Ion Energy	Approx. 1. Similar to EI.
Source temperature	150 °C

#### Adjust the Reagent Gas for CI+

When running in the CI+ mode with reagent gas off, the resulting EI spectra have about 10x lower sensitivity than with the EI source.

If using methane reagent gas, the reagent ions at m/z 17  $(CH_5^+)$  and 29  $(C_2H_5^+)$  should be of approximately equal intensity. Maximize the m/z 29 intensity. With m/z 29 maximized, the ion at m/z 16 should be about 1% of the m/z 17 peak height. (Higher indicates a leak at the transfer line/inner source connection.) Operate slightly to the low-pressure side of the maximum to minimize gas load on the MS. (The vacuum gauge pressure will be  $1.5 \times 10^{-4}$  to  $5 \times 10^{-4}$  Torr.)

If using ammonia reagent gas, reagent ions at m/z 18 (NH<sub>4</sub><sup>+</sup>) and 35 [(NH<sub>3</sub>)<sub>2</sub>H<sup>+</sup>] should be present and the ions at m/z 35 should be optimized.

If using isobutane reagent gas, the reagent ions at m/z 43 ( $C_3H_7^+$ ) and 57 ( $C_4H_9^+$ ) should be tuned in the approximate ratio of 1:2.

The following example uses methane reagent gas.

**NOTE:** The CI gas adjustment knob controls a delicate needle valve. To avoid damaging the needle valve, do not overtighten it. Always use the CI gas button on the screen to turn off the CI gas.

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#### Figure 66 CI reagent gas needle valve adjustment knob

- 1. Carefully turn the delicate CI Gas adjustment knob fully clockwise until you feel it stop.
- 2. Select **CI Gas On** from the Gas menu. A check mark appears next to the option.
- **NOTE:** Always turn on the CI gas before Operate to avoid a pressure surge hitting the filament.

- 3. Click **Press for Operate** and monitor the Penning gauge as you adjust the CI gas. Observe that mass 16 initially grows larger. As pressure increases in the ion chamber of CI source, the mass 29 peak will begin to grow. Keep the pressure below 5 e-4 Torr.
- 4. When using methane gas, carefully turn the CI adjustment knob counterclockwise until m/z 16 is low or non-existent, and m/z 29 is maximized. As you turn the knob, reduce the multiplier voltage to keep the peaks on scale. A typical multiplier value is 235. m/z 17 and 29 will typically be 80 100%.
- 5. Continue to turn the knob counterclockwise. Observe that the pressure increases and mass 41 will start to grow. Stop when mass 29 is at 100%.

6. Turn the knob to maximize the intensity of mass 29. Also verify that mass 16 is small (< 1.0% of the height of the peak at mass 17).

TunePage - c:\ci.pro\acc	udblei+.ipr														2 🔀
	5 18 = = ks ks.	8													
CI+ Source Diagnostics	Status Vacuum OK		Mas 4 17 82 41	1 	Span I I I	Gain 16 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		C	Torr	)					
		53.7.N		17.0		100	0%	29.0		×	3.5%		41.0		20
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Multiplier (V) 225				i		1.									
		5.0	16.0	17.0	18.0	19 7.0	28.0	29.0	30.0	31	3.0	40.0	41.0	42.0	43
Acquire												P	hess for Sta	ndby	
leady		1					Vacuum (	ж			1	Operate			

If mass 16 does not appear as a small peak, STOP. You probably have a gas leak at the transfer line/inner source connection. Locate and correct the leak.

7. After you have maximized the peak, slightly decrease the reagent gas by turning the knob clockwise 1/8 turn.

8. Tuning may be optimized on the m/z 69, 219, 414, and 652 ions of the heptacosa reference gas.

🗹 TunePage - c:\ci.pro\acqudb\ci+.ipr				
File Ion Mode Calibration Gas Options Help				
Cl+ Souce Disprovince	Mass 9 V 1 69 4 V 2 200 4 V 3 414 4 V 4 652 4	Gain         Gain           20         4           2         16	Torr 4.9e-5	
GC Interface Intel Line Temperature 193 200 Source Parameters	69.0 11.9% ×	219.0 X 56.7% ×4	414.0 X 100.0% ×2	652.0 🔀 13.6% ×16
Electron Energy         30         30				
MS Parameters M Res  125M Res  120 Ing Energy Rang  15 Mukpler (M)  450	7.0 69.0	71 219.0	414.0	652.0
Acquire				Press for Standby
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Start TurboMass - Cl 🗹 TunePage - c:\c	ACQUD8	🖴 My Documents 🛛 🖳 Docum	ent1 - Mi Type to search	G 🕤 🔇 🥙 🥵 2:30 PM

9. Click **Press for Standby** to turn off Operate, followed by the CI gas.

You are now ready to run your CI+ analysis.

#### Adjust the Reagent Gas for CI-

1. Open the CI gas inlet by selecting **CI Gas** from the **Gas** menu. Wait at least 10 seconds before clicking **Press for Operate**.

Parameter:	CI– value and comments:
Electron Energy	30 to 70 eV (This parameter should be optimized.)
Emission	$200$ to $300 \ \mu A$ is acceptable (Emission measures the real emission current, i.e. the source current from the source block, there is no separate measurement of source current in CL)
Lens 1 and 2	The tuning of these lenses may be different from the optimum values set for EI, since the source pressure is much higher in CI.
Multiplier	200V to 600V
Source temperature	150 °C is standard. Higher temperatures keep the source cleaner, but may increase fragmentation.
	For example, down a little from EI to minimize fragmentation. 120 °C is the practical lower limit.
Ion Energy	Approx. 1 or 2

2. Optimize the amount of reagent gas flowing into the source by using two heptacosa ions, m/z 452 and 633, which usually produce relative intensities of 65 – 85% and 95% respectively.

Heptacosa can be used to calibrate the m/z range for negative ion CI analyses.

3. Maximize the peak intensities, then slightly decrease the reagent gas by turning the knob clockwise 1/8 turn.



Optimize the tuning parameters for maximum intensity.

- 4. Save the Tune page parameters by selecting **Save As...** from the **File** menu.
- 5. Select **Calibrate Instrument** from the Tune page **Calibration** menu.
- 6. Select **heptaneg.ref** from the drop-down menu.

**CAUTION** *Make sure the "Use Air Refs" check box is not selected.* 

7. Click the **Start** button to display the following dialog box.

Automatic Calibration
Types
✓ Static Calibration
☑ Scanning Calibration
Scan Speed Compensation
Acquisition Parameters
Process
Acquire & Calibrate
Acquire & Verify
Print <u>R</u> eport
OK Cancel

8. Click Acquisition Parameters and enter the following values.

Calibration Acquisition	n Setup		×
Acquisition Parameter Scan <u>F</u> rom Scan <u>I</u> o <u>B</u> un Duration	210 700 .5	amu amu mins	Cancel
<u>D</u> ata Type	Centroid	<b>-</b>	
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Static S <u>p</u> an ±	4	amu	
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Slow <u>S</u> can Time	2	sec	
<u>F</u> ast Scan Time	0.1	sec	
Inter S <u>c</u> an Delay	0.1	sec	
			]

Click **OK** to begin calibration.
 You are now ready to run CI analysis.

# Troubleshooting 5

Clarus 600 MS Hardware Guide

->>

The following sources of problems can occur in gas chromatography and mass spectrometry:

- *The operator:* When the operator is new to chromatography/mass spectrometry and/or a new instrument, problems can be introduced during the learning curve. Once the operator becomes familiar with both the technique and the instrument, this problem source diminishes greatly.
- *The sample:* Unlike clean standards, real world samples such as environmental samples can introduce problems because they are difficult to handle, have complicated matrices, contain unknown constituents, etc.
- *The column:* The column is most often the major factor contributing to poor analyses. The more a column is used, the greater the possibility of contamination, loss of substrate, etc. Columns do not last forever and should be changed when results become suspect.
- *The gas flow system:* Gas leaks are a major concern in gas chromatography and can lead to many problems.
- *The vacuum system:* Vacuum leaks are a major concern in mass spectrometry and can lead to many problems.
- *Ion Optics:* Over time, the ion optics can become contaminated. This results in reduced sensitivity and difficult or impossible tuning.
- *The electronics:* The problem must be identified as either chromatographic or hardware. Electronics used in the system can malfunction.
- **Data handling:** Today, most chromatographers rely on sophisticated data handling systems to integrate their results. Some problems can be related to the incorrect data handling parameter settings or hardware problems with the computer.

## Spare Components

The following list contains items you should have on hand to help solve problems.

- *New syringes:* a syringe can break, become plugged or begin leaking. Always have spare syringes available.
- **Duplicate columns:** a column does not last forever; always have a duplicate column on hand in the event that your separation begins to degrade. Also, capillary columns can be damaged if oxygen is introduced at high temperatures. A duplicate column will allow you to determine if the column is the cause of the problem.
- *Septa:* this is the one area of the gas chromatograph that requires routine maintenance. Always have spare septa available.
- *Leak detector:* the gas flow system can be a problem as fittings wear with age and can begin to leak. You should have a thermal conductivity leak detector to help find and fix leaks.
- *Injector liners:* are made of glass or fused silica and can be easily broken when removed. You should keep a supply of spare liners on hand. Please remember that you cannot run satisfactory analyses without an injector liner.

## Logical Troubleshooting Steps

There are some simple steps that you should take when trying to locate a problem. Use the following guide to troubleshoot your system.

- 1. Note the symptoms define the problem. Compare your runs with good analysis, that is, with the results normally obtained.
- 2. Systematically eliminate possible causes.

The first rule here is, "What did you change last?" Many times a problem arises when a change is made to the system, such as changing a gas tank, column, septum or glass liner. If the problem occurred after such a change, then the change is the most likely cause of the problem.

Change the simplest thing first. For example, if you suspect a gas leak, the easiest change to make is the GC septum instead of replumbing the internal pneumatics.

Change only one parameter at a time and check for its effect. If you change three items at once and your problem goes away, you may not know which of the three moves or combination of moves corrected the problem. This way, if the problem happens again, you will know exactly what corrective action to take.

## Troubleshooting Chart

Problem	Probable Cause	Solution
Mass Spectrometer will not turn on (no indication of power to the instrument).	AC line cord not plugged into an AC outlet.	Plug the Mass Spectrometer AC line cord into an AC outlet.
	No AC power to the outlet.	Check the outlet.
	Fuse blown.	Call a PerkinElmer service engineer.
Mass Spectrometer is on but the forepump is not running.	Forepump is not plugged into the AC outlet on the rear of the mass spectrometer.	Plug the forepump line cord into the mass spectrometer.
	Forepump is not operating correctly (mass spectrometer does not pump down).	Make sure the forepump is switched on.
	Blown fuse in Mass Spectrometer.	Call a PerkinElmer service engineer.
Problem	Probable Cause	Solution
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The ultimate pressure is poor.	Is the cooling inadequate?	Check the cooling-air flow and correct if possible.
		Check the cooling-air duct for obstructions and correct as necessary.
		If the cooling air flow is fine and there are no obstructions contact your PerkinElmer service representatives.
	Is the backing pressure high?	Check for a leak in the backing pipeline and poor backing pump performance. Correct as necessary.
The pump is very noisy or there is excessive vibration or both.	Is the noise irregular and getting progressively worse?	If so, a bearing may be defective. Contact your PerkinElmer service representative.
	Is the pump making a constant high pitched noise?	If so, the rotor may be out of balance. Contact your PerkinElmer service representative.

Problem	Probable Cause	Solution
Turbo pump will not accelerate	Pump malfunction.	Call a PerkinElmer service engineer.
	Pump controller malfunction.	Call a PerkinElmer service engineer.
	GC is not properly configured.	Set the proper GC configuration for your site.
Vacuum light continues to blink.	Large leak.	Locate vacuum leak and correct.
	Foreline trap has excessive moisture.	Replace filter.
	Rotary pump set to gas ballast.	Switch the gas ballasting off.
	Rotary pump requires oil change.	Change oil.
High mass spectra appears as a large blotch, or loss of high mass spectra.	Bad tune.	Run AutoTune.
Drastic change in mass peak shape for no apparent reason.	Bad tune.	Run AutoTune.
No spectra, or large blotch.	Bad tune.	Run AutoTune.

Problem	Probable Cause	Solution
No spectra, or very little spectra at the low mass end.	Bad tune.	Run AutoTune.
No spectra, not even noise at a high PMT voltage.	Loose electrometer cable.	Reset the cable.
	Defective electrometer board.	Call a PerkinElmer service engineer.
No filament current.	Defective filament.	Replace the filament.
Wavering baseline (by several hundred counts).	Defective outer source temperature sensor or a defective electrometer.	Replace the temperature sensor.
		Call a PerkinElmer service engineer.

Problem	Probable Cause	Solution
Poor or inadequate sensitivity.	Leaking injector septum.	Replace the septum.
	Leak from injector ferrules.	Tighten/replace ferrules.
	Foreign material in the injector.	Clean the injector.
	Peak splitting.	Prevent double injections.
		Dry the outside of the injector needle.
		Replace the injector septum.
	Injector and column are more active toward acid/base compounds.	Install a silanized injector liner, or silanize the current injector liner.
		Check or replace the injector packing material, such as quartz wool.
		Cut off the first 30 cm of the column and rerun the test mix. If the results do not improve replace the column.

Problem	Probable Cause	Solution
Solvent tailing.	Inadequate splitter flow.	Increase the splitter flow.
	Column not properly installed in the injector.	Reinstall the column in the injector.
Loss of high end compounds.	Temperature setting too low on the injector, column oven or transfer line.	Increase the injector, column oven or transfer line temperature to allow the less volatile compounds of the sample to reach the mass spectrometer.
Peaks at masses 28 (nitrogen) and 32 AMU (oxygen) are in a ratio of < 4 to 1, and the peak at mass 28 is larger than the peak at mass 18.	Leaks in or around vacuum or column fittings.	Tighten the fittings and connection points to the high vacuum system. Set the injector split flow to 50:1.
Peaks at masses 14 and 16 are larger than the peak at mass 28.	Leaks or improper tuning.	Tighten the fittings and connection points to the high vacuum system. Set the injector split flow to 50:1. Run AutoTune.

Problem	Probable Cause	Solution
Poor sensitivity (correct amount of sample is reaching the mass spectrometer).	Column is improperly positioned in the ion source.	Reinstall the column and check the cut at the end that fits in the source.
	Improper tuning, or a dirty or defective ion source.	Check the tuning. Increase the PMT voltage.
		Shut down the system, remove the inner source, clean or replace lenses in the outer source, and install a new filament.
Loss of resolution (especially at high mass).	Dirty prequadrupole rods.	Clean the prequadrupole rods.
Mass assignment drifts.	Large temperature fluctuations in the laboratory.	Stabilize the lab temperature, or isolate the GC/MS system from large temperature fluctuations.
Mass assignment incorrect.		Run mass calibration.
Skewed spectra.	Improper scan speed (too slow for the narrow peaks produced by capillary columns).	Increase the scan speed.
Unusually high repeller voltage.	Repeller dirty.	Clean repeller.

Problem	Probable Cause	Solution
Unusually high emission setting.	Ion volume dirty.	Clean the inner source.
	Prefilters dirty.	Clean the prefilters.
	Poor tuning.	Retune.
Tuning peaks show precursors (forward slope shoulders).	Poor tuning.	Retune.
	Dirty prefilter.	Clean prefilters.
	Dirty source.	Clean source.
	Particulates on analyzer rods.	Clean the particulates of the rods with a methanol-dampened lab wipe.
		Blow the particulates off the rods with helium or dry nitrogen.
	Defective or damaged analyzer.	Call a PerkinElmer service engineer.
No reference peak.	Reference gas off.	Turn on the reference gas.
	Empty reference vial.	Visually check and refill.
	Faulty solenoid.	Listen for click when activating/deactivating the valve.

Problem	Probable Cause	Solution
Inconsistent peak widths.	Poor tubing.	Retune.
	Ground loop from GC and MS on different power supplies.	Unify supplies.
Peaks shifted from their nominal mass position.	Poor calibration.	Perform mass calibration.
Tuned peaks are too narrow.	Over-resolved tuning.	Retune.
Tuned peaks are too wide.	Under-resolved tuning.	Retune.
Big peaks observed at m/z 18, 28, 32.	Air leak developed.	Check column connections.
	Change carrier gas tank.	Fit oxygen scrubber.
	Moisture from recent source clean/column change.	Bake out source overnight.
No ion beam but the filament status OK.	Large air leak.	See procedure for leak- checking.
	Detector voltage too low.	Increase PMT value.
	Electronics failure.	Call a PerkinElmer service engineer.
Poor sensitivity. Beam instability/peaks breaking up.	Column improperly installed.	Check and reinstall the column if necessary.

Problem	Probable Cause	Solution
	Piece of column broken off in the ion chamber.	Remove the inner source, check for and remove piece of column.
	Source filament is bent.	Check and replace filament if necessary.
	RF generator malfunction.	Call a PerkinElmer service engineer.
	Analyzer drive electronics malfunction.	Call a PerkinElmer service engineer.
Total Ion Chromatogram too high.	Dirty source.	Clean the source.
	Contamination from poor handling technique.	Set source and transfer line to 250 °C and maintain this temperature overnight.
	Stationary phase of column de-polymerizing (bleeding).	Change column.
	Air leak.	Find the leak and fix it.
	Poor quality carrier gas.	Replace the carrier gas tank.
	Carrier gas filter is ineffective and needs replacing.	Replace the carrier gas filter.
No noise on mass chromatogram.	Detector multiplier voltage too low.	Increase the multiplier voltage.

Problem	Probable Cause	Solution
Excessive noise.	Dirty source.	Clean the source.
	GC and MS on separate power supplies.	Connect GC and MS together with the ground strap.
	PMT voltage too high.	Run AutoTune.
	Data acquisition thresholds set too low.	Raise the thresholds.
Instrument won't calibrate (after retuning and recalibrating).	Poor AutoTune/Manual tune.	Retune.
	Contaminated ion source.	Clean the ion source. Set the source temperature to 250 °C and maintain this temperature overnight.
	Source too hot/cool.	Set the correct source temperature.
	Air leak.	Find the leak and fix it.
	Wrong calibration reference file selected.	Select the correct file.
	Incorrect calibration calculation parameters.	Set the calibration parameters to the default values.
	No calibration gas.	Refill the calibration gas vial.
	Incorrect electron energy.	Reset to 70 eV.

# Chromatography Related

Problem	Probable Cause	Solution
Inconsistent retention time.	Injector septum leak.	Replace the septum.
	Carrier gas manifold leak.	Locate and fix the leak.
Rising Total Ion Chromatogram baseline.	Column bleed.	Disconnect the column from the mass spectrometer and condition the column.
	Vacuum leak.	Locate and fix the leak.
Discreet high intensity contaminant peaks.	Column bleed.	Disconnect the column from the mass spectrometer and condition the column.
	Injector septum bleed.	Replace the septum and/or glass liner.
Tailing peaks (sloping on RHS).	Improperly installed column.	Check the column and reinstall if necessary.
	Injector too cool.	Raise the injector temperature.
	Interface temperature too cool.	Raise the interface temperature.
	Inadequate carrier gas flow.	Set proper flow.

Problem	Probable Cause	Solution
	Dirty injector liner.	Clean or replace.
	Column has active sites.	Equilibrate or replace.
Chromatographic peaks too wide.	Injector too cool.	Raise injector temperature.
	Sample overloading the column.	Use a split injection or a smaller sample.
	Incorrect GC oven program.	Enter a new oven program.
Discrimination of relative peak intensities.	Poor resolution or improper tuning.	Retune.
	Unstable filament.	Replace filament.
	Poor calibration.	Recalibrate.
	Air leaks at detector.	Check He/Air ratio.
Peaks are flat-topped.	Signal strength exceeds dynamic range of detector.	Reduce PMT voltage.
	Sample is too strong.	Dilute or split.
High baseline.	Dirty sample.	Prepare and filter a new sample.
	Air leak at injector.	Locate and fix the air leak.
	Contaminated carrier gas.	Replace the gas tank.

Problem	Probable Cause	Solution	
Slowly falling baseline (from a high initial value).	Split valve left closed during acquisition.	Open the split valve.	
	Inadequate purge flow rate.	Increase flow rate.	
	Poor off for too long.	Reduce purge time.	
Low sensitivity.	Dirty source.	Clean the source.	
	Poor column performance.	Replace column.	
	Dirty injector.	Replace injector liner.	
	Source temperature not optimized.	Set the proper source temperature.	
	Detector voltage set too low.	Increase PMT voltage.	
	Tune not set correctly.	Run AutoTune.	
	Poor filament alignment.	Realign or replace filament.	
	Incorrect column position in the source.	Reposition the column.	

Problem	Probable Cause	Solution	
Poor reproducibility.	Dirty source.	Remove and clean the source.	
	Defective injector liner.	Replace injector liner.	
	Defective syringe.	Replace syringe.	
	Old or damaged filament.	Examine and replace filament.	
	Poor tuning.	Retune.	
	Poor calibration.	Recalibrate.	
	Air leak.	Locate and fix.	
	Active sites in column/liner.	Replace column/liner.	
	Intermittent source heater failure.	Call a PerkinElmer service engineer.	
Poor S/N on test standards.	See low sensitivity causes above.		
	Incorrect GC/MS method	Use the correct method.	
	Accidental split injection.	Set the proper split.	
	Detector voltage set too low.	Increase PMT voltage.	
	Column flow rate too high.	Reset the column flow rate.	

# Spectral Related

Problem	Probable Cause	Solution
Noisy spectra.	Dirty source.	Remove and clean the source.
	Peak detection threshold set too low.	Raise the thresholds.
	PMT voltage set too high.	Lower the PMT voltage.
Spectrum distortion.	Scanning too fast or slow.	Reset the scan rate.
Incorrect Isotope ratios.	Poor calibration.	Recalibrate.
	Incorrect tune.	Retune.
	Defective filament.	Replace filament.
	Air leak.	Find and fix.
Missing Isotopes in spectrum.	Bad calibration.	Recalibrate.
	Poor tuning.	Retune.
	Dirty source.	Clean source.
	Sample too weak.	Use a higher sample concentration.
	Peak detection thresholds set too high.	Lower the thresholds.
	PMT voltage set too low.	Raise the PMT voltage.

Problem	Probable Cause	Solution	
	Contamination.	Locate the contamination and eliminate it.	
	Co-eluting components.	Change your sample preparation or chromatography.	
	Incorrect column alignment.	Reinstall the column.	
Section of a mass range missing from a spectrum.	Corruption of data file.	Reacquire data.	
	Scanning too fast.	Reduce the rate.	
	Hard disk has too much fragmentation.	Defrag the hard drive.	
	Hard disk full.	Remove unnecessary files.	
Molecular Ion too weak.	Source temperature too high.	Reduce the source temperature.	

# **Communications Related**

Problem	Probable Cause	Solution
Will not boot MS.	PC (computer) to MS cable has a loose connection.	Check and reset the cable.
	Transient in power supply has halted communications.	Reboot the PC (computer).
Will not control GC.	RS 232 communications cable loose connections.	Check and restart the mass spectrometer.
	Power failure/transient surge to GC or autosampler.	
	GC electronic malfunction.	Call a PerkinElmer service engineer.
Communication cable intermittent contact.	GC electronics malfunction.	Call a PerkinElmer service engineer.
Crashes when starting an acquisition.	Software corrupted.	Reload software.
	Rotary pump malfunction.	Call a PerkinElmer service engineer.

# Forepump Related

Problem	Probable Cause	Solution	
Pump does not start.	Forepump switched off.	Switch on the pump.	
	Blown fuse.	Call a PerkinElmer service engineer.	
	Electrical supply voltage does not match that of the pump motor.	Determine the correct voltages, and correct. Check the voltage switch at the pump.	
	The outlet filter is blocked.	Find and unblock.	
Pump has failed to reach vacuum.	Pressure measurement or gauge head gives an incorrect indication of pressure.	A contaminated Pirani gauge can indicate a pressure several times higher that the actual. Replace if necessary.	
	Pump contains the wrong type of oil.	Drain and refill with the correct oil - Edwards Ultragrade 19 Oil. Consult your Edwards Pump instruction manual.	
	Mode selector and/or gas ballast control are incorrectly set.	Check and set to correct position.	
	High oil level.	Drain to the high oil level mark.	
	Low oil level.	Check and fill to correct level.	

Problem	Probable Cause	Solution	
	Contaminated oil.	Drain and refill with new oil.	
	Vacuum fitting dirty or damaged.	Check and replace if necessary.	
Noisy Pump.	Motor fan cover damaged. Call a PerkinElmer servi engineer.		
	Worn motor bearings.	Call a PerkinElmer service engineer.	
	Oil contaminated with solid particles.	Determine cause and replace oil.	
	Oil saturated from CI analysis.	Drain and refill with clean oil.	

Problem	Probable Cause	Solution	
External oil leak.	Outer shaft seal worn or damaged.	Call a PerkinElmer service engineer.	
	Oil box gaskets deteriorated.	Call a PerkinElmer service engineer.	
	Oil leak from gas ballast control.	Call a PerkinElmer service engineer.	
	Oil leak from drain plug.	Tighten the drain plug or replace.	
	Oil leak from sight glass.	Tighten sight glass screws or call a PerkinElmer service engineer.	

When operating the instrument message dialog boxes will sometimes appear. The following table is the Icon Key followed by tables that show the Message Title, icon, dialog message and recommended action.

## Icon Key

Icon	Meaning
8	Press this icon to close the message.
i	Press this informational icon to get more details on the message.

Message Title	Icon	Message	Action
Diffusion pump failure		There is a problem with the diffusion pump. Either the diffusion pump fan has failed or the pump has over heated. Please look in the Hardware Guide for additional instruction.	<ul> <li>Press OK to close the message.</li> <li>Check the cooling-air flow and correct if possible.</li> <li>Check the cooling-air duct for obstructions and correct as necessary.</li> <li>If the cooling air flow is fine and there are no obstructions contact your PerkinElmer service representatives.</li> </ul>
System not at pressure	Caution	The system has not reached the proper operating pressure. The filament could be damaged by starting the system now. Do you wish to continue?	<ul><li>Press Yes if you wish to continue.</li><li>Press No if you wish to stop.</li><li>See the Maintenance chapter in this <i>Hardware Guide</i> for the procedure to replace a filament.</li></ul>
Safe to vent	į	The vacuum system is off and the system can now be vented. The GC carrier gas should be turned off.	Press <b>OK</b> to close the message.

Message Title	Icon	Message	Action
Diffusion Pump failure		There is a problem with the diffusion pump. Either the diffusion pump fan has failed or the pump has over heated. Please look in the Hardware Guide for additional instruction.	<ul> <li>Press <b>OK</b> to close the message.</li> <li>Check the cooling-air flow and correct if possible.</li> <li>Check the cooling-air duct for obstructions and correct as necessary.</li> <li>If the cooling air flow is fine and there are no obstructions contact your PerkinElmer service representatives.</li> </ul>
Vacuum Leak Detected		The backing pump could not reach the necessary vacuum level to start the diffusion pump. There could be a problem with a vacuum leak, the backing pump or the vacuum gauge. The backing pump will be turned off. Make sure that the vent valve is closed before restarting the backing pump.	Press <b>OK</b> to close the message. Check the system for any leaks and correct. If the problem continues contact your PerkinElmer service representatives.

Message Title	Icon	Message	Action
Vacuum Gauge Failure	8	There is a problem with the vacuum gauge.	Press <b>OK</b> to close the message. Restart the system, if you still have this failure message contact your PerkinElmer service representatives.
Pump failure- Safe to Vent		The vacuum system is off and the system can now be vented. The carrier gas should be turned off. There is a problem with the diffusion pump. Either the diffusion pump fan has failed or the pump has over heated.	Press <b>OK</b> to close the message. Check the cooling-air flow and correct if possible. Check the cooling-air duct for obstructions and correct as necessary. If the cooling air flow is fine and there are no obstructions contact your PerkinElmer service representatives.

Message Title	Icon	Message	Action
Vacuum Leak- Safe to Vent		The vacuum system is off and the system can now be vented. The carrier gas should be turned off A Vacuum Leak has been detected. Please look in the Hardware Guide for additional instruction.	Press <b>OK</b> to close the message. Check the system for any leaks and correct. If the problem continues contact your PerkinElmer service representatives.
Vacuum Gauge Failure-Safe to Vent		The vacuum system is off and the system can now be vented. The carrier gas should be turned off. There is a problem with the vacuum gauge. Please look in the Hardware Guide for additional instruction.	Press <b>OK</b> to close the message. To replace the vacuum gauge contact your PerkinElmer service representatives.
Exit TurboMass- Vacuum System pumping down	i	The vacuum system is in the process of the pumping down the spectrometer. Exiting TurboMass at this time may prevent a successful pump down.	Press <b>OK</b> to close the message.
Exit TurboMass- Vacuum System pumping down	i	The vacuum system is in the process of shutting down. Exiting TurboMass at this time may prevent a successful completion of this task.	Press <b>OK</b> to close the message.

Message Title	Icon	Message	Action
Backing Pump is on	i	The system is not in an operating state. A diffusion pump failure, a vacuum leak or a vacuum gauge failure could have occurred. If the transfer line or the source temperatures are above 100C, please wait until they have cooled before pressing OK.	If the transfer line or the source temperatures are above 100C, please wait until they have cooled before pressing <b>OK</b> . Pressing the <b>OK</b> button will turn off the backing pump.
		Pressing the OK button will turn off the backing pump.	
Problem with Vacuum	i	The backing pump could not reach the necessary vacuum level to start the diffusion pump. There could be a problem with a vacuum leak, the backing pump or the vacuum gauge. The backing pump will be turned off. Make sure that the vent valve is closed before restarting the backing pump.	Press <b>OK</b> to close the message.

Contact PerkinElmer for Columns, Supplies, Accessories, and Replacement Parts.

Supplies, accessories and replacement parts can be ordered directly from PerkinElmer's catalog service. PerkinElmer offers a full selection of high-quality chromatography data handling products and gas chromatography supplies and columns through the *Gas Chromatography Supplies Catalog* and the *Gas Chromatography Column Catalog*.

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Telephone:

- U.S. only: Call toll free 1-888-PE-CHROM, 8 a.m. to 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- Worldwide: Call your local PerkinElmer sales or service office or call PerkinElmer, Shelton, CT USA 1-203-925-4600.

Internet: http://www.perkinelmer.com

e-mail: chrom@perkinelmer.com

Clarus 600 MS Hardware Guide

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