

DSMS Gas Analysis System Operator's Manual

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Welcome to the DSMS Operator's Manual

The purpose of this manual is to provide information on the DSMS Gas Analysis system.

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Warnings and Cautions

In this Manual, a **Warning** is an instruction that draws the operator's attention to the risk of injury or death; a **Caution** is an instruction that draws attention to the risk of damage to the product or process.

Warnings and Cautions are placed immediately before the text to which they refer; they are headed by **WARNING** or **CAUTION** respectively. The associated explanatory text is in **bold**. If several Warnings or Cautions apply at one point in the text, they are numbered with the most important appearing first.

Typographical conventions

For ease of identification the names of menu commands, keys, dialog items and screen text are typographically distinct from the ordinary text of this Manual. These distinctions are as follows:

Menu commands, dialog items, such as buttons and check boxes, and text that appears on the display screen are presented in bold typeface; thus **File** menu, **Enter** button.

Keys are presented in bold, italic text; thus *Esc*, *Return*, *Space bar*.

Terminology

Terminology that accords with basic Windows principles is included in this Manual.

Instructions

For clarity, the instructions given in this Manual are presented in two columns. The left-hand column provides imperative instructions that are numbered sequentially to provide a step-by-step guide through the functions. The right-hand column describes the system's response (where appropriate) and gives any additional information that may be of relevance.

Importance of this Manual

This Manual should be regarded as part of the product described herein.

Technical assistance

Hiden Analytical Limited try to ensure the information presented in this manual is comprehensive, relevant and accurate. If the reader thinks there is a mistake, requires some clarification or needs further information please do not hesitate to contact Hiden.

Technical assistance can be obtained from the Hiden Analytical Limited Service Department which can be contacted on:

Email:	service@hiden.co.uk
Tel:	+44 (0)1925 445225
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In the U.S.A. and Canada, technical assistance can be obtained from Hiden Analytical Inc.:

Email:	service@hideninc.com		
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Toll-free phor	ne: 1-888-96 HIDEN Option 1 U.S.Sales Office Option 2 U.S.A. & Canada Corporate Office & Service Department Option 3 U.K. Manufacturing Facility		

Details of worldwide local support can be found on the Hiden Analytical website: www.HidenAnalytical.com.

Amendments

This Manual will be updated, as necessary, to cover modifications to the product. Minor amendments may take the form of Addenda, which will normally be located at the back of the Manual.

1 Safety

1.1 Relevance

This manual relates to the Hiden Analytical Limited DSMS quadrupole mass spectrometer gas analysis system.

This manual describes the hardware, installation procedure, operation and maintenance.

DSMS component modules have their own manuals which will be supplied with this manual.

Hiden Analytical's MASsoft operating software used in conjunction with the DSMS is described in the MASsoft manual. The DSMS may be supplied with other Hiden software applications which will have their own manuals.

1.2 Warnings and cautions

In this manual WARNINGS and CAUTIONS have the following meanings and are denoted as shown.





CAUTION

CAUTIONS draw attention to procedures, practices, conditions which if not performed correctly or adhered to could result in damage to the equipment described in this manual and/or to peripheral equipment connected to the equipment described herein.

1.3 System use



If in doubt as to its suitability for an application, please contact Hiden Analytical Limited for advice.

WARNING

- 1. The equipment must be fully installed and configured for normal operation before it is powered-up; failure to do this may cause injury to the operator and damage to the equipment.
- 2. The equipment must be earthed; failure to do this may cause injury to the operator and damage to the equipment.



CAUTION

- 1. Any connections to other equipment should be undertaken by suitably qualified personnel and must not compromise equipment safety.
- 2. Any equipment connected to any input/output port must operate at Safe Extra Low Voltage (SELV) levels (normally in the range -50 V to +50 V) in accordance with EN 61010:1993 to meet the requirements of the Low Voltage Directive (LVD) 73/23/EEC.



CAUTION

The mains power plug and socket is the primary disconnect device for the DSMS; it must be easily identifiable and accessible by the operator.



WARNING

HAZARDOUS GASES

Users must be fully aware of all safety requirements related to gases being sampled by the instrument. DSMS users must make themselves aware and adhere to all of the necessary safety precautions related to any hazardous gases, toxic gases or potentially explosive gas mixtures sampled by the instrument.

If in any doubt the user's on-site safety officer or department should be contacted for advice.

Information regarding potential gas hazards can be obtained by, but not limited to, reading the applicable COSHH (Control of Substances Hazardous to Health) data sheet and taking advice from the gas supplier.







Do not use silicone grease on any seals connected to the mass spectrometer vacuum chamber as there is a risk of ion source/quadrupole contamination leading to significant loss in system sensitivity.



WARNING

Protective gloves and face mask must be worn when removing the inlet unit's cover.

The Heated Molecular Leak Assembly incorporates ceramic paper insulation which may pose a long term health risk due to sub six micron filament size fibres in the material.

1.4 **Symbols**

1.4.1 General

The following symbols may be found on the equipment:



Caution, risk of electric shock.

Background colour: yellow. Symbol and outline: black.

The above symbol is displayed adjacent to connectors carrying voltages greater than 1000 V.

Symbol and outline: black

Background colour: yellow. Symbol and outline: black.

The above symbol is displayed on surfaces that reach high temperatures or on covers which, when removed, reveal hot surfaces.

The WEEE directive symbol on the rear panel of the RC Interface

The disposal of the equipment described in this manual MUST be in accordance with local regulations.

For EU member countries please contact Hiden Analytical Limited for

2 Introduction

2.1 Documentation

This manual describes the DSMS quadrupole mass spectrometer gas analysis system. The system hardware, installation, operation and maintenance procedures are described in this manual. Additional options and features may be described in addenda which accompany this manual.

DSMS systems are used with Hiden's MASsoft Windows based software which is described in the MASsoft manual.

In addition to this manual each of the modules used in the DSMS has its own manual which is supplied with the system.

Manuals for the vacuum pumps and gauges will also be supplied with the system.

2.2 Contents sheet

Each DSMS system is supplied with a configuration information document placed at the front of the white Hiden manuals ring binder. This is a custom document describing the features and options fitted to the particular system. This document has a reference number beginning HA-111-. The first page of this document is the Contents Sheet which lists all the manuals relevant to the particular instrument.

2.3 Introduction to the DSMS

Hiden Analytical Limited quadrupole mass spectrometers may be directly connected to user's systems where the monitored pressures are from the Ultra High Vacuum (UHV) region up to approximately 1x10⁻⁴ mbar; analyses at higher pressures require additional vacuum pumps and the mass spectrometer gauge head mounted in a suitable housing. The DSMS Gas Analysis System provides these features.

The DSMS Gas Analysis System is designed for applications requiring optimum sensitivity and stability. The system is typically configured with cross-beam sample ionisation to minimise contamination and memory effects.

The basic system is configured for use with a UHV turbomolecular pumping system and combined cold cathode/ion gauge pressure measurement. A variety of inlet systems, for sampling from the user's vacuum system or process, are available; these are selected to suit the application.

Each DSMS System is integrated with one of the Hiden Analytical Limited high sensitivity quadrupole mass spectrometers, with mass ranges up to 510 amu.

An optional liquid nitrogen cryo-panel may be fitted which, when combined with the UHV turbomolecular pumping system, will enhance detection levels to the part-per-billion (ppb) regime for many species.

3 System description

3.1 Introduction

Figure 1 shows a typical DSMS Gas Analysis System.

The system is mounted on a supporting frame assembly. This assembly may be trolley-mounted for increased mobility. The trolley mounting option provides additional storage space and rack mounting facilities (front panel mounting) for electronic equipment.

3.2 The ultra-high vacuum pumping system

The mass spectrometer gauge head, total pressure gauge and optional cryo-shield are mounted in the mass spectrometer vacuum chamber. Ports are provided for the turbo pump and inlet system.

Two inlet connection ports may be incorporated in the UHV housing:

- 1. 70 mm diameter Conflat flange, type DN-35-CF, for connection of the main inlet system.
- 2. 34 mm diameter Conflat flange, type DN-16-CF, for connection of additional sample lines generally sampling from medium vacuum levels through an isolation valve and incorporating replaceable orifices for pressure reduction.

The UHV turbo pump is attached to the mass spectrometer vacuum chamber. The UHV turbomolecular pumping set comprises:

- Turbo pump.
- Turbo pump backing pump; this is a scroll pump. Optional a two-stage rotary pump or membrane (diaphragm) pump may be used. The turbo pump and scroll pump provide an oil-free vacuum in the UHV housing.
- Drive electronics.
- Vacuum Control Unit (VCU).
- Venting device.

A turbo isolation valve may be fitted between the UHV housing and the turbo pump to allow isolation of the housing under vacuum before venting; this reduces the pump-down time on restarting the turbo pump.

The VCU interfaces the turbo pump controller, backing pump and venting operations; it also gives a single switch start-up of the pumping set. The VCU also provides interlocking for protecting the mass spectrometer filaments. Automatic venting protects the mass spectrometer and mass spectrometer vacuum chamber from pump oil contamination when the turbo pump is not running.



Figure 1 Typical DSMS System

3.3 Bypass/inlet evacuation rotary pump

Bypass pumping provides pressure reduction when sampling using the atmospheric capillary, and evacuation of the inlet system volume. Pressure in the bypass inlet will, typically, be in the millibar region.

Where a scroll pump is used to back the turbo pump bypass pumping is provided by connection to the backing line. Where a rotary backing pump is used a bypass/inlet evacuation rotary pump is used. Typically, a 2.5 m^3 /h two-stage rotary vane pump is connected to the inlet system via a flexible hose. An integral isolation valve isolates the vacuum line when the pump is switched off.

A foreline trap may be fitted to ensure that there is no pump oil contamination.

3.4 Pressure gauges



CAUTION

In systems where a pressure gauge is not fitted, wait 15 to 30 minutes after switching on the turbo pump before running the mass spectrometer; failure to do this may damage the instrument. Use mass 18 and mass 28, or the mass spectrometer total pressure reading, as an indication of the manifold total pressure.

A cold cathode or active ion gauge is usually fitted to monitor the mass spectrometer vacuum chamber total pressure. An interlock prevents mass spectrometer operation at too high a pressure.

A Pirani gauge may be used for measuring the bypass line vacuum.

3.5 Pressure units

The International System of Units (SI) derived unit for pressure is the Pascal (Pa).

Since the Pa is such a small unit the common multiple unit used for vacuum pressure measurement is the hectopascal (hPa) where 1hPa = 100Pa.

Standard atmospheric pressure is 101,325 Pa or 1013hPa.

In Europe the common unit for vacuum pressure measurement is the millibar (mbar).

Atmospheric pressure is approximately 1bar or 1000mbar and 1bar = 100,000Pa

So very conveniently 1hPa = 1mbar.

In the USA the common unit for vacuum pressure measurement is Torr.

Atmospheric pressure is 760Torr.

Therefore 760Torr = 101325Pa

and 1Torr 133Pa or 1.3hPa and 1.3mbar

 $1hPa = 1mbar \quad 0.76Torr$

3.6 Inlet system

The inlet system and connection lines for the DSMS will depend on the application. The inlet will comprise:

• A heated molecular leak inlet assembly, described in this section

either

• A heated capillary inlet, described in it's own manual

or

• A high pressure capillary inlet, described in it's own manual.

Note

Information in this section is taken from the Hiden Analytical document reference HAV-002-003C.

3.6.1 Safety



During normal operation it is not necessary to remove the unit's cover.

The cover must be removed to carry out maintenance operations such as replacing the molecular leak as described in Section 8.3 of this manual. When removing the cover and carrying out work on internal parts of the unit a face mask and protective gloves must be worn.

The Heated Molecular Leak Assembly incorporates ceramic paper insulation which may pose a long term health risk due to sub six micron filament size fibres in the material.

For further information and Safety Data Sheets please contact Hiden Analytical Limited.

3.6.2 General description

Figure 2 shows the Heated Molecular Inlet Assembly.

The unit is configured for use in conjunction with the HPR20 and DSMS range of mass spectrometer sampling systems.

The inlet enclosure consists of a steel box with ceramic paper insulating lining.

The inlet incorporates a molecular leak assembly, an all-metal bypass isolation/control valve and an all-metal sample isolation valve.

Two heaters are fitted, one inside the enclosure and one around the direct inlet assembly/mounting flange; these may be operated to reduce condensates in the inlet system.





Figure 2 Heated molecular inlet assembly

The normal operating temperature inside the enclosure is 120 °C.

All fittings are stainless steel and the ferrules are Vespel or stainless steel rated to temperatures in excess of 250 $^{\circ}$ C.

Sample enters the inlet via the sampling capillary and passes over the molecular leak into the bypass pump. The pressure in the inlet during sampling will typically be in the 1 mbar region. The sample enters the mass spectrometer ion source via the molecular leak to give a typical mass spectrometer ion source pressure in the 1x10⁻⁶ mbar region.

3.7 The mass spectrometer



Please refer to the appropriate mass spectrometer Operator's Manual; this is supplied with the system.

3.8 Heaters



CAUTION

- **1.** Ensure that the system is under vacuum prior to operating the heaters.
- 2. Switch off the heaters and allow the system to cool for 30 minutes before venting.

3.9 Vacuum Control Unit

DSMS vacuum system control is accomplished by the Vacuum Control Unit (VCU). The VCU provides:

- One button vacuum system start up and shut down
- A display of mass spectrometer vacuum chamber pressure

- A setpoint output to protect the mass spectrometer filament
- Two addition vacuum gauge inputs
- Two additional setpoints for vacuum system interlocking
- 24V DC power for additional control units and ancillary components.



Figure 3 VCU display

- a Pressure reading from the gauge connected to the rear panel G1 connection.This is the total pressure gauge (either a cold cathode or active ion gauge) fitted the mass spectrometer vacuum chamber.
- b Pressure reading from the gauge connected to the rear panel G2 connection. Not used in standard DSMS systems and will be blank because no gauge is connected.
- c Pressure reading from the gauge connected to the rear panel G3 connection. Not used in standard DSMS systems and will be blank because no gauge is connected.
- d The gauge type for each of the three possible gauges. The options are AIM (cold cathode gauge) and AIGX (active ion gauge).
- e Pressure units for the gauge. The options are torr, mbar.
- f Status for the three Setpoints. The options are OK or blank.

Setpoint 1 provides the over-pressure trip signal for the mass spectrometer and is connected to the RC Interface TRIPS MSC11 connector. Setpoints 2 and 3 are not usually used in DSMS systems.

- g Pump status. The options are ON and OFF.
- h Turbo pump speed. Once the pumps are started the turbomolecular pump will start to accelerate, when it has reached full speed 100% will be displayed.

The DSMS total pressure gauge is connected as gauge 1 (VCU rear panel G1 connection) and provides the Set Point 1 signal. Typically the trip pressure is set to $9x10^{-5}$ mbar. When the pressure is greater than $9x10^{-5}$ mbar the Set Point is enabled. The mass spectrometer inhibit circuit is connected to this optically isolated transistor output.

For systems using a mass spectrometer with a pulse counting detector the trip pressure is set to a lower value, typically, $5x10^{-6}$ mbar.

The total pressure gauge may be a cold cathode gauge or an active ion gauge. The gauge type makes no difference to the system's operation.

3.10 Identification

The Works Reference (WR) No. uniquely identifies the instrument and is used by MASsoft.

The number is displayed on the serial number label attached to left side of the system's top cover. The WR number is the five digit number displayed in the **WORKS REF** (**WR**) box, WR 16543 in Figure 4. It is worth making a note of this number if the left side of the system will be inaccessible.

The unit's WR number can be obtained by using the MSIU Test program and typing the command:

pget ID

the WR number will be returned.

INST. TYPE	MASS SPECTROMETER
MANUFACTURER	HIDEN ANALYTICAL
PLACE OF ORIGIN	UNITED KINGDOM
MODEL	DSMS
DATE PRODUCED	25-12-18
VOLTAGE	230 V ac
CURRENT	10 A max,
WORKS REF (WR)	WR 16543
HIDEN	
ANALYTICAL	www.HidenAnalytical.com

Figure 4 Serial number label

4 Technical data

4.1 Electrical specification

Voltage: 100, 110 or 230 V a.c. (~).
Frequency: 50 to 60 Hz.
Power: 1000 VA typical.
Mains fuse: 20 mm HRC, 6.3 A T, 250 V (standard).
Installation category (overvoltage category): II.
Pollution degree: 2.

4.2 Environmental data

5 Installation

5.1 Introduction



The system may be supplied in kit form or fully assembled; this will depend on the system configuration and transit requirements.

For the typical system shown in Figure 1:

- the RF Head would be removed to reduced the chance of damage to the mass spectrometer feedthrough
- the capillary would be removed from the inlet
- the pump isolating plate would be secured with two transit bolts.

5.2 Preparation

Before unpacking the DSMS system, check for signs of shipping damage. If the packaging is damaged, notify the carrier immediately.

Check all the box contents against the packing list. If any parts are missing, notify the carrier and Hiden Analytical Limited immediately.

Having inspected all the parts, place the main support frame close to the area in which it is to operate but with room to work around the frame.

5.3 System assembly

Refer to Figure 1.

A basic tool kit is required, including 10 mm and 13 mm spanners, metric hex keys, screwdrivers, etc.

Refer to the appropriate manufacturer's manuals for the operating instructions for the various items not of Hiden Analytical Limited manufacture.

For benchtop DSMS systems a suitable work bench capable of supporting the weight of the DSMS must be used. Ensure there is sufficient space to work around the system during installation. The DSMS must not be moved once it is started.



For cart mounted DSMS systems ensure there is sufficient space to work around the DSMS during installation. The DSMS must not be moved while the pumps are running.

The scroll pump is mounted onto a sub-plate secured to the lower trolley shelf by two M8 transit screws. As part of the installation procedure:

1.	Move the trolley in to its operational location.	Ensure it is not powered up and the pumps are therefore not running.
2.	Lock the wheels.	Press the tab downwards until it locks into position.
3.	Remove the two M8x25 cap head screws that secure the scroll pump sub-plate to the trolley lower shelf.	There is one mid-way on each side of the plate.
		Use the 6mm hex key supplied with the system.
		Retain the screws for future use.
4.	Lower the four jacking legs by turning the rose knobs in a clockwise direction.	Once the jacking legs are in contact with the floor the sub-plate will rise above the trolley shelf.
		Lower the legs as far as they will go.

If the system is moved reverse the above procedure to lock the scroll pump in place.

To install the DSMS system:

- 1. Remove any protective caps, cable ties, etc. that have been fitted for shipping purposes.
- 2. Connect the RF Cable **HEAD** connector to the RF Head.
- 3. Connect the SEM detector supply cable (reference HA-072-031) to the bayonet connector on the probe mounting flange.
- 4. Connect the RF Head to the mass spectrometer probe.

Protective plastic caps are fitted to the backing line port, scroll pump and stainless steel bellows.

The 37 way D-type to 37 way D-type cable, reference HA-077-041, connects the RF Head to the RC Interface **RF HEAD EL02** connector.

This cable is secured with plastic ties for shipping. Cut the ties.

This cable is secured with plastic ties for shipping. Cut the ties.

The RF Head mates directly with the vacuum feedthrough via the 12-way feedthrough connector.

The centre pin on this connector is spring loaded and engages with the centre pin on the vacuum feedthrough. Ensure this is making a good connection.

The 12-way feedthrough connector locates using a key-way machined in the connector surround. Fit the RF Head to the vacuum feedthrough so that the key-way engages.

The screws may be M4 grub screws (requiring a 2mm Allen key) or thumbscrews.

Use the supplied seals.

For cart systems; the scroll pump is fitted in the cart and connected to the turbo pump by the stainless steel bellows. Therefore, steps 6. to 9. are not required.

Use the supplied seals.

5. Tighten the clamping screws that lock the RF Head in place on the Probe.

- Connect the DN-25-KF stainless steel bellows to the DN-25-KF Tee connected to the turbo pump outlet port.
- 7. Connect the other end of the stainless steel bellows to the scroll pump inlet port.

8. Connect the scroll pump control cable.

- 9. Connect the scroll pump mains inlet to the DSMS mains distribution block.
- 10. Using one of the communications cables provided, link the RC Interface to the PC comms. port (or network hub).

D-type to D-type cable, reference HA-057-592, connects the VCU **E1** connector to the scroll pump logic interface connector.

This cable is secured with plastic ties for shipping. Cut the ties.

Use the IEC to IEC connector lead supplied.

Communications may be via RS232, USB or 10/100Base-T, but only one type can be used at one time.

All three types of communications cable are supplied with the system.

Refer to the Hiden Software Suite Installation Guide.

the RC Interface unit.

Install the drivers before switching on

11. If the USB port is being used to communicate with the PC install the USB drivers from the Hiden Software disc.

- 12. If the system is to be vented by gas other than ambient air, connect the gas supply to the vent valve.
- 13. If the system is fitted with the gas purge option connect the purge gas supply to the gas sealing valve fitted to the turbo pump.

14. If the scroll pump is fitted with the gas ballast option connect a source of regulated dry inert gas such a nitrogen.

A supply of dry gas connected to the vent valve at system shutdown will reduce the water vapour level and hence reduce the pump-down time for the next operation. The gas pressure

should be in accordance with the vent valve manufacturer's instructions, typically 0.3barg.

The fitting will typically be a 1/4 inch compression fitting.

Connect a regulated supply of dry inert gas such as nitrogen. Set the regulator pressure to 0.3barg (5psig).

The valve is factory set to allow a flow of approximately 50ml/min.

The fitting will typically be a 1/4 inch compression fitting.

Set the regulator pressure to 0.3barg (5psig).

The flow meter valve is typically set to 0.5 to 1 l/min.

15. If the system incorporates rotary pumps, add oil to the pumps.

Refer to the appropriate manufacturer's manuals.

Suitable oil is supplied with the system. A funnel is supplied with the system.



CAUTION

The mains power plug and socket is the primary disconnect device for the DSMS; it must be easily identifiable and accessible by the operator.

- 15. When the rest of the mechanical and electrical installation is complete, connect the IEC320 cordset between the system power connector and a suitable mains power socket.
- 16. Install the MASsoft application by following the instructions in the Software Suite Installation Guide.

Check that the DSMS is suitable for the local supply. The voltage rating is given on the serial number label.

The mains power socket must be easily accessible by the operator.

Refer to the Hiden Software Suite Installation Guide.

6 Operation

3.

4.

6.1 System start-up from atmospheric pressure

To start the DSMS system following assembly or shut down:

1. Ensure the inlet bypass isolation valve is open.

Move the Vacuum Control Unit

Pumps switch to the **ON** position.

When the pressure indicated by the

total pressure gauge is less than 9x10⁻⁵

mbar switch on the mass spectrometer

2. Power up the system.

Check the local supply is suitable for the system.

Connect the system's mains distribution block to the mains supply and move the mains distribution power switch to the on position.

The switch will illuminate providing mains power is available.

The Vacuum Control Unit display will illuminate and show **Pumps Off** and **SP1 off**.

The scroll pump or backing and bypass rotary pumps will start and the turbo pump will start to accelerate.

The turbo pump vent valve will be energised and will close.

Turbo pump speed is indicated towards the bottom of the display.

Before the turbo pump reaches full speed the total pressure gauge will switch on automatically.

The value of 9×10^{-5} mbar may vary depending on system type. Systems with mass spectrometers fitted with Ion Counting detectors will typically have the trip level set to 5×10^{-6} mbar. Refer to the Contents Sheet supplied with the system.

The VCU display will show **SP1 OK** once the pressure has fallen below the setpoint pressure.

5. Start MASsoft.

RC Interface unit.

6.2 Sampling using the inlet system



Note:

The total pressure gauge should be used in preference to the mass spectrometer for total pressure measurement.

To sample:

1.	Follow the start-up procedure.	Refer to Section 6.1.
2.	Ensure the pressure at the sampling point is in the correct region for the inlet system being used.	Refer to the capillary inlet manual.
3.	Ensure the vacuum system is operating and that the pressure at the mass spectrometer is below 1×10^{-6} mbar.	
4.	Ensure the bypass pump is operating.	For scroll pumped systems bypass pumping is via the backing line.
5.	Ensure the bypass isolation/control valve is fully open.	
6.	To operate the inlet system hot, apply power to the heaters.	Allow ¹ / ₂ hour (minimum) for the inlet to heat up.
7.	Open the sample isolation valve.	



CAUTION

If using the mass spectrometer multiplier detector, the maximum pressure must not exceed $5x10^{-6}$ mbar.

8. Monitor the sample pressure as displayed on the mass spectrometer.



9. If necessary, set the pressure at the mass spectrometer to a maximum of 1×10^{-5} mbar for normal use, by adjusting the bypass pumping using the bypass isolation/control valve.

Slowly turn the valve clockwise to increase the pressure at the mass spectrometer.

To end sampling:

- 1. If used, switch off the heater.
- 2. Close the sample isolation valve.
- 3. Wait for a minimum of 1 minute, to allow evacuation of the inlet.
- 4. Close the bypass/isolation valve.

6.3 Cryo-shield operation

Using the cryo-shield will reduce background levels by enhancing pumping in the ion source region. This allows detection of lower partial pressures from the sample, thus increasing the detection capability.



- 1. Ensure that the UHV housing is being pumped by the turbo pump and is under vacuum.
- 2. Add liquid nitrogen to the cryo-shield chamber through the vertical filling port.
- 3. Add more liquid nitrogen as necessary.

The liquid nitrogen will boil vigorously at first; keep adding liquid nitrogen until the boiling reduces.

This normally will be every few hours.

6.4 System shut down

The DSMS will need to be shut down for maintenance or if it is to be moved. The user may wish to shut down the system if it is not to be used for some time.



CAUTION

If a cryo-shield is being used, ensure that cryo-shield chamber is empty before the system is shut down; this avoids water condensing in the UHV housing.

To shut down the DSMS:

1. Refer to the MASsoft manual. Switch off the mass spectrometer filament(s) and shut down MASsoft. 2. Switch off the RC Interface unit. If appropriate, switch off any heaters. Allow a minimum of 10 minutes for 3. the filaments to cool. Allow an additional 20 minutes for the heaters to cool. 4. Close the sample isolation valve and the bypass line isolation valve as appropriate. 5. Move the Vacuum Control Unit The turbo pump is switched off and **Pumps** switch to the **OFF** position. starts to decelerate. After a delay the vent valve opens and the backing and bypass pumps are switched off. The total pressure gauge will be switch off automatically. Wait for the system to vent.

5. Move the rocker switch on the mains distribution block to the off position.

Power to the Vacuum Control Unit will be switched off.

7 Troubleshooting



CAUTION

The procedures described in this section must be performed by suitably qualified personnel with experience of high vacuum systems.

The procedures described in this section are for troubleshooting only; they are not required for normal operation.

7.1 Fault finding guide

This section aims to help the user identify and resolve problems which may prevent the DSMS from operating correctly.

7.1.1 Vacuum Control Unit

As soon as the DSMS system is powered on the Vacuum Control Unit display should illuminate.

Symptom	Fault	Action
Display fails to illuminate at	No mains supply to DSMS.	Check the mains supply to the DSMS mains distribution block.
power on.		Check the distribution block switch is in the ON position. The switch should illuminate when switched on with mains power present.
	VCU mains cable disconnected.	Check the mains cable to the VCU is fully connected to both the mains distribution block and the VCU rear panel connector.
	VCU unit has failed.	The VCU unit will need to be replaced.
		Contact Hiden Analytical for advice.

7.1.2 Scroll pump

In DSMS systems the scroll pump acts as both the turbo backing pump and the inlet bypass pump. It should start as soon as the system is started by moving the VCU **Pumps** switch to the **ON** position. It should be obvious when the pump is running due to the sound and vibration.

Symptom	Fault	Action
Scroll pump does not run when system started.	No mains supply.	Check the mains cable to the pump is fully connected to both the pump mains inlet and the DSMS mains distribution block.
		Check mains power to the pump is being provide by the DSMS mains distribution block.
		Temporarily try powering the scroll pump directly from a mains power outlet and see if it then runs.
	No control signal.	Check that the control cable connecting the VCU to the scroll pump 15 way D-type plug is connected and both connectors are correctly seated.
	Pump fault.	Contact Hiden Analytical for advice, a replacement pump may be needed.
Turbo pump fails to reach full speed	Worn tip seals.	Scroll pump requires servicing and tip seals replacing.
and/or scroll pump		Test the pump, see Section 8.6.1.
than normal noise.		Contact Hiden Analytical for advice.

7.1.3 Rotary pump

Note

Some DSMS systems do not have a separate bypass rotary pump. Only follow this part if the DSMS has two rotary pumps.

Start the bypass rotary pump. Some rotary pumps have a switch on the top of the pump others just plug into the mains.

The bypass rotary pump is powered by the PSTU from the BACKING PUMP outlet via a Y-cable.

Check the pump is running by listening for the sound or feeling the vibration of the pump body.

Symptom	Fault	Action
The rotary pump is not running.	It is not switched on.	Check the switch on the top of the pump is in the on position.
	There is no mains supply to the pump.	Check the pump is plugged in to the DSMS mains distribution block or Y-cable.
		Check the mains supply is present.
		Check the mains supply is correct for the pump. There will be a rating plate on the pump body.
		Check the mains fuses.
		Check the circuit breaker on the DSMS mains distribution block.
		Temporarily try powering the pump directly from a mains power outlet and see if it then runs.
	The bypass rotary pump is faulty.	The bypass rotary pump may need to be replaced. Please contact Hiden Analytical for advice.
Rotary pump oil on the floor or base of system.	Spillage from an oil change.	Clean up and check if more oil appears.
	Rotary pump shaft seal has failed.	Check the pump for signs of oil dripping from the oil sump/motor housing seal.
		Follow the manufacturers instructions to replace the seal. Contact Hiden Analytical for advice.

Symptom	Fault	Action
There is a large air leak on the system.	There is a leak on one of the vacuum seals.	Check all the bolts are tight on the CF fittings.
		Check the clamps are tight on the KF fittings.
	No backing pump.	Check the backing pump is running.
	Worn scroll pump seal.	Worn tip seals generally cause louder than normal running noise. See Section 7.1.2.
	The air admittance valve is leaking.	Check whether air is being sucked in through the automatic vent valve.
		Check that 24V DC is being supplied to the valve by the turbo pump. If it is not there could be a fault with the cable.
		Try replacing the automatic vent valve with a manual vent vale (one is supplied with the system).
		The automatic vent valve will need to be replaced if it is leaking.

7.1.4 Turbo pump fails to reach full speed

Also please refer to the manufacturers documentation of fault finding information.

7.1.5 Total pressure gauge

The DSMS mass spectrometer vacuum chamber will be fitted with either an Edwards AIM-XL cold cathode gauge or an Edwards AIGX-S active ion gauge. The type of gauge fitted is recorded in the configuration section of the Contents Sheet.

The gauge head is connected to the G1 connector on the VCU rear panel. The pressure reading is shown on the VCU display as G1. In addition to displaying the chamber total pressure this gauge provides the mass spectrometer over-pressure trip signal to protect the filaments and detector.

When the system is started and before the turbo pump reaches full speed the total pressure gauge will be switched on and the pressure will be displayed. This pressure will typically be in the 10⁻⁵ mbar region and will be falling rapidly.

Please refer to the manufacturers documentation in addition to the following sections.

Symptom	Fault	Action
VCU display is blank where pressure reading should be	Gauge head not connected.	Check the cable is plugged into the gauge head and the VCU unit.
shown.		rry an alternative cable.
Pressure displayed is lower than normal.	Capillary inlet is blocked.	Remove any adapters and sample ambient air and monitor the pressure.
		A mass spectrum with an absence of nitrogen and oxygen as the main peaks when sampling air indicates a blockage.
Pressure displayed is higher than normal.	Leak on the system.	Check all the vacuum seals particularly any that have recently been disturbed.
	Increase in gas sample pressure.	Disconnect the capillary from the gas sample source and sample ambient air, look for a change in pressure.
	Worn scroll pump tip seals.	Scroll pump requires servicing and tip seals replacing.
		Test the pump, see Section 8.6.1.
		Contact Hiden Analytical for advice.

7.1.6 Edwards AIGX-S ion gauge

The gauge has a tri-coloured LED which gives some status information.

Off	No power to gauge or a fault.
Amber	Power on, gauge disabled.
Green	Emission on.
Red	Over-pressure trip.

Refer to the manufacturers documentation for further information.

7.1.6.1 Edwards AIM-XL cold cathode gauge

Symptom	Fault	Action
VCU displays Striking	The cold cathode gauge has not started.	Try un-plugging and plugging in the gauge head cable.
	The cold cathode gauge requires servicing.	Please contact Hiden Analytical for advice.
VCU displays pressure as 8.5x10 ⁻³ .	This is the maximum pressure reading and could mean the gauge is contaminated with metal deposits.	The gauge requires servicing please contact Hiden Analytical for advice.

Refer to the manufacturers documentation for further information.

7.1.7 Mass spectrometer

Providing the pressure indicated by the total pressure gauge controller is below 9x10⁻⁵ mbar the mass spectrometer can be switched on and used to leak check the DSMS system.

Move the switch on the RC Interface to the ON position. The switch should be illuminated and the RC Interface should emit a short beep.

Symptom	Fault	Action
The RC Interface switch does not illuminate when in the ON position.	Mains cable not plugged in.	Check the mains cable is plugged into the DSMS mains distribution board and RC Interface unit.
	No mains supply to the DSMS.	Check the mains supply is present and the correct voltage for the RC Interface.
		Check the mains fuse.
		Check the circuit breaker on the DSMS mains distribution block.
		Try connecting the RC Interface to an external mains supply.
	The RC Interface is faulty.	Please contact Hiden Analytical for further assistance.

There is no beep sound made by the RC Interface.	The RC Interface has a fault.	Please contact Hiden Analytical for further assistance.
The RC Interface emits a long and continuous beep to indicate that the RC Interface is detecting an external trip.	The trip signal from the total pressure gauge control is not connected.	Check the 15 way D-Type connector is plugged into the TRIPS connector on the rear panel of the RC Interface.
	The over-pressure trip signal from the mass spectrometer RF Head is not connected.	Check the 37 way D-Type connector is plugged into the RF HEAD connector on the rear panel of the RC Interface.
	The chamber pressure is above the total pressure gauge set point.	A trip condition is indicated by the VCU SP1 showing as blank rather than OK .
The total pressure gauge controller indicates a trip condition.	The chamber pressure is above the total gauge set point.	If the total pressure gauge controller indicates a pressure below $9x10^{-5}$ mbar the total pressure gauge set point may be incorrect.
		The set point will usually be set to $9x10^{-5}$ mbar for analogue instruments and $5x10^{-6}$ for pulse counting instruments.
		Refer to the Contents Sheet for the correct setting.
	The chamber pressure is above the total pressure gauge set point and the set point is correct.	The chamber pressure is in the region between 1×10^{-4} mbar and the set point (9×10^{-5} mbar). Fit the linking plug. Refer to the text below and the total pressure gauge manual.

If the total pressure gauge switches on, the pressure must be below its internal trip level, set by Hiden to 1×10^{-4} mbar.

The total pressure gauge set point is set by Hiden to $9x10^{-5}$ mbar ($5x10^{-6}$ mbar for RC-PIC instruments) and the relay output is used to trip the RC Interface, thereby protecting the filaments and the electron multiplier. Neither the filaments or the multiplier can be switched on if the external trip (provided by the total pressure gauge) is active.

In the small pressure region of $8x10^{-5}$ mbar to $1x10^{-4}$ mbar with care the mass spectrometer may be used to leak check the system to locate any air leaks which will be the most likely cause of high mass spectrometer chamber pressure.

The external trip may be disabled by un-plugging the cable from the TRIPS connector on the rear panel of the RC Interface and fitting a linking plug. Alternatively, the total pressure gauge set point may be adjusted, refer to the total pressure gauge documentation for a description of adjusting the set point.

Hiden Analytical supplies a linking plug. Alternatively, refer to the over-pressure protection section in the mass spectrometer manual.

Using the SEM at high pressure will dramatically reduce its useful operating life.

7.1.8 Emission Failure

When a MASsoft recipe is run (when leak checking for instance) a filament is switched on and the correct emission current (flow of electrons from the filament to the source cage) is established. When operating correctly the Filament 1 and/or Filament 2, Emission and Run LEDs on the RC Interface front panel should all be illuminated.

Symptom	Fault	Action
The Emission LED is flashing.	There is an open circuit on the filament circuit.	Check the RF cable is properly connected to both the RF Head and RC Interface.
		Check the RF Head is connected to the probe and the connector is fully pushed on to the probe.
		Check the filaments are not open circuit. Refer to the Trouble-shooting chapter in the mass spectrometer manual to check the filaments. If the filaments are open circuit they will need to be changed, refer to 8.2.
Neither Filament LED is illuminated.	The MASsoft recipe is not switching the filament on.	In the MASsoft recipe Global RGA box check that a filament is selected.

7.2 Leak checking

To operate correctly the only gas entering the DSMS and being detected by the mass spectrometer probe must be through the DSMS inlet valve assembly. A leak on the mass spectrometer vacuum chamber would, at best, give an air background spectrum that would limit the instrument's performance. At worst a large leak could prevent the system from operating at all.

If there is a leak one or more of the following symptoms may be observed:

- 1. The turbo pump fails to reach full speed.
- 2. The total pressure gauge gives a higher reading than expected.
- 3. A persistent air background spectrum is observed.

An air spectrum would have mass 28 and mass 32 peaks in the ratio 5 to 1.

The mass spectrometer's ability to detect gas species is used to find any leaks in the DSMS vacuum system. The mass spectrometer is configured to monitor a probe gas (helium is the most common leak checking gas) which is carefully sprayed over the vacuum system. Any signal detected by the mass spectrometer will be due to the leak check gas entering the system through the leak.

A helium cylinder fitted with a regulator and a length of hose is required to leak check the system.

Another gas such as argon can be used if helium is not available. Any grade of gas may be used.

To leak check:

- 1. Start MASsoft.
- 2. Select Leak Detect from the Gallery.
- 3. Set the Mass: to 4 (if using helium) or an appropriate mass for the leak checking gas (mass 40 for argon). Refer to the Leak Detect scan section in the MASsoft User's Manual for further details regarding the configuration of MASsoft.
- 4. Adjust the helium cylinder regulator to give a gentle flow of gas. Spraying large amounts of helium over the vacuum system will make it difficult to precisely locate any leaks.
- 5. Spray helium over the vacuum chamber paying particular attention to the vacuum seals as these are the most likely source of a leak. Start at the top and work down the system.

8 Maintenance



Procedures described in this Section are for system maintenance only; they are not required for normal operation.

8.1 Maintaining System safety

The following points should be checked every two years in order to maintain safe operation of the equipment:

	Item	Test	Pass condition
1.	Mains lead(s) and connectors.	a) Visual inspection.	No damage to cable or connectors.
		b) Mains plug.	Correct fuse fitted.
		c) Attempt to pull the cable from the IEC connector.	No movement.

Note:

If the lead or one of its connectors is damaged it must be discarded and replaced by a suitably approved cordset with a moulded IEC 320 socket and mains plug; the cable must contain a double layer of insulation and must have an adequate current capacity for the application.

2.	Mains on/off switch	Visual inspection.	Correct operation, no damage.
3.	Conducting cases and metalwork.	a) Visual inspection.	No damage.

	b) Using an earth tester which will check resistance and pass a current of at least twice the fuse rating, check between the mains plug earth, and the DSMS metalwork.	Earth resistance must be less than 0.1 ohm.
	c) High voltage insulation 500 V a.c. minimum test, check between the mains plug live and earth.	No fault indicated after 5 seconds.
Accessible fuse holders.	Visual inspection.	No damage.

The above information has been derived from the United Kingdom Health and Safety Executive Guidance Note PM32.

8.2 Filament renewal

4.

The mass spectrometer ionisation source filament lifetime depends on operating conditions and may be up to several years. Two filaments are fitted to the mass spectrometer ionisation source so that when one fails the other filament may be used. Follow the instructions below to renew the filaments; also refer to the instructions in the mass spectrometer manual.

1.	Shut down the system.	Follow the procedure described in Section 6.4.
2.	Remove the RF Head and secondary electron multiplier detector cable (if fitted).	
3.	Make a note of the mass spectrometer probe feedthrough flange orientation.	The probe will need to be fitted in the same orientation. The radial M3 screw and metalised feedthrough pin may be used for reference.
4.	Remove the mass spectrometer probe.	Remove the six or eight M6 or M8 bolts using 10mm or 13mm spanners.
5.	Follow the filament renewal procedure detailed in the mass spectrometer manual.	

6.	Refit the mass spectrometer probe onto the vacuum chamber using a new copper gasket seal.	Refer to the "Assembling Conflat-type flange joints" section in the mass spectrometer manual.
7.	Refit the RF Head.	
8.	Re-start the DSMS system.	Refer to Section 6.1
9.	Leak check the DSMS system.	Refer to Section 7.2.

8.3 Inlet assembly

8.3.1 Removal



The cover must be removed to carry out maintenance operations such as replacing the molecular leak as described in this section. When removing the cover and carrying out work on internal parts of the unit a face mask and protective gloves must be worn.

The Heated Molecular Leak Assembly incorporates ceramic paper insulation which may pose a long term health risk due to sub six micron filament size fibres in the material.

For further information and Safety Data Sheets please contact Hiden Analytical Limited.

Refer to Figure 5.

To remove the inlet assembly from the mass spectrometer vacuum housing:

1. Remove the knobs from the bypass isolation/control valve and the sample isolation valve.

Each knob is secured by two grub screws.

- 2. Remove the M3 cap head screws (4-off) retaining the outer cover.
- 3. Remove the outer cover.
- 4. Unscrew the tapped spacers (4-off) retaining the inner cover.
- 5. Remove the inner cover.



Figure 5 Heated Molecular Inlet Assembly interior

- 6. Loosen the heater clamped around the mass spectrometer vacuum housing mounting flange and slide the heater off the flange joint.
- 7. Remove the mounting flange fixing bolts.
- 8. Remove the heated inlet assembly from the mounting flange.

8.3.2 Fitting the inlet

Refer to Figure 5.

To fit the inlet assembly to the mass spectrometer vacuum housing:

 Mount the Heated Molecular Inlet Assembly onto the mass spectrometer vacuum housing mounting flange.
 A new copper gasket must be used in the flange. The copper gasket is used as the sealing medium, the seal being formed when the flanges builty adverse

formed when the flange knife-edges embed in the copper as the fixing screws are tightened.

It is essential that the flanges are tightened uniformly:

First tighten diametrically opposite screws finger-tight only, then tighten the remaining screws finger-tight only.

Each screw is then tightened progressively, a little at a time, until all screws exhibit strong resistance. It is not necessary to achieve flange-to-flange contact. The normal tightening torque required to achieve a good seal is 7 to 9 Nm; over-tightening can cause later problems with gasket removal.

- 2. Refit the heater around the mounting flange.
- 3. Refit the inner cover.
- 4. Refit and tighten the tapped spacers (4-off) to secure the inner cover.
- 5. Refit the outer cover.

Do not fit the screws at this stage.

- 6. Refit the knobs to the bypass isolation/control valve and the sample isolation valve and tighten their grub screws.
- 7. Refit and tighten the four M3 cap head screws to secure the outer cover.

The outer cover will need to be pushed away from the knobs to gain access to the grub screws.



WARNING

The Heated Molecular Inlet Assembly must be earthed.

8. Use a suitable earth tester to check that the Heated Molecular Inlet Assembly is connected to the DSMS system earth. See also Section 8.1.

8.3.3 Replacing the molecular leak

To renew the molecular leak/flange assembly:

- 1. Follow the steps detailed in Section 8.3.1.
- 2. Undo the Swagelok nut connecting the inlet fittings to the molecular leak assembly.
- 3. Remove the enclosure fixing screws from the flange.
- 4. Remove the molecular leak/flange assembly.
- 5. Fit the new molecular leak/flange assembly.
- 6. Fit the enclosure fixing screws to the flange.
- 7. Tighten the Swagelok nut connecting the inlet fittings to the molecular leak assembly.

8. Follow the steps detailed in Section 8.3.2. to refit the inlet.

8.4 Bake-out

汕	CAUTION
	1. The RF head and all cables must be removed from the gauge head prior to bake-out.
	2. The system must be under vacuum during bake-out.
	3. The maximum bake-out temperature for the UHV housing and gauge head is 250 °C.
	4. The maximum bake-out temperature for the bypass inlet system will depend on its construction and seal materials; Vespel/metal-sealed systems may be baked to 200 °C, PTFE/Kel F-sealed systems may be baked to 80 °C.
	5. Wrap the feed-through flange in aluminium foil prior to baking; this avoids damage to the feed-through due to sudden temperature changes.
	6. The UHV housing temperature must not be increased or decreased at more than 2 °C/min.
	7. The cold cathode gauge head must be partially dismantled before bake-out; refer to the manufacturer's instruction manual.
	8. The turbo pump must not be subjected to excessive temperatures; refer to the manufacturer's instruction manual.
	9. A minimum of two hours must be allowed for cooling after bake-out, before venting the system or reconnecting the electronics to the system.

Note:

The bypass inlet system fitted to standard DSMS systems uses all metal valves and Vespel ferrules; unheated sampling lines may include PTFE ferrules.

The relevant parts of the system may be cleaned by heating under vacuum. Heating tapes may be wrapped around the parts, or they may be enclosed in a purpose-built oven.

8.5 Rotary pumps

Renew the rotary pump oil according to the instructions in the pump manufacturer's operating manual.

When pumping corrosive species it is recommended that the frequency of oil changes be increased above the manufacturer's standard recommendations. The increased frequency will be determined by the user, based on the nature of the pumped species and any effect on the pump fluids.

Standard DSMS systems use mineral oil, corrosive systems use a silicone oil. Oil must be replaced with the same type. Refer to the Contents Sheet for the oil type.



Material Safety Data Sheets for both mineral oil and silicone oil are available from Hiden Analytical and the pump manufacturer.



Normally, the user will be aware of any potentially dangerous contaminants in the pump oil due to sampling hazardous gases.

Any necessary safety precautions must be taken.

8.5.1 Testing the pump

Rotary pumps can develop oils leaks over time. Usually, oil will seep from the joint between the casing and the motor housing due to a worn shaft seal. Consult the manufacturer's documentation for further information.

To test if a rotary pump is working seal the inlet using a total pressure gauge. A Pirani gauge or a baratron are suitable types of gauge. Switch on the pump. The gauge should read less than 1 mbar within 1 minute from starting the pump. If it does not the pump may need servicing.

8.6 Scroll pumps

The pump tip seals will wear in time and eventually need replacing. Refer to the manufacturers instructions to do this.

8.6.1 Testing a scroll pump

To test if a scroll pump is working seal the inlet using a total pressure gauge. A Pirani gauge or a baratron are suitable types of gauge. Switch on the pump. The gauge should read less than 50 mbar within one minute from starting the pump. If it does not the pump may need servicing.

8.7 Diaphragm pumps

A small number of DSMS systems use one or two diaphragm pumps. Renew pump diaphragms and flap valves in accordance with the manufacturer's instructions.

8.7.1 Testing a diaphragm pump

To test if a diaphragm pump is working seal the inlet using a total pressure gauge. A Pirani gauge or a baratron are suitable types of gauge. Switch on the pump. The gauge should read less than 10 mbar within 30 seconds from starting the pump. If it does not the pump may need servicing.

8.8 Turbo pumps

The turbo pump bearings will typically require replacement every two to three years. This task must be performed by the pump manufacturer; this can be arranged through Hiden Analytical if preferred.

8.8.1 Testing the turbo pump

Modern turbomolecular pumps are far more reliable than the older models. The three most likely causes of failure are:

- Faulty drive electronics
- Worn bearings
- Foreign object in the turbine stack jamming the rotor

Refer to the manufacturers documentation.

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