# Models CIS100, CIS200, and CIS300 Closed Ion Source Quadrupole Gas Analyzer



STANFORD RESEARCH SYSTEMS

Revision 1.1 (July, 00)

#### Certification

Stanford Research Systems certifies that this product met its published specifications at the time of shipment.

#### Warranty

This Stanford Research Systems product is warranted against defects in materials and workmanship for a period of one (1) year from the date of shipment.

#### Service

For warranty service or repair, this product must be returned to a Stanford Research Systems authorized service facility. Contact Stanford Research Systems or an authorized representative before returning this product for repair. All users returning a CIS Analyzer back to the factory for repair and/or service must submit a correctly completed "Declaration of Contamination of Vacuum Equipment" form along with the instrument. The SRS personnel carrying out the repair and service of vacuum equipment must be informed of the condition of the components before any work is done on the parts. A copy of the form is attached to the end of this manual.

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Printed in U.S.A.

# **Safety and Preparation For Use**

#### WARNING!



Dangerous voltages, capable of causing injury or death, are present in this instrument. Use extreme caution whenever the instrument cover is removed. Do not remove the cover while the unit is plugged into a live outlet.

Line Cord

The CIS Analyzer's built-in power module option (Opt02) has a detachable, three-wire power cord for connection to the power source and to a protective ground. The exposed metal parts of the instrument are connected to the outlet ground to protect against electrical shock. Always use an outlet which has a properly connected protective ground.

Service



Do not attempt to service or adjust this instrument unless another person, capable of providing first aid or resuscitation, is present.



Do not install substitute parts or perform any unauthorized modifications to this instrument. Contact the factory for instructions on how to return the instrument for authorized service and adjustment.

Fan

The fans in the CIS Analyzer are required to maintain proper operation. Do not block the vents in the chassis or the unit may not operate properly.

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# **Specifications**

## Operational

Mass Range:	
CIS100	1 to 100 amu
CIS200	1 to 200 amu
CIS300	1 to 300 amu
Ion Source	High Conductance Closed Ion Source (CIS)
Mass filter type	Quadrupole (Cylindrical rods, rod diameter: 0.25", rod length: 4.5")
Detector type	Faraday cup (FC) – standard
	Electron multiplier (CDEM) – optional
<b>Resolution</b> (per AVS standard 2.3).	Better than 0.5 amu @ 10% peak height Adjustable to constant peak width over the entire mass range
Sensitivity (A/Torr):	Measured for $N_2$ @ 28 amu with 1 amu full peak width @ 10% height.
RGA Mode <sup>1</sup>	10 <sup>-5</sup> (FC) <10 (CDEM). User adjustable throughout high voltage range.
CIS Mode <sup>1</sup>	10 <sup>-6</sup> (FC)
	<1 (CDEM). User adjustable throughout high voltage range.
Minimum detectable partial pressure:	Measured as the lowest detectable signal at mass 28 with 1mTorr of Argon present.
RGA Mode <sup>1</sup>	10 <sup>-9</sup> Torr (FC)
	10 <sup>-11</sup> Torr (CDEM)
CIS Mode <sup>1</sup>	10 <sup>-8</sup> Torr (FC)
	10 <sup>-10</sup> Torr (CDEM)
Maximum operating pressure	10 <sup>-2</sup> Torr to UHV (FC) 2 <sup>-10<sup>-3</sup></sup> Torr to UHV (CDEM)
Max. bakeout temperature (without ECU)	350°C (FC) 300°C (CDEM)
Recommended bakeout temperature	200°C

1. <u>RGA Mode</u>: 70eV electron energy, 0.5mA electron emission current, 8 eV ion energy, optimized extraction voltage. <u>CIS Mode</u>: 70eV electron energy, 0.05mA electron emission current, 8 eV ion energy, optimized extraction voltage

## Ionizer

Design	High Conductance Closed Ion Source	
Conductance (nominal)	$0.4 \text{ L s}^{-1}$ .	
Material	Gold coated, 304 stainless steel.	
Operation	Electron impact ionization.	
Filament	Tungsten (standard) or Thoriated Iridium (optional) with firmware protection. Field replaceable.	
Electron energy	15 to 105 eV, programmable.	
Ion energy	4 or 8 eV, programmable.	
Extraction voltage	0 to 150 V, programmable.	
Electron emission current	0 to 1.0 mA, programmable.	

## General

Probe dimension8.75" from feedthru flange face to top of CIS Port	
Probe mounting flange	2.75" CF.
Pumping system mounting flange	2.75" CF.
Pumping system	An oil-free turbopump based system with a minimum pumping speed of 40 L s-1, and a base pressure better than $2*10^{-9}$ Torr, is recommended to meet the instrument's specifications.
ECU dimensions	9.1" x 4.1" x 3.1". Easily separated from the probe for bakeout.
LED indicators	Power (ON/OFF), Filament (ON/OFF), Degas (ON/OFF), Elec. Mult (ON/OFF), RS-232 (Busy signal), Error, Leak, and Burnt Filament.
Computer interface	RS-232C, 28,800 Baud with high level command set and fully enabled RTS/CTS handshaking.
Software	Windows OS based application. Requires 486 66MHz PC or faster.
Power Requirement	24 VDC @ 2.5 Amps. Male DB9 connector. Optional 110/120/220/240 VAC (50/60 Hz) built-in power module.
Weight	7 lbs.
Warranty	One year parts and labor on materials and workmanship.

# **CIS Command List**

#### Initialization

Name	Description	Parameters	Echo
ID	Identification Query	?	ID String
IN	Initialization	0,1,2	STATUS Byte

#### **Ionizer Control**

Name	Description	Parameters	Echo
EE	Electron Energy	15-105, *, ?	STATUS Byte or query
FL	Electron Emission Current	0-1.00, *, ?	response STATUS Byte or query
IE	Ion Energy	0, 1, *, ?	response STATUS Byte or query
VF	Extraction Plate Voltage	0-150, *, ?	response STATUS Byte or query

#### **Detection Control**

Name	Description	Parameters	Echo
CA	Calibrate All	none	STATUS Byte
CL	Calibrate Electrometer	none	STATUS Byte
HV	CDEM's High Voltage	0-2490, *,?	STATUS Byte or query
			response
MO	Multiplier Option	?	CDEM option
NF	Noise Floor	0-7,?	Query response

#### **Scan and Measurement Control**

Name	Description	Parameters	Echo
AP	Analog Scan Points	?	Query response
HP	Histogram Scan Points	?	Query Response
HS	Histogram Scan Trigger	0-255, *, none	Ion Currents
MF	Final Mass	1-M_MAX, *, ?	Query response
MI	Initial Mass	1-M_MAX, *, ?	Query response
MR	Single mass measurement	0, M_MAX	Ion Current
SA	Steps per amu	10-25,*,?	Query response
SC	Analog Scan Trigger	0-255,*, none	Ion Currents

#### **Parameter Storage**

Name	Description
MG	CDEM gain storage
MV	CDEM Bias Voltage storage
XA	Parameter Address
XV	Parameter Value

#### Parameters

0.0000-2000.0000,?	Que
0-2490, ?	Que
0-10,?	Que
0 - +/-999999.9999, ?	Que

# response

Echo
STATUS Byte
STATUS Byte
STATUS Byte or query
response
CDEM option
Query response

#### Echo

ery Response ery Response ery response ery response

Mass filter			
Name	Description	Parameters	Echo
ML	Mass Lock	0.0000-M_MAX	none
Tuning			
Name	Description	Parameters	Echo
CE	Calibration Enable Query	?	Query response
DI	DI parameter	0-255, *, ?	Query response
	(Peak Width Tuning)		
DS	DS parameter	-2.5500-2.5500, *, ?	Query response
	(Peak Width Tuning)		- • •
RI	RF_Driver @0 amu	-86.0000-86.0000, *,	Query response
	(Peak Position Tuning)	?,none	
RS	RF_Driver @ 128 amu	600.0000-1600.0000, *,	Query response
	(Peak Position Tuning)	?,none	
Error Repo	-		
Name	Description	Parameters	Echo
ER	STATUS Byte Query	?	Query response
EP	PS_ERR Byte Query	?	Query response
ED	DET_ERR Byte Query	?	Query response
EQ	QMF_ERR Byte Query	?	Query response
EM	CEM_ERR Byte Query	?	Query response
EF	FIL_ERR Byte Query	?	Query response
EC	RS232_ERR Byte Query	?	Query response

**Note:** M\_MAX= 100 for CIS100, 200 for CIS200 and 300 for CIS300.

# Chapter 1 Getting Started

This chapter describes the process of unpacking, checking and installing the SRS CIS Analyzer on a vacuum system.

Please read and follow all installation instructions to insure that the optimum performance of the instrument is not compromised during the installation process.

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# Unpacking

#### Before You Open the Box

- 1. Please read and follow all installation instructions in this chapter to insure that the optimum performance of the instrument is not compromised during the installation process.
- 2. Do not start the installation process until all the necessary hardware components are available.
- 3. Follow good high vacuum practice. Set aside a clean, dust free, work area next to the vacuum chamber before installation begins. Do not remove the plastic plugs from the CIS probe ports until moments before the analyzer is ready to be installed in the vacuum system. Handle all vacuum hardware components with extreme care.
- 4. A pumping system is required to complete the installation of the CIS Analyzer. An oil-free, turbo pump based, pumping system, compatible with the CIS Analyzer, is available directly from Stanford Research Systems (Option O100TDP). If a pumping system is already available, unpack and/or check its integrity before the installation process begins. Consult the Pumping System Requirements section in the Quadrupole Probe chapter of this manual for more information on this subject.
- 5. For applications requiring the direct sampling of gas pressures >2mTorr, a Pressure Reducing Gas Inlet System, placed between the gas chamber and the CIS ionizer, is required to complete the installation of the CIS Analyzer. If a Pressure Reducing Gas Inlet System is already available, unpack and/or check its integrity before the installation process begins. Otherwise, Gas Inlet systems for several different pressure ranges can be ordered directly from Stanford Research Systems. Consult the High Pressure Sampling section in the General Operation chapter of this manual for more information on this subject.
- 6. Read the General Operation chapter of this manual for an overview of the instrument and its functions.
- 7. Take a moment at this time to read the CDEM Handling and Care section of the Maintenance chapter if your unit includes the electron multiplier option (Option 01).
- 8. Do not power up the instrument until it is instructed in the installation procedure.
- 9. Inspect all components of the SRS CIS Analyzer upon unpacking. Report any damage to Stanford Research Systems immediately. Compare the contents of the shipping container with the Checklist below and report any discrepancies.

#### Checklist

#### **Standard Equipment / Supplies**

- 1. One CIS Probe (with plastic plugs sealing vacuum ports.)
- 2. One Electronics Control Unit box.

- 3. Two 3.5 inch floppy disks with RGA Windows software.
- 4. DB9-DB9 RS232 cable (25' length).
- 5. DB25 to DB9 connector adapter.
- 6. Operating Manual and Programming Reference.

#### Accessories

1. Female DB-9 cable connector, with two wires for 24V power supply connection (not provided with Option 02).

#### **Optional Equipment**

- 1. Option 01 Electron multiplier, with HVPS. Preinstalled at the factory.
- 2. Option 02 Built-in power module for AC line operation. Preinstalled at the factory. Includes one power cord.
- 3. O100RFW Replacement Tungsten Filament for CIS, pkg. of 5 filaments.
- 4. O100RFT Replacement Thoriated-Iridium Filament for CIS, pkg. of 2 filaments.
- 5. O100EM Replacement Electron Multiplier.
- 6. O100RIC Replacement Ionizer Kit (includes filament) for CIS.
- 7. O100TR Computer output card, cable, relay card.
- 8. O100TS Computer output card, TTL screw terminal card.
- 9. O100CS Computer system: IBM compatible PC with factory installed RGA Windows software.
- 10. O100RM 19" Rack Mount enclosure for computer and monitor
- 11. O100BV Bypass Valve assembly with pressure reduction orifice, 2.75" CF Flanges.
- 12. O100TDP Turbomolecular pump with 2.75" CF Flange and Diaphragm Pump.

# Installation

#### Introduction

A complete SRS CIS Analyzer system consists of:

- 1. Pressure Reducing Gas Inlet System (for operation above 2 mTorr)
- 2. CIS Probe
- 3. Pumping system
- 4. Electronics Control Unit (ECU.)
- 5. RGA Windows Software.

Specific hardware requirements and installation instructions are needed for each one of the components.

#### **Important**

- Follow the installation steps in the strict order in which they are presented in this chapter.
- Do not power up the instrument until it is indicated in the procedure.
- Read the hardware requirements before installation begins. Do not start the installation procedure until all requirements are met.
- Please read and follow all installation instructions to insure that the optimum performance of the instrument is not compromised during the installation process.

#### Pressure Reducing Gas Inlet System

For applications that involve sampling gas pressures >2mTorr, a Pressure Reducing Gas Inlet System, placed between the gas chamber and the CIS ionizer, might be required to complete the installation of the CIS Analyzer. Consult the High Pressure Sampling section in the General Operation chapter of this manual for more information on this subject.

The Pressure Reducing Gas Inlet System must be available before the installation process can begin. Pressure Reducing Gas Inlet systems for several different pressure ranges can be ordered directly from Stanford Research Systems or from your authorized local representative.

Unpack and/or check the integrity of the Gas Inlet System components before the installation process starts. Also read the installation instructions at this time to make sure all the necessary hardware required by the manufacturer is available. If everything is present and in order, install the Gas Inlet System carefully following the manufacturer's

instructions. As a double check, remember that once the inlet system is installed it must have a 2.75" CF flange available to attach the CIS Probe.

#### Warning

Verify that the vacuum port is electrically grounded before attempting the installation of the Gas Inlet Valve on the vacuum chamber.

#### **Probe Installation**

For applications requiring the sampling of gas pressures <2mTorr, the CIS Probe is directly attached to any standard 2.75" CF port of the vacuum chamber. The CIS ionizer does not penetrate into the process chamber (see Figure 1). A short, wide piece of tubing establishes the connection to the process region. Due to the high conductance of the ionizer, the pressure in the ionization area is the same as in the vacuum chamber.

For systems requiring the use of Pressure Reducing Gas Inlet Systems, the CIS probe is directly attached to the 2.75" CF flange of the Pressure Reducing Gas Inlet valve.

#### Warnings:

- Use proper vacuum procedures while installing the CIS probe: Set aside a clean, dust free, work area next to the vacuum port. Wear gloves! Do not talk or breathe directly into the probe's ionizer. Use clean tools.
- Do not remove the plastic plugs from the vacuum ports until the instrument is ready to be installed in the vacuum system.
- Do not substitute parts or modify the CIS probe in any way without first consulting Stanford Research Systems or an authorized representative. Failure to do this will automatically waive the manufacturer's warranty, and may result in damage to the instrument and serious personal injury.
- Protect the integrity of the Vacuum seals: Do not use nonmetal seals. Do not dent or scratch the knife-edges of the CF flanges.
- Verify that the vacuum port is electrically grounded before attempting the installation of the CIS Probe on the vacuum system.

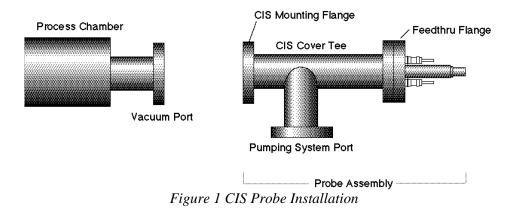
#### **Hardware Requirements**

- 1. If the presence of corrosive gases in the vacuum environment is known or suspected, consult SRS before installing the probe in the vacuum chamber.
- 2. Do not operate the CIS Analyzer in the presence of strong magnetic fields. Magnetic fields, such as those caused by superconducting magnets or unshielded cold cathode total pressure gauges (i.e. Penning type), can affect the ion trajectories through the quadrupole filter resulting in very unreliable measurements. If the presence of magnetic fields in the vacuum environment is known or suspected, consult SRS before mounting the probe on the vacuum chamber.

- 3. The maximum operating pressure of the CIS Analyzer is 10<sup>-2</sup> Torr. A Pressure Reducing Gas Inlet system is required if the operating pressure of the vacuum chamber is routinely greater than 10<sup>-3</sup> Torr. Consult the High Pressure Sampling section in the General Operation chapter of this manual for more information on this subject.
- 4. The probe can be mounted directly onto any standard 2 3/4" CF port of a vacuum chamber provided the following placement requirements are met:
  - The CIS ionizer must be located close to the point where partial pressures are to be measured. If placed near a pump, the pressure in the ionizer may be considerably lower than in the rest of the system. If placed near a gas inlet or source of contamination, the pressure in the gauge may be much higher. Long tubulation or other constrictions between the ionizer and the rest of the vacuum system can cause large errors in the partial pressure readings.
  - The probe must be protected from evaporation sources that could coat the ionizer.
  - Any mounting orientation of the probe itself may be used; however, keep in mind that enough clearance must be allowed for the Pumping System and the ECU box that are attached to the CIS Probe.
  - Choose the orientation of the pumping system prior to the installation of the CIS probe. Keep in mind that some of the older turbomolecular pumps can only be operated in a vertical orientation. In all cases, consult your pump's operation manual before you bolt anything down.
- 5. The following equipment must be available before the installation procedure starts:
  - One standard OFHC copper gasket for 2 3/4" CF flange. New and clean!
  - Six high-strength stainless steel bolts for 2 3/4" CF flanges: Use 1/4-28 x 1 3/8" bolts, with one nut and two washers per screw, for flanges with through holes; or six 1/4-28 x 7/8" bolts, with one washer per screw, to attach the probe's flanges to tapped vacuum ports. Both standard hex and 12-point bolt heads are compatible with the CIS flanges. Silver-plated bolts are preferred over messy anti-seize lubricants whenever possible.
  - Wrenches: One or two wrenches will be needed to tighten the bolts and compress the copper gasket. Use a 7/16" 12-point wrench for standard hex head bolts and nuts, or a 1/4" 12-point wrench for 12-point bolt heads.
  - Optional adapter flanges: Install an adapter flange in the vacuum system if no 2 3/4" CF ports are available. Zero-Length Adapter flanges, with a 2 3/4" CF flange on one side and a bigger size flange on the other, are economical alternatives to reducing flange sizes.

#### Procedure

- 1. Determine the placement of the probe in the vacuum chamber following the instructions listed in step 4 of the Hardware Requirements section.
- 2. Remove the plastic plug from the CIS Mounting Flange (See Figure 1). Avoid contamination following good high vacuum practices. Do not touch with bare fingers any part of the probe that will be exposed to the vacuum. Do not talk directly at an open vacuum port.
- Hold the probe in a secured upright position and do a thorough visual inspection of the part. Check for loose, damaged or misaligned components. Check the integrity of the knife edges in the exposed CF flanges.
   Important: Abort the installation procedure and contact SRS immediately if the probe appears damaged in any way.
- 4. Position the copper gasket and attach the CIS probe to the vacuum system. Avoid scratching the vacuum seals and do not use non-metal gaskets. Check the correct orientation of the Pumping System Port before bolting things down. Tighten the bolts according to the standard tightening procedure for 2.75" CF flanges. Use 1/8 turns in crisscross order: 1, 4, 2, 5, 3, 6, 4, 1, 5, 2, 6, 3, 1.... until the flanges are in contact, and finish the tightening process with an extra 1/16 of a turn on each bolt. Get an extra hand from a co-worker if necessary.



#### **Pumping System Installation**

The Pumping System usually attaches directly to the probe's Pumping System Port (see Figure 1). Carefully follow the manufacturer's instructions while mounting and interconnecting the pumps and their controllers.

A typical upgrade to the Pumping System includes a total pressure gauge dedicated to monitor the pressure on the quadrupole rods. If a total pressure gauge is part of the Pumping System, install the gauge between the CIS probe and the pumps, carefully following the manufacturer's instructions. Additional vacuum hardware is usually required to accommodate the extra gauge.

As long as the pressure in the vacuum chamber is under  $10^{-2}$  Torr, start pumping down the CIS Analyzer as soon as all the vacuum connections are completed.

If present, use the Pumping System's total pressure gauge to check the pressure on the quadrupole rods. Under normal operating conditions (i.e. gas pressure  $\approx 10^{-3}$  Torr in the process chamber, and a well matched pumping system) the pressure on the quadrupole rods should be approximately two decades lower than the chamber pressure (i.e.  $1-2^{-1} 10^{-5}$  Torr). Consult SRS or your authorized representative if large deviations in this pressure reduction factor are observed.

#### **Important:**

- If a shut-off valve is connected between the CIS Ionizer and the gas chamber and an extra total pressure gauge is part of the pumping system, the following procedure can be followed to leak test the vacuum connections: close the shut-off valve and use the output of the extra gauge to leak test the vacuum seals. The CIS probe is pre-cleaned and leak tested at the factory. The system should pump down very quickly with an ultimate pressure only limited by the outgassing rate (i.e. mostly water) from the internal walls of the CIS Probe. Check the vacuum seals if a leak in the vacuum system is detected or suspected after the vacuum components are installed.
- Contact SRS immediately if a leak in the feedthru flange is detected or suspected.
- A thorough bakeout of the CIS Probe is recommended before the CIS is used for actual measurements. Consult the Probe Bakeout instructions in the Maintenance chapter of this manual for details.
- An overnight pump-down is needed in instruments with the electron multiplier option (Option 01). Consult the CDEM Preconditioning section in the Maintenance chapter of this manual before applying any voltage across the CDEM.

#### **Electronics Control Unit Installation**

The Electronics Control Unit (ECU) attaches directly to the probe's feedthru-flange. Its correct alignment to the probe is very important during installation. Carefully follow the instructions below to insure proper alignment.

#### Warning:

Failure to follow the installation instructions below could result in damage to the ceramic feedthru connectors of the probe's flange, and would require sending the entire unit back to Stanford Research Systems for a complete flange replacement.

#### Hardware Requirements

- 1. External Power source:
  - Standard ECU boxes require an external 24 +/- 2V DC power supply @ 2.5 Amps (i.e. 60 Watt) to power its electronics.

Units with the optional built-in power module (Option 02) plug directly into a wall AC outlet and require no extra power sources.

2. Power cables:

External 24V power supplies must have a cable with a 9 pin, type D, female connector on the free end, wired as described in the "ECU- 24VDC power connector" diagram shown below.

Note: A power cable, with a properly wired, female, DB-9 cable connector, is

provided by SRS with all CIS units that do not include the built-in power module (Option 02). Attach the connector's wires to a longer two-wire cable, of similar or bigger gauge, to make a power cord compatible with the CIS's power connector. CIS units with the optional built-in power module (Option 02) are connected directly to a wall outlet using the three-wire power cord provided by SRS.

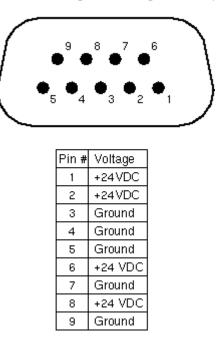


Figure 2 ECU 24 VDC Power Connector

- RS232 cable: A straight through RS232 cable with 9-pin, type D connectors is needed to connect the IBM compatible PC to the ECU box. An adapter will be needed if the RS232 port of the computer has a 25 pin Type D connector.
- 4. The probe must be installed in the vacuum chamber before the ECU box is mounted on its flange.

#### Procedure

1. Begin by inspecting the front panel of the ECU box. Use the following diagram as a reference during installation.

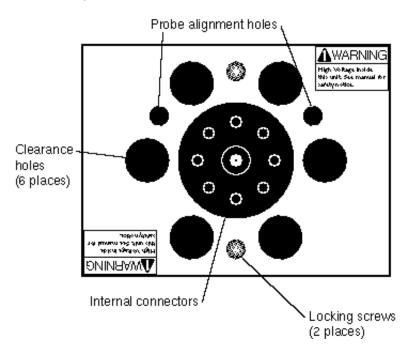


Figure 3 ECU Front Panel

- Next, inspect the probe's feedthru flange: Eight ceramic feedthru connectors on a 1" diameter circle surround a center tube terminated by a coaxial connector. Two alignment rods (1/4" diameter) insure the correct alignment of the ECU box to the probe during installation and two threaded holes (1/4-28) line up with the locking screws of the ECU box.
- 3. Push the locking screws of the ECU box as far as they will go into the box.
- 4. Orient the ECU so that the two probe alignment holes (1/4" diameter) on its front panel and the alignment rods of the flange line up. Note that there is only one correct way to do this!
- 5. Slide the ECU into the probe using the alignment rods as a guide, and until physical contact is made between the probe and the ECU's internal connectors.
- 6. Using **gentle pressure** on the back of the box, push the probe's conductors into the ECU's connectors until the front surface of the ECU rests flat against the back of the feedthru flange.

#### Warning

The alignment of the probe/ECU connections is tested at the factory, for each individual CIS Analyzer and only slight resistance should be experienced during this step. If significant resistance is noted while sliding the ECU onto the probe do not exert excessive force since that might damage the ceramic-to-metal seals of the

feedthrus. Instead, rock the ECU box up-and-down and side-to-side, while gently pushing on its back, until the connectors line up. Once the connectors are all lined up, push the box in the rest of the way.

- 7. Once the ECU is in place, use the knobs on the back panel of the ECU box, to turn the locking screws and lock the assembly in place. Do not overtighten! (Hand tighten.)
- 8. ECU Power Connection:

**Important**: Do not power up the CIS Analyzer at this time. Standard ECU boxes must be connected to the external 24 VDC power supply. Insert the 9 pin, Type D female cable connector into the ECU connector marked +24 VDC @ 2.5A on the back panel of the ECU box. Units with the optional built-in power module (Option 02) plug directly into an AC outlet and require no extra power sources: Use the power entry module on the back panel of the ECU box to power the CIS Analyzer directly from an AC outlet. Use the three-wire power cord provided by SRS to connect the CIS Analyzer directly to a properly grounded wall outlet.

9. ECU RS-232 connection: Use the straight through RS232 cable with 9-pin, type D connectors to connect the computer (usually DTE) to the CIS Analyzer. Insert the 9 pin, Type D male cable connector into the ECU connector marked: RS232/DCE/28.8k on the back panel of the ECU box. Insert the 9 pin Type D female cable connector into the RS232 port on the computer. Use the DB25 to DB9 connector adapter if the computer has a 25 pin Type D connector.

#### **RGA Windows Installation**

The RGA Windows Software is preinstalled at the factory in CIS Analyzer systems that include the optional Computer system (option O100CS).

Minimum System Requirements (Single Head Operation)

- IBM compatible 486 CPU machine at 66 MHz with 8 Mbytes of RAM or greater
- Mouse or equivalent pointing device.
- Serial port (16550 UART recommended but not necessary).
- Straight through, DB-9 to DB-9, RS-232 cable.
- 5 Mbytes of free hard disk space (for RGA installation and runtime use).
- 1.44Mbyte 3.5" floppy drive (for the installation disks).
- Super VGA graphics card running in 800x600, 256 color mode.
- A sound card if the audio features are needed.
- Microsoft Windows version 3.1 or later.

<u>Note</u>: The RGA program system requirements vary depending on the performance required of the CIS Analyzer and on how many heads are connected and run simultaneously. The CIS Analyzer can scan data at different scan speeds that effect noise floor and averaging. If you choose to scan at the fastest speed and want the RGA program to "keep up" with the head, then a faster computer, I/O card, and graphics card might be required.

#### Procedure

- 1. Turn on the computer and start MS Windows.
- 2. Insert Disk 1 of the RGA Windows software in the 1.44 Mbytes 3.5" floppy drive of the computer.
- 3. Run Setup.
- 4. The software is automatically and completely installed by the RGA installation program. Read and follow all instructions.
- 5. An "SRS RGA" program group with the "RGA Program" icon is automatically created at the end of the installation process.
- 6. Take a moment at this time to read the RGA Windows Software Chapter of this manual.

#### Turning on the CIS Analyzer

- 1. Power up the CIS Head: Standard CIS heads are powered up by turning on the external 24V power supply. Units with a built in power module (Option 02) have a power switch on the back panel of the ECU box.
- 2. When power is applied to the ECU, a firmware routine automatically checks the external voltage level and turns on the green Power LED if the voltage is within the acceptable range of 24 +/-2 V DC. The CIS Head is now ready to communicate with the computer.

# **Running the CIS Quadrupole Gas Analyzer**

This section describes how to launch the RGA Windows program and start acquiring data from the CIS Analyzer. An analog scan from 1 to 50 amu is executed as an example. For best results the pressure in the ionization region should be between  $10^{-7}$  and  $10^{-3}$  Torr.

**Important**: The following steps assume that all the installation instructions described in the previous section were completed, and no serious problems were encountered. The CIS Head should be mounted on the vacuum chamber, pumped down by its Pumping System, powered up and connected to the RS232 port of the IBM compatible PC computer. RGA Windows should be installed in the computer and ready to run.

#### Warning:

Do not operate the CIS Analyzer if the pressure in the CIS ionizer is greater than  $10^{-2}$  Torr.

- 1. Turn on the computer and start Microsoft Windows.
- 2. Start the RGA Windows Software:

To start the RGA software simply double-click on the RGA icon in the "SRS RGA" program group created by the RGA installation program:



- 3. Connect to the CIS Head:
  - Click on the toolbar's RS232 Setup button or select RS232 Setup from the Utilities menu.
  - Select the port to which the CIS head is connected (often COM2.)
  - Click on the Connect button.
  - The connection is made when the dialog box disappears, the toolbar's GO button turns green, the RS232 button is highlighted, and the Scan and Head menu commands get enabled.
- 4. Turn on the filament:
  - Click on the toolbar's Filament button
  - The ionizer is biased with default voltage values and the filament is turned on to the default emission current. It takes a few seconds for the filament wire to warm up during which a small informative dialog box is active. The Filament button remains highlighted as long as the filament is emitting electrons.

- The filament emission can be toggled on/off at any time clicking on the Filament button. The ionizer settings can be changed selecting Ionizer from the Head Menu. A green status LED on the back panel of the ECU box indicates the emission status of the filament at all times and it provides the fastest way to verify if the filament is emitting electrons.
- 5. Perform an analog scan under the current scan conditions: Analog mode is the spectrum analysis mode common to all Quadrupole Gas Analyzers. The X-Axis represents the mass range displayed in the Mass Spec Parameters menu. The Y-Axis represents the ion current amplitudes of every mass increment measured. RGA starts in the Analog mode of display by default. The scan range defaults to 1 to 50 amu.
  - Select Start from the Scan menu, or simply click on the toolbar's GO button , to trigger an analog scan.
  - After a small delay, the analog scan data starts to be displayed on the screen.
  - Rescale the Y-Axis clicking on the Auto Scale button 🖄 or selecting Auto Scale from the Graph Menu.
  - Change the Y-Axis to a logarithmic scale selecting the Log option of Select Scale in the Graph menu item.

Please refer to the RGA Windows User's Manual, and the RGA On-Line Help files, provided with the program disks, for detailed information on all the features, procedures, and commands available in the RGA Windows program.

Read the General Operation chapter of this manual for a general overview of the SRS CIS Analyzer and its basic operating modes. Consult Appendix D for information on the application of the CIS Gas Analyzer to PPM-Level Gas Analysis.

# Chapter 2 General Operation

This chapter describes the basic properties and operating modes of the Stanford Research Systems CIS Quadrupole Gas Analyzer (CIS Analyzer).

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# The SRS Closed Ion Source Quadrupole Gas Analyzer

The Closed Ion Source (CIS) Quadrupole Gas Analyzer manufactured by Stanford Research Systems is a differentially pumped quadrupole mass spectrometer specifically designed for the direct measurement of minute trace gas impurities in process gases at pressures below  $10^{-2}$  Torr.

In its most simple configuration, an SRS CIS Analyzer consists of a **CIS probe** and an Electronics Control Unit (**ECU**). The basic setup is shown in Figure 1.

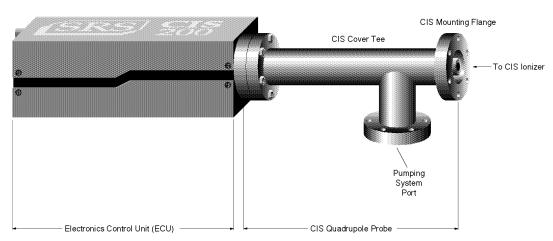


Figure 1 SRS CIS Analyzer components

The **CIS Probe** consists of a quadrupole mass spectrometer equipped with a high conductance, differentially pumped, CIS ionizer mounted inside a 2.75" Conflat Tee (CIS Cover Tee). It is described in detail in the Quadrupole Probe chapter of this manual. The total probe equipment consists of three parts: the CIS **ionizer** (electron impact), the **quadrupole mass filter** and the **ion detector**. The closed design of the ion source allows the sensor to sample at total pressures as large as  $10^{-2}$  Torr directly, while at the same time attaining sub-ppm detectability throughout the entire mass range. In the absence of spectral overlaps (i.e. overlapping background peaks), the mass spectrometer sensor is capable of detecting gas impurity concentrations under the 100 ppb level. The detector measures the ion currents directly (Faraday Cup) or, using an optional electron multiplier detector (Option 01), measures an ion signal proportional to the ion current. The probe assembly is connected directly to the process chamber through its standard CIS Mounting Flange (2.75" CF connection).

The side port of the CIS Cover Tee (i.e. Pumping System Port) provides the connection for the pumping group required to differentially pump the CIS ionizer and keep the quadrupole mass analyzer and the filaments at high vacuum during process monitoring. Please consult the Pumping System Requirements section in

the Quadrupole Probe chapter of this manual for more information on this subject.

Process pressures as large as 10 Torr can be monitored by adding an extra step of pressure reduction between the process chamber and the CIS, and without the need for any extra pumps. Consult the High Pressure Sampling section in this chapter for more information on this subject.

The **ECU** is a densely packed box of electronics (3" x 4" x 9") that mounts directly on the probe's feedthru-flange and connects to a host computer. It contains all the electronics necessary to operate the instrument.. It is described in detail in the "Electronics Control Unit" chapter of this manual. It includes several regulated power supplies, a microprocessor, control firmware, and a standard RS232 communications port. It is powered by either an external 24 VDC (@2.5 Amps) power supply or an optional built-in power module (Option 02) which plugs directly into an AC outlet. A unique, temperature-compensated, logarithmic picoammeter built into its box measures the ion currents collected by the Faraday cup (FC), or electron multiplier (CDEM). The electrometer is completely autoranging and measures both positive and negative currents with the same accuracy and resolution. Its operating range covers current magnitudes between  $10^{-7}$  and  $10^{-15}$  A, providing six orders of magnitude dynamic range during single mass measurements and scans, and detectable partial pressures better than  $10^{-11}$  Torr during electron multiplier detection.

The instrument is completed with the **RGA Windows** software package that runs on IBM compatible PC's (486 or greater). The intuitive graphical user interface allows measurements to be made and displayed in a variety of modes. The program is fully interactive and measurements are set up quickly and easily with the click of a mouse. The software also supports **multiple head operation** when more than one CIS head is needed. Please refer to the RGA Windows chapter of this manual, and the RGA On-Line Help files, provided with the program disks, for detailed information on the features, procedures, and commands available in the RGA Windows program.

Intelligent firmware, built into the CIS Head, completely controls the operation of the instrument, and provides the three basic modes of data acquisition of the mass spectrometer:

- Analog scanning
- Histogram scanning
- Single mass measurement

RGA Windows provides fast access to all the CIS Analyzer functions and modes of operation without the need for any computer programming; however, the instrument can also be programmed directly using the CIS Command Set supported by its serial interface. Consult the Programming chapter of this manual for information on the programming options and a complete listing of the CIS Command Set. The CIS Analyzer is available in three different models with mass ranges of 1 to 100 (CIS100), 1 to 200 amu (CIS 200), and 1 to 300 amu (CIS300). All models operate in the "Constant Resolution" or "Constant  $\Delta m$ " mode, with  $\Delta M_{10\%}$  preset to one amu (nominal) at the factory.

The operating pressure is UHV to  $10^{-2}$  Torr for all models.

A complete product specifications listing is included in the introductory sections of this manual.

# **Basic Data Acquisition Modes**

The SRS CIS Analyzer is a mass spectrometer that analyzes gas mixtures by ionizing some of the gas molecules (positive ions), separating the resulting ions according to their respective masses and measuring the ion currents at each mass. Partial pressure measurements are determined with the help of previously calculated sensitivity (i.e. calibration) factors by reference to the abundance of the individual mass numbers attributed to each gas type.

#### **Principles of operation**

During analysis, positive ions are formed within the ionization volume and directed towards the spectrometer's quadrupole mass-filter. The type of ionizer used in the CIS Analyzers is referred to as a closed ion source (CIS). The CIS sits on top of the quadrupole mass filter and is simply a short, gas-tight tube with two very small openings for the entrance of electrons and the exit of ions. The source is exposed to the process environment (<10 mTorr) and ions are produced by electron impact directly at the process pressure. The ions enter the quadrupole through the small aperture available. A pumping system, attached to the Pumping System Port of the CIS Cover Tee, keeps the filament and the rest of the quadrupole mass spectrometer at pressures below  $10^{-5}$  Torr through differential pumping. The closed design of the ionizer enhances the signal from the sample gas relative to the signals arising from the background gases present in the analyzer and is directly responsible for the sub-ppm detectability limits that are attainable with the CIS Analyzer.

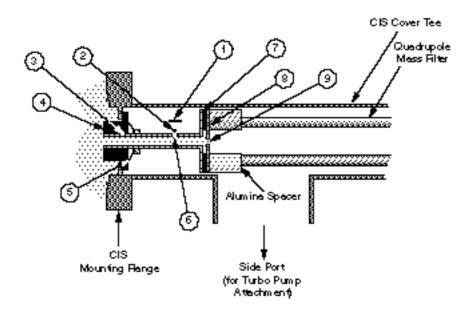


Figure 2 Cross section of the Closed Ion Source (CIS)

1. Repeller, 2. Filament, 3. Ionization Tube (Anode), 4. Alumina insulator and seal, 5. Spring washer, 6. Emission Slit, 7. Alumina insulator and seal, 8. Exit plate, 9. Exit aperture The mass filter determines which ions reach the detector at any given time. It is operated by a combination of RF and DC voltages and the filtering action is based on the mass-to-charge dependency of the ion trajectories on the RF and DC fields. The magnitude and frequency of the RF determine the **mass-to-charge ratio** of the ions that can pass through the filter without striking the rods (i.e. with stable oscillations). The RF/DC ratio determines the filter selectivity. Ions that successfully pass through the filter are focused towards the **detector** and the resulting analog current is measured by the very sensitive electrometer.

#### A brief note on Mass Units in Mass Spectrometry:

Since molecules are so small, it is convenient to define a special type of mass units to express the masses of individual ions. The **atomic mass unit, amu**, defined as 1/12 of the mass of a single carbon atom, isotope 12 (i.e. <sup>12</sup>C), is the unit of molecular mass most commonly used in mass spectrometry (**1 amu = 1.660 540 x 10<sup>-27</sup> kg**). To a very accurate approximation, the mass of a molecule in atomic mass units (amu) is equal to its **mass number M**, defined as the sum of the number of protons and neutrons in the molecule.

Mass spectrometers do not actually measure the molecular mass directly, but rather the mass-to-charge ratio of the ions. The mass-to-charge ratio, M/Q, is defined as the ratio of the mass number M of the ion to its charge Q, measured in units of the electron charge e<sup>-</sup>. For example: doubly charged ions of argon isotope 36 ( ${}^{36}\text{Ar}^{2+}$ ) and singly charged ions of water,  ${}^{1}\text{H}_{2}{}^{16}\text{O}^{1+}$ , have M/Q = 18, and cannot be differentiated from each other with most mass spectrometers.

For singly charged ions, the mass to charge ratio is numerically equal to the mass of the ion in atomic mass units (amu).

Mass spectrometer users often use the term "mass of an ion" when they really mean the mass-to-charge ratio. This convenient way of speaking is strictly valid for singly charged ions only.

#### The CIS Analyzer as a Mass spectrometer

The CIS Analyzer can perform both analog and histogram scans over its entire mass range. Partial pressure gas analysis relies on the interpretation of the spectral data generated by these two data acquisition modes to completely characterize, both qualitatively and quantitatively, a vacuum environment.

RGA Windows uses the two modes to generate the data for the Analog and Histogram Scan Modes.

**Analog scanning** is the most basic operation of the CIS Analyzer as a quadrupole mass spectrometer. During analog scanning the quadrupole mass spectrometer is stepped at fixed mass increments through a pre-specified mass-range. The ion current is measured after each mass-increment step and transmitted to the host computer over RS232. Analog scanning allows the detection of fractional masses and provides the only direct view of the peak shapes and resolution of the instrument.

A **Histogram (Bar Mode) Scan** consists of a succession of individual peakheight measurements over a pre-specified mass range. A single value is used to represent the peak heights at each integer mass within the range. The peak height measurements are made with the **Peak-locking** scanning procedure described in the next section. Histogram scanning is one of the most commonly used modes of operation for the CIS Analyzer. Its two main advantages are a faster scan rate than analog scans, and a reduced amount of data being exchanged during the scan.

#### The CIS Analyzer as a Single Gas monitor

The CIS Analyzer can measure individual peak heights at any integer mass within its mass range.

This mode of operation is used to generate data for leak testing measurements, and to track changes in the concentrations of several different components of a mixture as a function of time. The outputs provided by a set of single mass measurements are often used in process control programs to control alarms, analog and digital outputs, and relays.

RGA Windows uses this mode to generate its data for the Table, Pressure vs time, Annunciator and Leak Detection modes.

**Peak Locking procedure**: During a Single Mass Measurement the CIS Analyzer performs a Miniscan around the mass requested, and the **maximum current value** measured is **sent out over RS232**. The scanning procedure, referred to as **Peak-Locking**, is designed to measure peak currents for individual masses in a mass spectrum without being affected by drifts in the mass-axis calibration. The Miniscan covers a 0.6 amu range centered at the mass requested, and selects the maximum current from 7 individual measurements performed at 0.1 amu mass increments.

# **Basic Operating Modes**

The low detection limits, together with the wide dynamic range provided by the quadrupole mass spectrometer fitted with an electron multiplier, makes the CIS Analyzer an extremely versatile system for:

- on-line process monitoring and control,
- verification of process gas purity at the point of use,
- high vacuum residual gas analysis,
- and process equipment leak checking.

The user can easily switch between the above applications by simply selecting one of two basic CIS Operating Modes. The two modes of operation correspond to different ionizer settings and can be easily selected from the RGA Windows software.

The following table describes the ionizer parameter settings for both modes of operation.

CIS Parameter	Programmable range	RGA Mode	CIS Mode
Electron emission current (mA)	0 to 1	0.5	0.05
Electron energy (eV)	25 to 105	70	70 or 35
Ion Energy (eV)	4 or 8	8	8
Extraction Voltage (V)	0 to -150	-40 typical	-40 typical
Sensitivity (for N2 @28 amu)		10 <sup>-5</sup> A/Torr	10 <sup>-6</sup> A/Torr
Linear range upper limit (Torr)		10-4	2*10-3
MDPP (Torr)			
Faraday Cup Detector		10-9	10 <sup>-8</sup>
CDEM Detector		10-11	10 <sup>-10</sup>

<u>Table notes</u>: The CIS tests were performed using a 70 L s<sup>-1</sup> hybrid turbo pump, backed up by a high performance diaphragm pump.

#### RGA Mode (UHV < $P < 10^{-5}$ Torr)

In the RGA mode the CIS Analyzer is exposed to pressure levels below  $10^{-5}$  Torr and used for high vacuum residual gas analysis and process equipment leak checking.

This mode is used, for example, during the first stage of a sputtering process when the chamber is evacuated to a low pressure and the quality of the vacuum is checked for leaks and harmful contaminants. The electron ionization energy is 70 eV so that the mass spectra show fragmentation patterns identical to those obtained with conventional Residual Gas Analyzers (RGA). The electron emission current is set to 0.5 mA to bring the sensitivity to levels comparable to those expected from conventional RGA's. Under these operating conditions, typical sensitivities are in the order of 10<sup>-5</sup> Amp/Torr or better. This sensitivity is about an order of magnitude lower than what is observed in a typical RGA; however, the difference can easily be made up by running the electron multiplier at higher gain levels. The sensitivity remains constant from UHV until pressures as high as 10<sup>-4</sup> Torr. The MDPP levels, as measured with the Faraday Cup (FC) detector, are in the order of 10<sup>-9</sup> Torr and can be improved by three orders of magnitude using the CDEM's gain.

#### CIS Mode $(10^{-5} < P < 10^{-2} \text{ Torr})$

In the CIS mode, the ionizer is exposed to pressure levels between  $10^{-5}$  and  $10^{-2}$ Torr and used for on-line process monitoring and quality control.

This mode is used, for example, during the second stage of a sputtering process after the chamber is backfilled with a few mTorr of Ar and sputtering is started.

By far, the most common application of the instrument in the CIS Mode of operation is to monitor process gas purity at the point of use. Modern day purity requirements usually fall in the ppm and sub-ppm levels, and immediate response to contamination events is required to minimize costly defects and failures in finished products. The closed design of the ionizer is entirely responsible for the low detection limits that are achieved with the CIS Quadrupole Gas Analyzer. Ionization takes place directly at the process pressure inside a gold-coated anode tube that does not contribute to the background signals. The ionizer is also isolated from the rest of the sensor through differential pumping which minimizes the backsteraming of impurities from the quadrupole assembly volume into the ionization region. The combination of direct sampling and differential pumping provides the potential for ppm and even sub-ppm detection limits for even the most pervasive residual gases. A well matched Pumping System combined with careful control of contamination sources is all that is generally required to properly operate the instrument. Read the Pumping System Requirements section in the Probe Chapter of this manual for details on pumps' specifications. Consult Appendix D for information on the application of the CIS Analyzer to PPM-Level Gas Analysis.

In the CIS Mode of operation, the electron emission current is reduced to 0.05 mA to bring down the ion densities in the ionizer, and minimize signal nonlinearities in the mTorr pressure range. The electron energy can be set to either 35 or 70 eV. The 70 eV setting (i.e. **CIS-70** mode) provides spectra that are virtually identical to those obtained from RGA detectors. The 35 eV setting (i.e. **CIS-35**) is commonly used in sputtering monitors to eliminate the <sup>36</sup>Ar<sup>++</sup> peak (350 ppm for 70 eV electrons) that interferes with water detection at 18 amu. Typical sensitivities are in the order of  $10^{-6}$  Amp/Torr for 70 eV electrons, and very linear sensitivity response is observed all the way up to 1 mTorr of total pressure. The overall sensitivity of the CIS Analyzer in the CIS Mode is about two orders of magnitude lower than what is observed in a typical RGA; however, the larger density of molecules in the ionizer makes up the difference and results in ion currents very comparable to those observed in conventional RGAs. A slight decrease in sensitivity is often observed when the electron ionization energy is reduced from 70 eV to 35 eV. MDPP levels, as measured with the FC, are in the order of  $10^{-8}$  Torr and can be easily improved up to three orders of magnitude using the CDEM's gain. The MDPP values clearly indicate that the CDEM must be activated in order to obtain sub-ppm detectability.

# High Pressure Sampling (P>1 mTorr)

A Pressure Reducing Gas Inlet System (PRGIS) must be used in combination with the CIS Analyzer to sample gas pressures above  $2 \cdot 10^{-3}$  Torr. In its most simple form, a PRGIS consists of a restriction that, when placed between the gas chamber and the CIS Analyzer, keeps the ionization region at a suitable operating pressure (i.e. <2 mTorr). Common restrictions, used to create pressure differentials, are pinholes and capillaries.

PRGIS's are available directly from Stanford Research Systems. Please consult the factory or your local representative for information on this optional equipment. However, due to the large number of highly specialized applications of CIS Analyzers, it is not uncommon for some users to design their own custom-made inlet systems.

In addition to achieving the desired pressure reduction, the design of a PRGIS should provide for a fast response and high signal-to-background ratio. At pressures common to many vacuum processes (i.e. 10 mTorr to 10Torr), a simple aperture based PRGIS is suitable. At atmospheric and higher pressures, a two stage reduction based on a capillary and aperture should be used. A carefully designed system, with minimized internal surface area and vacuum compatible components will provide the lowest background contribution to the signal.

In order to keep the versatility of the CIS Analyzer intact, SRS recommends that the restriction be part of a dual path inlet system configuration so that the ionizer can still be exposed "unrestricted" to the process pressure during RGA Mode operation.

Users designing their own inlet systems will need to know that the nominal conductance of the CIS Ionizer is  $0.4 \text{ L s}^{-1}$ . For example, a 10 mTorr gas, can be sampled into the CIS Ionizer through an aperture with a maximum conductance of  $0.04 \text{ Ls}^{-1}$ (=  $0.4 \text{ Ls}^{-1}$ /10) which provides a minimum 10-fold reduction in pressure.

General information on the design and basic operation of differentially pumped vacuum systems can be found in most vacuum technology textbooks. A particular good treatment of the subject can be found in the book by John. H. Moore et.al.: "Building Scientific Apparatus", Addison Wesley Publishing Co, Redwood City, CA, 1989 (ISBN 0-201-13187-0). Information on Pumping System Requirements for the CIS Analyzer can be found in the Quadrupole Probe Chapter of this manual. General information on the use of quadrupole mass spectrometers to sample high-pressure gases can be found in Appendix B.

### **Partial Pressure Analysis Basics**

The CIS Analyzer can perform both qualitative and quantitative analysis of the gases in a vacuum chamber. Obtaining spectra with the CIS Analyzer is very simple. Interpreting the spectra, that is, understanding what the spectra is trying to tell you about your process requires some work.

The following sections will introduce some basic concepts of Spectral Analysis emphasizing the main aspects of Partial Pressure Gas Analysis. No assumptions are made as to the objective of the partial pressure analysis. The CIS Analyzer is a very versatile intrument and can be used for a very large range of applications. Different users will need to taylor this information to their specific needs. For additional information on the subject of Partial Pressure Gas Analysis refer to:

- 1. J. Drinkwine and D. Lichtman, Partial Pressure Analyzers and Analysis, AVS Monograph Series published by the Education Committee of the American Vacuum Society
- Basford et. al., J. Vac. Sci. Technol. A 11(3) (1993) A22-40 "Recommended Practice for the Calibration of Mass Spectrometers for Partial Pressure Analysis. Update to AVS Standard 2.3".

For information on multiple linear regression analysis consult:

- William H. Press, et. al., 1992, Numerical Recipes in C, The Art of Scientific Computing, Second Edition, Cambridge Univ. Press, section 15.4, page 671.
- 2. Bevington, P.R., 1969, Data Reduction and Error Analysis for the Physical Sciences, New York, McGraw-Hill, Chapters 8-9.

#### How Mass Spectra are Interpreted

A mass spectrum, taken in a real system, will almost always contain signals from a mixture of various gases. Careful and complete interpretation of the spectrum (i.e. a complete spectral analysis) should reveal the identity, as well as the concentrations, of the various components which have produced the spectrum.

The first step in the spectral analysis process is to correctly identify the mass-tocharge ratio of all the peaks in the mass spectrum. A well calibrated mass scale is essential to this task. See the Tuning Chapter of this manual for a detailed description of the mass scale calibration procedure.

Once all the peaks have been labeled, the next step is to identify the residual gases that have produced the spectrum. Knowledge of your process or the recent history of your system may provide very valuable clues as to the possible gases that may be present in the vacuum chamber. A familiarity with the standard spectra of commonly expected gases will generally help to determine the major and minor components in the system. Any peak in the spectrum may consist of contributions from molecular ions and/or fragment ions, or multiply ionized

species. The qualitative spectral analysis is completed when all the peaks in the spectrum have been "uniquely assigned" to the components of a gas mixture, in complete agreement with the known fragmentation patterns of the components.

In cases where only the major components are of interest, some of the minor peaks of the spectrum will remain unassigned. If only a few species are being monitored, only the peaks corresponding to the substances of interest need to be assigned and monitored.

**Notes on Fragmentation Patterns**: The electron impact type of ionizer used in modern quadrupole mass spectrometers almost always causes more than one kind of ion to be produced from a single type of gas molecule. Multiple ionization, molecular fragmentation and changes in the isotopic composition of the molecule are responsible for the effect. All ions formed contribute to the mass spectrum of the molecule and define its **fragmentation pattern**. The identification and interpretation of mass spectra must begin with a knowledge and understanding of the standard fragment patterns of atoms and molecules that may exist in the system. The standard fragment patterns of most molecules commonly encountered in residual gas analysis are well established and listed in the general Partial Pressure Analysis Literature. A very complete library can also be accessed through the Library Search Utility of the RGA Windows software. The Gas Library has a standard text file format, and can easily be read, extended or modified by the user to fit his individual needs.

**Partial Pressure Gas Analysis Tip:** Virtually every vacuum system will have detectable amounts of hydrogen (2 amu), water (18 amu), carbon monoxide (28 amu) and carbon dioxide (44 amu). Become familiar with these species and their fragmentation patterns, and use their peaks to verify the correct performance of the instrument (i.e. mass scale calibration and mass resolution) while operating the CIS Analyzer.

#### **Partial Pressure Measurement**

Once the different components of a mixture have been identified it is possible to use the CIS Analyzer to obtain quantitative values for the various partial pressures. This section describes the basic steps needed to perform quantitative measurements with the instrument. The formalism presented assumes multiple gas analysis, but is equally valid for single gas measurements. Please consult the suggested references for details and examples of these procedures. The entire mathematical formalism used to derive the partial pressures of a mixture based on a single mass spectrum is based on one assumption:

The total spectrum is a linear combination of the spectra of the different species that are present in the mixture. In other words, the total spectrum is equal to the sum of the individual peaks that would be observed if each constituent were alone in the system.

In mathematical terms, the assumption stated above can be written as the following linear equation:

$$H_{\rm M} = \Sigma_{\rm g} h_{\rm Mg} \tag{1}$$

where:

g is an integer variable that represents the gases present (i.e. assign an integer to each gas starting with one)

 $\mathbf{M}$  is an integer variable that represents the mass numbers for the entire mass range of the spectrum.

 $H_M$ = total peak height (amps) of the spectrum at mass number M.

 $h_{Mg}$  = peak height contribution (amps) from gas g at mass M.

 $h_{Mg}$  is related to the fragmentation pattern, the CIS Analyzer's sensitivity and the partial pressure of gas g by the equation:

$$h_{Mg} = \alpha_{Mg} \, S_g \, P_g \tag{2}$$

where:

 $\alpha_{Mg}$  = Fragmentation factor of gas g at mass M: Ratio of ion signal at mass M to the ion signal at the principal mass peak for gas g.  $S_g$  = CIS Analyzer's partial pressure sensitivity factor for gas g, in amp/Torr (see Partial Pressure Sensitivity Factor below)  $P_g$  = Partial pressure of gas g in the system.

Equations (1) and (2) are combined to obtain the system of equations:

$$H_{\rm M} = \Sigma_{\rm g} \left( S_{\rm g} \, \alpha_{\rm Mg} \right) \, P_{\rm g} \tag{3}$$

Since all gases have more than one peak in their fragmentation pattern, the number of peaks (M) in a real spectrum is generally larger than the number of gases (g). As a result, the system of equations (3) usually has more equations than unknowns. This situation is sometimes simplified eliminating some of the extra equations; however, the best results are obtained using all the equations and a multiple linear regression procedure to calculate the best possible fit to the data.

Obviously, accurate results can only be obtained if the constants  $\alpha_{Mg}$  and  $S_g$  are well known for the analyzer being used.

**Note:** The Analyze Utility of RGA Windows uses a multiple linear regression algorithm, as mentioned above, to automatically calculate the composition of a "typical residual gas environment" at the end of any 1-50 amu spectral scan. Please see the RGA On-Line Help files for details.

Standard fragmentation patterns (for example, the fragmentation patterns included in the RGA Library of RGA Windows) can be used as a source of  $\alpha_{Mg}$  values in moderately quantitative determinations. However, when very precise numbers are desired, one should obtain the appropriate fragment patterns by introducing pure gas into the instrument being used. The fragment patterns must be obtained under the same conditions that will be used during regular spectral analysis since they depend on many instrumental parameters, including: electron energy, emission current, ionizer design, mass filter settings, detector type, multiplier gain, etc. The principal mass peak of a fragmentation pattern is simply the most intense peak of the spectrum, and the intensity of all the other peaks in the pattern are normalized to its height for the calculation of fragmentation

factors. Note that by our definition the  $\alpha_{Mg}$  value for the principal mass peak of any gas is equal to one. Principal mass peaks are used in the calculation of the sensitivity of the CIS Analyzer to different gases as shown below.

#### Partial Pressure Sensitivity Factors

The partial pressure sensitivity of the CIS Analyzer to a gas g,  $S_g$ , is defined as the ratio of the change (H-H<sub>0</sub>) in principal mass peak height to the corresponding change (P-P<sub>0</sub>) in total pressure due to a change in partial pressure of the particular gas species. H<sub>0</sub> and P<sub>0</sub> are background values.

 $S_g = (H-H_0) / (P-P_0)$ 

The units of  $S_{\rm g}$  are of ion current per unit pressure (amp/Torr, for example).

The sensitivity of the CIS Analyzer varies with different gases, changes with time due to aging of the head, and is a strong function of the operating mode of the instrument. Careful quantitative analysis requires that the sensitivity factor,

 $S_g$ , be determined for every gas which may be a component gas in the system being analyzed. The sensitivity factors must be obtained under the same operating conditions that will be used during general partial pressure analysis since they depend on many instrumental parameters, including: ionization energy, emission current, mass filter setting, type of detector, etc. For example, the RGA, CIS-70 and CIS-35 modes of operation will all have different sensitivity factors for the same gas, since they correspond to three different sets of ionizer conditions.

In order to separate the gain of the electron multiplier from the intrinsic sensitivity of the CIS head, the sensitivity factors of the CIS Analyzer are defined for Faraday Cup detection. A separate Electron Multiplier Gain Factor, is used to correct the ion signals when the electron multiplier is turned on. See the Sensitivity and Electron Multiplier Tuning sections of the Tuning Chapter for details.

The basic procedure for determining the sensitivity of a particular gas in the CIS Analyzer is the following:

- Introduce the pure gas into the vacuum system, at a known or calculable pressure (typically around 10<sup>-6</sup> Torr).
- Measure the output signal from the CIS for the principal mass peak of that gas using the Faraday cup detector.
- The ratio of this output signal to the pressure of the gas is the sensitivity factor,  $S_{\rm g}$ .

During these measurements it is very important to insure that the partial pressures of all other gases in the system are small enough so that they may be neglected. The sensitivity factors calculated can only be applied to situations where the CIS Analyzer is used with the same operating parameters. See the Sensitivity Tuning section in the Tuning chapter of this manual for more details on this calibration procedure.

The underlying assumption when using sensitivity factors in quantitative calculations is that there is a linear relation between the partial pressure and the corresponding CIS Analyzer signals of the gases. Deviations from linearity are to be expected above certain pressure values due to space charge effects in the ionizer and ion-neutral scattering interactions in the filter. A more thorough check of the CIS Analyzer's sensitivity involves measuring the ion signals over several orders of magnitude of partial pressure to determine the range over which a linear relationship exists. The sensitivity factor for the gas is calculated as the slope of the "signal vs. partial pressure" response over the linear range.

**RGA Windows** uses three sensitivity factors stored in the non-volatile memory of the CIS Head. The sensitivity factors, one for RGA Mode of operation and two for the two different values of electron energy (i.e. 35 and 70eV) in the CIS Mode of operation, are used as conversion factors between the ion currents received form the head and the pressure units selected by the user. The sensitivity factors are measured with the Faraday Cup detector and can be updated or changed very easily using the Sensitivity Tuning command in the Head menu. A separate Electron Multiplier Gain Factor, stored in the nonvolatile memory of the CIS Head, is used to correct the ion signals for the gain of the electron multiplier. The gain of the electron multiplier is highly mass dependent and defined relative to the corresponding FC signal. An automatic Electron Multiplier Gain Adjustment command, built into the program, can adjust the CDEM voltage for any gain between 10 and 10<sup>6</sup>. Consult the RGA On-Line Help Files for details on the automated tuning procedures built into the RGA Windows program. Also see the Sensitivity and Electron Multiplier Tuning sections of the Tuning Chapter for more general information.

The Table mode of RGA Windows offers scaling factors for all of its channels eliminating the limitations imposed by the single sensitivity factor on multiple partial pressure calculations. For example, the scaling factors can be used to display correct partial pressure for all the species in a table if the ratios between the partial pressure sensitivities of the different components are known and only principal mass peaks are used to monitor them. The scaling factors can also be adjusted to correct against the mass discrimination of the electron multiplier's gain.

**Important**: Following current industry standards, the partial pressure sensitivity factors stored at the factory correspond to N<sub>2</sub> measured at 28 amu with  $\Delta m_{10\%}=1$  amu, default ionizer settings for the corresponding mode of operation, and Faraday Cup Detection.

#### Single gas measurement example:

Monitoring the concentrations of one or few components in a system is easy in the absence of severe spectral interference.

Suppose a system where argon is measured at 40 amu (principal mass), in the absence of any other gases that contribute a signal at that mass value. The sensitivity to argon was previously measured at  $S_{Ar}=10^{-5}$  amps/Torr (RGA Mode), and the electron multiplier is biased and its gain at mass 40 was previously measured at  $g_{CDEM}=1.02\cdot10^3$  relative to the FC signal.

The partial pressure of argon,  $P_{Ar}$ , is easily calculated measuring the intensity (i.e. peak height) of the ion current at mass 40,  $I_{40}$ .

$$P_{Ar} = I_{40} / (g_{CDEM} \cdot S_{Ar})$$
 , units of Torr (4)

The peak value,  $I_{40}$ , can be extracted from a spectral scan or measured directly using the single mass measurement mode of the CIS Analyzer. For example, a  $10^{-10}$  amp peak value corresponds to  $9.8 \cdot 10^{-9}$  Torr of Ar. Note that equation (4) is a particular case of equation (3), and that the fragmentation factor for the principal peak of Ar is one by definition.

# Chapter 3 Quadrupole Probe

This chapter describes the design and principles of operation of the components of the CIS Analyzer Probe.

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## Introduction

The Closed Ion Source (CIS) Quadrupole Gas Analyzer manufactured by Stanford Research Systems is a differentially pumped quadrupole mass spectrometer specifically designed for the direct measurement of minute trace gas impurities in process gases at pressures below 10 mTorr.

In its most simple configuration, an SRS CIS Analyzer consists of a **CIS probe** and an Electronics Control Unit (**ECU**) which mounts directly on the probe's feedthru flange and contains all the electronics necessary to operate the instrument. The basic setup is shown in the following figure.

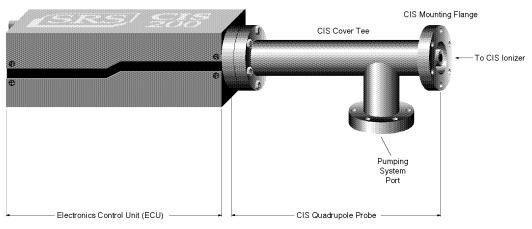


Figure 1 SRS CIS Analyzer components

The **CIS Probe** consists of a quadrupole mass spectrometer equipped with a high conductance, differentially pumped, CIS ionizer mounted inside a 2.75" Conflat Tee (CIS Cover Tee). The total probe equipment consists of three parts: the **closed ionizer** (electron impact), the **quadrupole mass filter** and the **ion detector**.

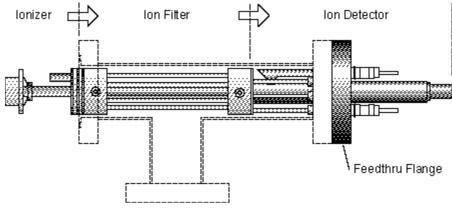


Figure 2 CIS Probe Components

The side port of the CIS Cover Tee (i.e. Pumping System Port) provides the connection for the pumping group required to differentially pump the CIS ionizer and keep the quadrupole mass analyzer and the filaments at high vacuum during process monitoring.

### Ionizer

The type of ionizer used in the CIS Analyzers is referred to as a **closed ion source (CIS)**. Positive ions are produced in the ionizer by bombarding gas molecules with electrons derived from a heated filament. The ions are then directed toward the entrance of the ion filter where they are separated based on their mass-to-charge ratio.

#### Description

The CIS sits on top of the quadrupole mass filter and is simply a short, gas-tight tube with two very small openings for the entrance of electrons and the exit of ions. The source is exposed to the process environment (<10 mTorr) and ions are produced by electron impact directly at the process pressure. Electrons enter the ionizing region through an entrance slit of dimensions: 0.190" x 0.025". The ions are formed close to, and attracted by, a single extraction plate and exit the ionizer through a circular aperture of 0.062" diameter centered on the quadrupole axis. Alumina rings seal the tube from the rest of the QMS and provide electrical insulation for the biased electrodes.

A pumping system, attached to the Pumping System Port of the CIS Cover Tee, keeps the filament and the rest of the quadrupole mass spectrometer at pressures below  $10^{-5}$  Torr through differential pumping. The closed design of the ionizer enhances the signal from the sample gas relative to the signals arising from the background gases present in the analyzer and is directly responsible for the sub-ppm detectability limits that are attainable with the CIS Analyzer.

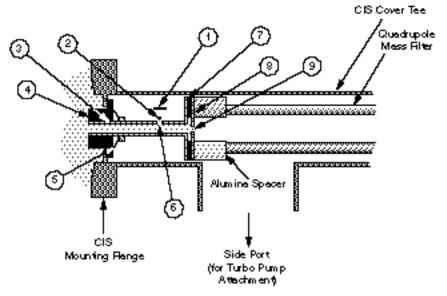


Figure 3 Cross section of the SRS CIS

1. Repeller, 2. Filament, 3. Ionization Tube (Anode), 4. Alumina insulator and seal, 5. Spring washer, 6. Emission Slit, 7. Alumina insulator and seal, 8. Extraction plate, 9. Exit aperture

The CIS was designed to fit the same quadrupole mass filter that is standard in the SRS RGA analyzers (SRS models RGA100, RGA200 and RGA300), while keeping in mind the same basic requirements of simplicity, cleanliness and ease of maintenance.

The calculated gas conductances of the standard CIS apertures, assuming room temperature <sup>40</sup>Ar gas in molecular flow regime, are  $0.3 \text{ L s}^{-1}$  for the electron slit and  $0.2 \text{ Ls}^{-1}$  for the ion exit hole. The tube itself is approximately 1" long and has an internal diameter of 0.27" corresponding to a conductance of  $1.5 \text{ Ls}^{-1}$ . The effective conductance calculated at the tube's entrance (i.e. considering the length of tube and the two holes in series) is approximately  $0.4 \text{ L.s}^{-1}$ . The pressure in the ionizing region is very uniform and virtually identical to the process pressure. The conductance numbers for the CIS are very similar to those in most commercially available CIS systems and correspond to a high conductance between the process chamber and the analyzer. A high conductance is desirable to minimize backstreaming from the analyzer chamber into the ionization region.

The entire ionizer is made out of gold-plated SS304. Gold plating serves several purposes:

- It reduces the background levels in the ionization region: Gold is very inert and does not adsorb species as readily as stainless steel does. This results in reduced outgassing and electron stimulated desorption (ESD). Gold is also a very good IR reflector and reflects the radiation emitted by the filament back into the analyzer chamber. As a result, the ionizer walls run cooler, and outgas less than with almost any other wall material.
- It improves the long term stability of the instrument. Surface contamination in the ionizer is one of the leading causes of performance degradation and filament failure in quadrupole gas analyzers. Gold is very inert and does not interact with many of the species that are present in modern day processes.
- It lets the ionizer be exposed to many reactive and corrosive gases that would otherwise react with the wall materials.

The ionizer is fitted with a straight Tungsten (W) filament  $(ThO_2 \cdot Ir is only offered as an option)$ . The straight filament wire is positioned in close proximity to the emission slit. The electrons are very efficiently directed into the slit by a negatively biased repeller plate mounted behind the wire. Tungsten material is the preferred choice for process monitoring in the semiconductor industry, because it minimizes chemical reactions reducing background and extending filament lifetime. The filament is easily replaced by the end user in a few minutes without any need to take apart the ionizer. Consult the Filament replacement section of the Maintenance Chapter in this manual for a complete filament replacement procedure. The tight design of the filament-electron slit region makes it very easy for the filament to maintain the requested emission currents even under the lowest accelerating fields. As a result, the CIS is very comfortable operating with 35 eV electrons. In factory tests, and in the absence of reactive or corrosive gases, tungsten filaments were operated continuously for weeks at a time without any degradation in performance or burn-outs.

#### **Principle of operation**

The filament is the source of the electrons used to ionize the gas molecules. It operates at a negative potential relative to ground and is resistively heated to incandescence with an electrical current from the emission regulator. The thermionically emitted electrons are accelerated towards the emission slit of the anode, which is positively charged with reference to the filament and ground. Most electrons do not strike the anode wall, but rather pass through the emission slit and into the ionization volume where they create ions, at the process pressure, through *electron impact ionization*. Electrons which pass

through the ionization volume without colliding with any molecules are simply lost to the internal walls of the ionization tube. The technique of electron impact ionization requires low pressure for the efficient production of electrons and the subsequent formation of ions. During normal operation, the filament must be kept at pressures under  $10^{-4}$  Torr, while the ionizer is directly exposed to the process pressures as large as 10 mTorr. A careful choice of the pumping system is necessary to achieve the required pressure differentials. Consult the Pumping System Requirements section in this chapter for details on this subject.

The ions formed within the ionization volume, are extracted from the ionizer by the electric field produced by the difference in voltage bias between the anode tube and the extraction plate. The extraction plate is kept at a negative potential (relative to ground) and its function is to draw the ions out of the ionization volume and into the filter section. The repeller plate, which backs the filament, is biased negative relative to the filament and reduces the loss of electrons from the ion source. **The Repeller and the Extraction Plate are only biased while the filament is emitting electrons.** 

The technique of electron impact ionization requires low pressures for the efficient production of electrons and the subsequent formation of ions. As mentioned above, the CIS Ionizer can be exposed to process pressures as large as 10 mTorr without any additional pressure reduction. However, two problems arise in the CIS Analyzer as the pressure at the closed source inlet goes above 2 m Torr:

 Deviations from the linear response to partial pressure appear in the current signals. As the ion density starts to approach the saturation level (i.e. as defined by space charge repulsion effects) the partial pressure sensitivity starts to decrease. The linearity range can be extended somewhat by further decreasing the electron emission current to reduce the ion density in the ionizing volume. However, as the pressure increases the mean free path for the ion-molecule collisions starts to get comparable to the CIS dimensions and increasing numbers of ions get diverted and lost before they can reach the exit aperture.
 The high conductance path between the CIS and the quadrupole mass analyzer results in elevated pressures on the quadrupole mass filter and detector that are not compatible with the safe operation of the CDEM.

The CIS apertures were designed such that, during normal operation, the pressure in the quadrupole volume can be at least 100x smaller than the pressure in the ionization region. Under these conditions, a 10<sup>-3</sup> Torr process pressure results in a "comfortable"  $10^{-5}$  Torr pressure on the filament, quadrupole and detector. A 100x pressure drop can be achieved pumping on the CIS with an effective pumping speed of at least 40 Ls<sup>-1</sup>. These pumping speed levels can be accomplished with even some of the smallest modern turbomolecular pumps. However, a large increase in effective pumping speed at the ionizer cannot be expected increasing the size of the turbo pump. Ultimately, the effective pumping speed is limited by the 50 Ls<sup>-1</sup> conductance of the 1.5" OD piping that connects the CIS probe to the pump. As a result, as the pressure in the ionization region increases above 1 mTorr, the pressure in the quadrupole rises above  $10^{-5}$  Torr, reducing the lifetime of the filament and the CDEM, and increasing the chances of contamination and chemical reactions at the quadrupole. The CIS Analyzer can still be operated, even at pressures as large as 10 mTorr. However, the CDEM should not be used and the linearity of the signal will be compromised. The use of the CDEM is definitely not recommended for pressures above 2 mTorr.

(Note that the signal non-linearities we are referring to are for the main components of the gas mixture and do not necessarily affect a composition analysis. For example for a sputtering process at 5 mTorr it might not be possible to do an accurate reading of the Ar partial pressure, however it is still possible to do a fairly accurate determination of water levels relative to the Ar readings.)

What SRS recommends for measurements of process gases above 1 mTorr is that the ion source inlet be operated under 2 mTorr by the addition of an extra pressure reduction stage. For more information on this subject consult the High Pressure Sampling section in the General Operation Chapter of this manual.

#### **Parameter Settings**

The parameters that affect the ionization efficiency of the CIS are: electron energy, ion energy, electron emission current and extraction voltage. The general principles by which they affect the performance of the source are well understood. The ECU contains all the necessary high voltage and current supplies needed to bias the ionizer's electrodes and establish an electron emission current. The ionizer settings can be directly controlled and monitored by the user through the instrument's high level command set, or from simple menu selections within the RGA Windows software.

The following table summarizes the ionizer settings available to the user, for the two basic modes of operation of the CIS Analyzer. For details on the two modes of operation consult the Basic Operating Modes section in the General Operation chapter of this manual.

CIS Parameter	Units	Programmable range	Minimum Increment.	RGA Mode	CIS Mode
Ion Energy	eV	4 or 8	n.a.	8	8
Electron energy	eV	15 to 105	1	70	70 or 35
Extraction Voltage	V	0 to -150	1	-40 def.	-40 def.
Emission current	mA	0 to 1	.02	0.5	0.05

The average **electron energy**, expressed in eV, is equal to the voltage difference between the filament and the anode tube and can be set anywhere in the range of 15 to 105 eV. For electrons to produce ionization of gas molecules by bombardment, they must have a certain minimum kinetic energy. This minimum energy is called the **ionization potential** and is different for every molecule. Above the threshold, the ionization efficiency increases linearly with the electron energy until a maximum is reached. For most molecules, this maximum is in the range of about 50 -100 eV, and for electron energies above the maximum, the ionization efficiency slowly decreases with electron energy. The minimum electron energy required to doubly ionize a gas molecule (i.e. to generate a doubly charged ion) is always larger than the energy required to remove a single electron from the same molecule. This property of the ionization potentials is used in the CIS Analyzer to eliminate spectral background interferences. For example, while looking for water impurities in Ar sputtering processes, 35 eV electrons are often used in the CIS Mode of operation to eliminate the <sup>36</sup>Ar<sup>++</sup> peak (350 ppm for 70 eV electrons) that interferes with water detection at 18 amu. This mode of operation, referred to as CIS-35., is so commonly used that it is standard menu option in the RGA Windows software.

The kinetic energy of the ions as they move down the ion filter, simply referred to as the ion energy, and expressed in eV, is equal to the voltage biasing of the anode tube. Two ion energy settings (i.e. anode voltages) are: 4 (low) or 8 eV (high). The ion energy setting affects the magnitude of the ion signals collected (i.e. sensitivity of the spectrometer) and limits the ultimate resolution of the mass filter. Newly formed ions are attracted by the negative potential of the extraction plate and passed to the ion filter where they are decelerated by the ground potential at the central axis of the quadrupole rod assembly. Hence, the anode voltage defines the kinetic energy of the ions as they enter the ion filter. Ion energy determines the time spent by the ions in the filter and hence, limits the resolution that can be obtained. It is well established that the resolution limit is governed by the number of cycles of RF field to which the ions are exposed before they reach the detector. In practice, the minimum resolution,  $\Delta m_{10\%}$ , is mass independent, linearly related to the ion energy, and inversely proportional to the square of the product of the quadrupole length and frequency. The two available ion energy settings correspond to ultimate resolutions of approximately 0.2 and 0.3 amu (well under the 1 amu factory default setting). Ion energy also determines the time spent by the ions in the fringing fields at the entrance and exit points of the filter. Ions passing through the fringing fields can collect high transverse velocities and are more likely to collide with the quadrupole rods and never be collected at the detector. As a result, ion signals (i.e. sensitivity) generally increase with ion energy.

The **extraction plate** negative potential can be adjusted to any value within the range of 0 to -150 V. In general, the voltage is selected so as to optimize the ion signals. The plate, with its small exit hole, serves several purposes: it separates the ionizer from the quadrupole assembly, it draws the ions away from the ionization volume, and it contains the ionizing electrons inside the source. Electron leakage into the filter is only detectable at low mass settings (1 to 10 amu) and can easily be eliminated biasing the extraction plate at negative potentials (generally -40V is all that is necessary).

The **electron emission current** is the electron current from the filament to the ionization tube. The available emission current range is 0 to 1.0 mA.

When an electron emission current is requested, the CIS Analyzer biases the ionizer's electrodes and activates the filament's heater until the desired emission current is achieved. A "**Background Filament Protection**" Mode is automatically enabled to monitor the performance of the filament while it is emitting electrons. The Filament LED remains on as long as the filament is emitting electrons. If a problem is detected in the operation of the filament (i.e. burnt filament or overpressure), the heater is immediately shut down and the problem is prompted by the LED's. If a current of 0 mA is requested, the filament's heater is shut down, the repeller and focus plate are biased to ground, the CDEM is turned off (if necessary), and the Filament LED is turned off to indicate the absence of emission in the ionizer.

The electron emission current is very tightly regulated by a feedback control loop which dynamically adjusts the operating temperature of the filament to keep the total emission current constant. The emission of electrons from the filament is limited by space-charge

effects (Child's Law limitations). The maximum current attainable is a function of the anode-to-repeller spacing and its potentials. The tight design of the filament-emission slit region makes it very easy for the filament to emit electrons even under the lowest accelerating fields. As a result, the SRS CIS is very comfortable operating with 35 eV electrons as required by the CIS-35 mode of operation.

Ideally, the rate of formation of ions should be proportional to the electron emission current. In practice, the exact dependence between ion signal and electron emission current in small ion sources is complicated by space-charge effects and only general trends can be predicted. Greater ionization efficiency is to be expected as the electron emission current increases. However, as the ion density starts to approach the saturation level (i.e. as defined by space charge repulsion effects in the ionization volume) the partial pressure sensitivity starts to decrease. The linearity range can be extended somewhat by further decreasing the electron emission current to reduce the ion density in the ionizing volume. However, as the pressure increases the mean free path for the ionmolecule collisions starts to get comparable to the CIS dimensions and increasing numbers of ions get diverted and lost before they can reach the exit aperture. A change in the emission current level is the main difference between the two basic operating modes of the CIS analyzer (RGA and CIS Modes of operation.) For details on the two modes of operation consult the Basic Operating Modes section in the General Operation chapter of this manual.

### **Quadrupole mass filter**

Positive ions are transferred from the ionizer into the quadrupole where they are filtered according to their mass-to-charge ratios. Ions that successfully pass through the quadrupole are focused towards the detector by an exit aperture held at ground potential.

#### Description

The quadrupole mass filter is an electrodynamic quadrupole operated by a combination of DC and RF voltages. It is constructed of four electrically-conducting, cylindrical rods accurately held in place by a set of two high-purity alumina insulators.

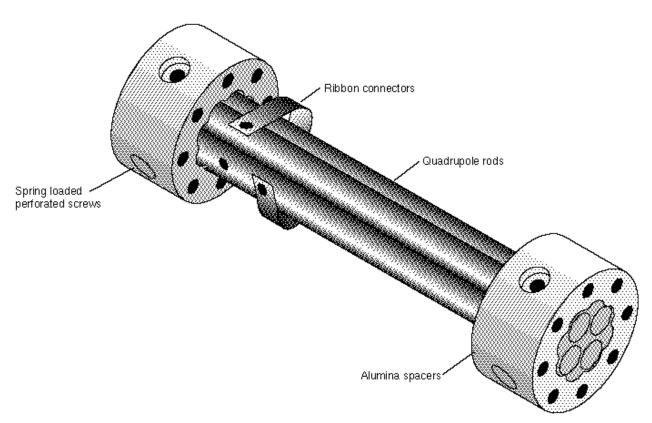


Figure 4 Quadrupole Mass Filter Components

Round electrodes are a common replacement for hyperbolic rods in modern quadrupoles and result in reduced cost and ease of construction. The stainless steel (type 304) rods are 4.5" long, have a 0.250" diameter (100  $\mu$ in tolerance) and are held in quadrature and parallel to each other, to tolerances better than 300  $\mu$ in, by the precision ground alumina spacers. Spring loaded, perforated screws fasten the rods to the spacers. The whole assembly is self-aligning, can easily be taken apart for cleaning and it can withstand repeated cycling to 350°C for bakeout. The exact placement of the rods is optimized to minimize the distortion of the electrodynamic quadrupole field by canceling higher order (multipole) potential terms. The radius of the circle inscribed by the rods is 0.109". The frequency of operation is f=2.7648 MHz.

#### **Principle of operation**

The following figure schematically represents the quadrupole mass filter and its connections.

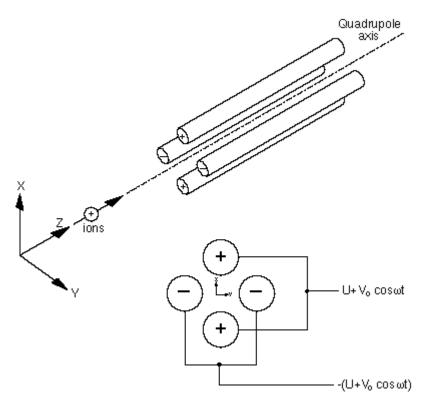


Figure 5 Quadrupole Connections

During operation, a two dimensional (X-Y) quadrupole field is established between the four cylindrical electrodes with the two opposite rods connected together electrically. Ions enter the filter along the Z-axis and start oscillating in the X- and Y- directions. The ions are separated based on their mass-to-charge ratio by lateral forces resulting from the electric potentials applied to the rods. A combination of DC and RF voltages control the quadrupole filter operation. Successful separation of a specific mass species requires setting the RF and DC values such that only the ions of interest have stable trajectories down the quadrupole rod assembly. By simultaneously varying the amplitude of the DC and RF voltages an entire mass spectrum can be scanned.

The CIS Analyzer is completely calibrated at the factory so that the ECU automatically programs the RF and DC voltages on the rods to the necessary levels during scans or individual mass measurements. The calibration parameters can be accessed and modified by the user in case recalibration or a change in resolution is required. (Please see the Tuning chapter for details.)

The quadrupole mass filter must be operated in a vacuum. Linear operation can be expected up to pressures of the order of  $10^{-4}$  Torr. In general quadrupoles can be operated at relatively high pressures compared to other types of analyzers. The upper limit of useful operation is determined by the collisions between the ions and the neutral

gas molecules. In order to avoid collisional scattering it is necessary to maximize the mean free path of the ions. The 100X pressure reduction between the CIS Ionizer and the quadrupole assembly is what makes the quadrupole filter compatible with ionizer pressures as large as 10 mTorr.

The general principle of operation of the filter can be visualized **qualitatively** in the following terms: One rod pair (X-Z plane) is connected to a positive DC voltage upon which a sinusoidal RF voltage is superimposed. The other rod pair (Y-Z plane) is connected to a negative DC voltage upon which a sinusoidal RF voltage is superimposed, 180 degrees out of phase with the RF voltage of the first set of rods. The potentials are represented by the expression:

 $V_{x/y} = +/- (U + V_0 \cos \omega t)$ where,

U is the magnitude of the DC voltage applied to either pair of rods,  $V_0$  is the amplitude of the RF voltage applied to either set of rods, and  $\omega$  is the angular frequency (=2 $\pi$ f) of the RF.

Light ions (low mass-to-charge ratio) are able to follow the alternating component of the field. For the **X direction**, those ions will stay in phase with the RF drive, gain energy from the field and oscillate with increasingly large amplitudes until they encounter one of the rods and are discharged. Therefore, the X direction is a **high-pass mass filter**: Only high masses will be transmitted to the other end of the quadrupole without striking the X electrodes. On the other hand, in the **Y direction**, heavy ions will be unstable because of the defocusing effect of the DC component, but some lighter ions will be stabilized by the alternating component if its magnitude and amplitude are such as to correct the trajectory whenever its amplitude tends to increase. Thus, the Y direction is a **low-pass mass filter**: Only low masses will be transmitted to the other end of the quadrupole without striking the Y electrodes.

The two directions together give a **mass filter suitable for mass analysis**: By a suitable choice of RF/DC ratio the filter can be made to discriminate against both high and low mass ions to the desired degree.

The RF voltage magnitude and frequency determine the mass of the ions that undergo stable trajectories down the filter. As the RF amplitude increases, heavier ions start to oscillate in phase with the RF and collide with the rods. The DC/RF ratio determines the filter selectivity As the DC increases (at constant RF) heavier ions are defocused by the negative DC component.

The attractive features of the quadrupole as a mass analyzer are evident from the above discussion: The quadrupole provides a convenient filter which can be tuned to the desired mass by varying the amplitude of the RF voltage; the mass selectivity (i.e. resolution) can also be varied electronically by simply adjusting the DC/RF ratio. Simultaneously varying the amplitude of the DC and RF voltages allows entire mass spectra to be scanned.

#### Mass Range, Resolution and Throughput

The most important characteristics of a quadrupole are the **mass range**, its **ultimate resolution** and its **throughput** characteristics.

The **mass range** is the range of masses defined by the lightest and the heaviest singly charged ions which can be detected by the mass spectrometer. The SRS CIS Analyzer is offered in three different models with mass ranges of 1 to 100 (CIS100), 1 to 200 amu (CIS 200) and 1 to 300 amu (CIS300). The main difference between the three models is given by the maximum supply voltage available to the rods.

The terms **Resolution** and **Resolving Power** are often used by mass spectroscopists to express the ability of a mass filter to **resolve** ions having different masses:

**Resolution, or Absolute Resolution** =  $\Delta M_{10\%}$ : The width  $\Delta M$  of the pass band of the filter, defined as the full width at which the ion current falls down to 10% of the maximum value. Units = amu.

**Resolving Power:**  $R = M/\Delta M_{10\%}$ : Ratio between a particular mass M and the resolution,  $\Delta M_{10\%}$ , at that mass. Dimensionless ratio.

It is common practice in modern quadrupole based partial pressure analyzers to keep  $\Delta M_{10\%}$  constant at a value which insures adequate separation of masses that are 1 amu apart. The quadrupole in the SRS CIS Analyzer operates in that "Constant Resolution" or "Constant  $\Delta m$ " mode.  $\Delta M_{10\%}$  is preset at the factory to 1 amu, but it can easily be adjusted by the user all the way to the theoretical ultimate resolution of the filter.

The following figure illustrates the resolution concepts explained above, using a Krypton mass spectrum as an example. The different isotopes of the gas are well separated from each other, and a 1 amu peak width (=  $\Delta m_{10\%}$ ) is measured on the <sup>86</sup>Kr peak at 10% of its peak height. Also notice the 20% valley between the <sup>82</sup>Kr and <sup>83</sup>Kr peaks.

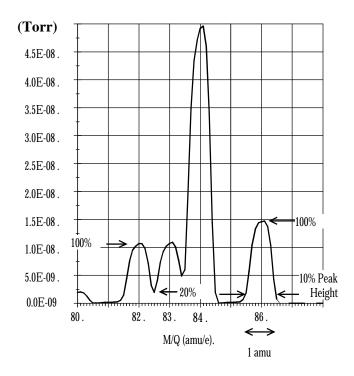


Figure 6 Peak Width Measurement

It is well established that the resolution attainable by a quadrupole is limited by the number of cycles of RF field to which the ions are exposed before they reach the detector. In practice, the minimum resolution ( $\Delta M_{10\%}$ ) value attainable is mass independent, linearly related to the ion energy, and inversely proportional to the square of the product of the quadrupole length and frequency. The two available ion energy settings of the CIS Analyzer correspond to **ultimate resolution** values of approximately 0.2 amu (4 eV) and 0.3 amu (8 eV) which are well under the factory default setting, and more than adequate to separate ions which differ in mass by 1 amu or less.

The resolving power, R, at a mass M is strictly related to the DC/RF voltage ratio. An increase in the resolving power usually results in a decrease in the effective **throughput** of the filter As the DC/RF ratio is increased the amplitude of the ion oscillations within the filter increase and a greater fraction of the ions are lost to collisions with the analyzer rods. The throughput of the quadrupole affects the overall **sensitivity** of the spectrometer to the mass being filtered. The exact relationship between resolution and sensitivity is very complex as it depends on the concentration and divergence of the ion beam leaving the source. It is complicated further by the defocusing action of the fringing fields between the ion source and the rods. However, as a general rule of thumb **Sensitivity decreases at 1 to 1.5 times the rate of resolving power increase.** 

The inverse relationship between sensitivity and resolving power is also responsible for a very important feature of quadrupole mass filters operated at constant resolution: **The throughput of the analyzer decreases with mass.** (Since  $\Delta M_{10\%}$  is constant, the resolving power, R= M/  $\Delta M_{10\%}$ , increases with mass reducing the effective throughput of the filter for high masses. This effect must be taken into account when calculating partial pressure sensitivities from ion currents.)

#### **Zero Blast Suppression**

When the applied potentials are small(or zero) as at the beginning of a scan, ions entering the filter may be transmitted even though their trajectories are mathematically unstable, just because of the weakness of the fields and the finite length of the filter. This gives rise to an output signal at the beginning of mass scans called the "Zero Blast". In the SRS CIS Analyzer the Zero Blast is suppressed preventing the DC voltage from reaching zero under 1.5 amu.

### **Ion Detector**

Positive ions that successfully pass through the quadrupole are focused towards the detector by an exit aperture held at ground potential. The detector measures the ion currents directly (Faraday Cup) or, using an optional electron multiplier detector, measures an electron current proportional to the ion current.

#### Description

The following figure describes the detector assembly including the electron multiplier option.

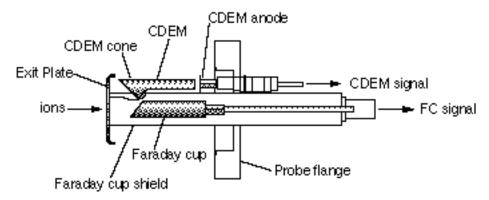


Figure 7 Detector Components

The **Faraday Cup** (**FC**) is a small stainless steel (type 304) metal bucket located on-axis at the end of the quadrupole. It is shielded from the intense RF and DC fields of the quadrupole by the grounded **exit plate**, which also provides some focusing of the ions into the detector. A cylindrical tube (**FC Shield**) encloses the FC, protecting it from the strong electrodynamic potentials of the adjacent rods and from collecting ions originated at sources other than the ionizer. The signal is carried to the electrometer through a coaxial feedthru which extends into the ECU box and efficiently shields the small ion currents. The FC is attached to the center feedthru with a push-on connector and can easily be pulled out for cleaning or modification.

Notice that an electron suppressor electrode (commonly found in several commercially available quadrupole mass spectrometers) is not part of this detection setup since the large aspect ratio (length-to-diameter) of the FC assures efficient recapture of secondary electrons.

The electron multiplier is a state-of-the-art Macro Multi-Channel Continuous Dynode Electron Multiplier (CDEM). It consists of a straight, four-channel tube made out of a special resistive glass (i.e. high secondary electron emission yield) with a cone of the same material attached to the front end. It is placed upright, next to the FC, and away from the axis of the analyzer (off-axis configuration). It is held safely in position by a clamp and a clip. The clamp is connected to the neighboring rod that carries the voltage from the High Voltage power supply, and is responsible for biasing the cone as well as assuring its correct placement next to the FC. A hole on the side of the FC Shield allows space for mounting the CDEM cone very close to the top of the FC. The clip

anchors the CDEM glass tube to the side of the FC Shield and holds the lower end of the tube at ground. Chrome electrical coatings, deposited at both ends of the tube provide the necessary electrical contacts. A plate (CDEM Anode) mounted at the exit of the CDEM collects the secondary electrons. The resulting electron current flows into the electrometer through a separate feedthru of the flange.

The entire setup is **self -aligning** and easily serviced by the user in the field. For example, removing the screw that fastens the clamp to the HV rod is all that is needed to replace the CDEM (Please see the Maintenance chapter for details.)

**FC detection is standard in all CIS probes**. All FC-only units can be upgraded to electron multiplier detection without the need to replace the probe (See the Upgrading to an Electron Multiplier Detector subsection below for details). When the electron multiplier is not turned on, multiplier systems operate exactly like the FC-only systems. The same electrometer is used to measure the ion (or electron) currents under both detection schemes. The ECU automatically connects the necessary electrode to the electrometer depending on the type of detector being used.

#### **Faraday Cup operation**

The Faraday Cup (FC) detector, measures the incident ion current directly. Positive ions enter the grounded detector, strike a metal wall, and are neutralized by electron transfer from the metal to the ion. The electrons given up in this process establish an electrical current that has the same intensity as the incoming ion current.

Since the nominal sensitivity of the CIS Analyzer in the CIS Mode is in the order of  $10^{-6}$  Amps/Torr, the currents measured are very small:  $10^{-9}$  to  $10^{-14}$  Amps for pressures in the order of  $10^{-3}$  to  $10^{-8}$  Torr, respectively. Minimum-detectable-partial pressures as low as  $10^{-9}$  Torr are possible with the FC in the RGA Mode. However, in real-time applications, the FC detector is rarely used to measure partial pressures below  $10^{-7}$  Torr in the CIS Analyzer. At pressures below that value, the CDEM option becomes a much better alternative allowing for faster scan rates and improved signal-to-noise ratios. The best characteristics of FC detection are: simplicity, stability, large dynamic range and lack of mass discrimination. All ions are detected with the same efficiency regardless of their mass.

The ECU automatically connects the FC electrode to the electrometer input upon poweron reset, and whenever the electron multiplier is not turned on.

#### **Electron Multiplier Operation**

An Electron Multiplier is recommended for all CIS Analyzers, particularly when subppm detectability is required from the instrument or when pressures under 10<sup>-7</sup> Torr are routinely sampled. **Macro Multi-Channel Continuous Dynode Electron Multipliers** are used in the SRS CIS Analyzers fitted with the electron multiplier option (Option 01). They consist of a straight four-channel tube made out of a special resistive glass (i.e. high secondary electron emission yield) with a cone of the same material attached to the front end. The extruded channels run along the length of the device and are twisted at the time the part is drawn to eliminate ion feedback. Each CDEM is placed upright next to the FC and away from the line of sight of the ionizer (i.e. off-axis configuration). When the cone is biased negatively (-1000 to -2500V) relative to the back end, positive ions are very efficiently attracted away from the FC and strike the cone at high velocity producing electrons by secondary electron emission. The secondary electrons are subsequently accelerated down the four channels and produce more secondary electrons. For each ion entering the cone of the CDEM, and depending on the bias voltage applied, up to 10<sup>7</sup> electrons come out at the back end and are picked up by a grounded plate (the CDEM anode). The resulting electron current is proportional to the ion current and is measured by the same logarithmic electrometer used in the FC measurements. The sign of the electron multiplier currents is reversed before the current value is sent out over RS232 so that the computer does not need to do any sign flipping on the currents received when the CDEM is activated.

The gain of the electron multiplier is a function of the bias voltage and is measured relative to the FC signal. The following figure shows a typical "gain characteristic" (i.e. gain vs bias voltage) curve obtained for  $H_2O^+$  ions at 18 amu.

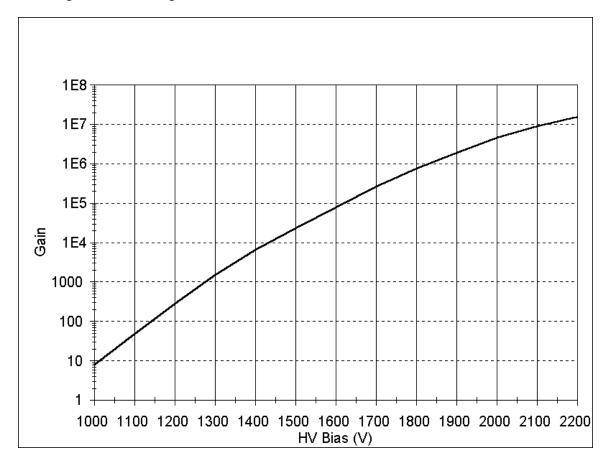


Figure 8 Typical CDEM Gain Characteristic Curve

Very high gain values can be obtained and controlled with negative voltages smaller than 2500 Volts in magnitude. **The sensitivity of the spectrometer increases with the gain of the electron multiplier**. Higher sensitivities provide lower minimum detectable partial pressures and faster spectral scans. Minimum detectable partial pressure limits lower than 10<sup>-11</sup> Torr are possible in the RGA Mode of operation. However, the increase in sensitivity is obtained at the price of limited dynamic range, mass discrimination

effects, gain instabilities and finite lifetime of the device. A good understanding of these limitations is very important to assure accurate quantitative measurements.

The **dynamic range** of electron multipliers is determined by their dark current at the low end, and by the bias current value at the high end.

The **bias current** is established by the external voltage drop along the resistive glass tube and flows along the channel walls replenishing their charge as secondary electrons are emitted. **Channel electron multipliers, operate linearly in the analog mode until the output current is approximately 10% of the bias current.** 

The **dark current** of a multiplier is the electron current measured at its output in the absence of an input ion current. **The minimum output current that can be accurately measured with the multiplier is equal to the dark current noise.** Example: For a typical resistance of 200 MOhms, and a bias voltage of -2000V, the bias current is 10  $\mu$ Amps and the output current must be kept under 1  $\mu$ Amp. Since the gain at that voltage is roughly 10<sup>6</sup>, the maximum input current at which the output current behaves linearly is 10<sup>-12</sup> Amps (1  $\mu$ Amp / 10<sup>6</sup>). Typical dark currents are lower than 10<sup>-13</sup> Amp, and the minimum input current that can be detected is 10<sup>-19</sup> Amps. For a sensitivity of 10<sup>-6</sup> Amp/Torr, this corresponds to an lower and upper limits of 10<sup>-13</sup> and 10<sup>-6</sup> Torr, respectively, and 7 orders of magnitude of dynamic range.

The total gain of electron multipliers varies as a function of the mass of the incident ions. As a rule of thumb, and for small molecules, **the gain decreases as mass increases**. This mass discrimination effect is caused by the dependence of ion-electron conversion efficiencies on the velocities of the ions entering the detector. For example, an inverse relationship with the square root of the mass has been reported for monoatomic ions of the same energy. For accurate quantitative measurements, it is essential to calibrate in advance the gain of the multiplier for the specific ionic species being detected.

An important problem when working with multipliers is that their gain changes with time. **Gain degradation** is unavoidable, and particularly serious just after the detector has been exposed to air, or after high quantities of reactive gases have been introduced into the vacuum system. The increased surface area provided by the extra channels in the multi-channel devices reduces this problem; however, frequent calibration of the multiplier gain against the FC output is recommended for reliable quantitative measurements. This is done automatically with the RGA Windows<sup>™</sup> software.

Gain degradation limits the **lifetime** of all electron multipliers. Eventually the gain drops to unacceptable values and the multiplier needs to be replaced. As a rule of thumb, **the multiplier should be replaced when the required gains can no longer be achieved by increasing the bias voltage.** The lifetime of electron multipliers is ultimately dependent upon the accumulated charge drawn from the multiplier (**Gain degradation typically starts at accumulated output current values of a few thousand µAmp-hr**). However, the lifetime also depends critically on the gas environment and the duration of transient signals. Contamination by organic compounds (i.e. diffusion or mechanical pump oil) and the interaction with highly reactive gases must be avoided at all times.

It has been found that, in many cases, channel multipliers may be successfully refreshed

by cleaning them in high purity isopropyl alcohol. The procedure is described in the Maintenance chapter (CDEM Refreshment section) and, even though **it is not guaranteed to always work**, it is worth trying as a last resort before discarding a multiplier.

Channel electron multipliers have a history of high performance and dependability in mass spectrometry applications. However, in order to achieve maximum useful lifetime and optimum performance, it is very important to handle them very carefully. Please read the **CDEM Handling and Care** section in the **Maintenance** chapter of this manual to familiarize yourself with some of the basic procedures that must be followed for the correct operation of the multipliers.

#### Upgrading to an Electron Multiplier Detector

An Electron Multiplier upgrade (Option 01) to the standard Faraday Cup (FC) detection setup is highly recommended for all CIS Analyzers, particularly when sub-PPM detectability is expected from the instrument or when pressures under 10<sup>-7</sup> Torr are routinely sampled. The detector upgrade consists of a state-of-the art Macro Multi-Channel Continuous Dynode Electron Multiplier (CDEM), and a negative high voltage power supply (0 to -2500V).

The advantages of operating the CIS Analyzer with a CDEM detector are more easily illustrated through examples:

Example #1: In the **RGA mode** of operation, the best detection limits that can be expected from a CIS Analyzer operated with a FC detector are in the order of  $10^{-9}$  Torr. Modern day pumping systems (with dry pumped turbo-pumps) can easily evacuate chambers down to the  $10^{-8}$  Torr level making it impossible for the CIS Analyzer to provide a detailed partial pressure analysis of the residual gas with the FC detector. Operation of the electron multiplier at gain levels above 100X, and under the same pressure conditions, brings the detection limits under  $10^{-11}$  Torr providing a cleaner baseline better suited for the analysis of the residual gas composition, and the detection of small leaks.

Example #2: A common application of the CIS Analyzer in the <u>CIS Mode</u> of operation is to monitor trace impurities in a gas phase process. The ionizer is typically exposed to the high purity gas in the mTorr range and sub-ppm ( $<10^{-9}$  Torr) impurities are often monitored. Partial pressure detection limits for FC detection in the CIS Mode are in the order of  $10^{-8}$  Torr. That corresponds to a 10ppm detection limit for common impurities. With the help of the CDEM gain, the partial pressure detection limit can be reduced down to  $10^{-10}$  Torr or better, which brings the detection limits down to the sub-ppm levels that are often required.

Example #3: CDEM detection can dramatically increase data throughput when speed is an important issue in the partial pressure analysis. There are two fundamental ways to increase the signal to noise ratio of any measurement. One way is to reduce the baseline noise while keeping the signal at the same level. Another way is to amplify the signal while keeping the baseline the same. Scanning at a lower scan speed, provides increased averaging and a reduction of the baseline noise while keeping the signal levels untouched. However, it also slows down the measurements and can result in a reduced data throughput that might not be compatible with the required data acquisition rates. A better alternative is to turn on the CDEM and adjust its gain so as to enhance the signal (not the noise) while keeping the scan speed the same (or even faster). The CDEM is a very quiet (i.e.low noise) amplifier and only starts to contribute to the baseline noise at its higher gains.

Electron Multiplier upgrades are the most frequent type of hardware enhancement performed by SRS on its entire line of quadrupole mass spectrometers. Contact your local representative or Stanford Research Systems for details and pricing.

### **Pumping System Requirements**

The **CIS Probe** consists of a quadrupole mass spectrometer equipped with a high conductance, differentially pumped, CIS ionizer mounted inside a 2.75" Conflat Tee (CIS Cover Tee). The side port of the CIS Cover Tee (i.e. Pumping System Port) provides the connection for the **Pumping System** required to differentially pump the CIS ionizer and keep the quadrupole mass analyzer, filament and detector at high vacuum during process monitoring (see Figure 1 for details).

The ionizer apertures were designed such that, during normal operation, the pressure in the quadrupole volume can be at least 100x smaller than the pressure in the ionization region. Under these conditions, a  $10^{-3}$  Torr process pressure corresponds to a "comfortable"  $10^{-5}$  Torr pressure on the filament, quadrupole and detector.

Oil-free, turbo-pump based stations, compatible with the CIS Analyzer, are available directly from Stanford Research Systems (see for example, Option O100TDP). When a Pumping System option is ordered along with the CIS Analyzer, the entire analyzer consisting of the quadrupole mass spectrometer and its pumps, is assembled, tested and calibrated at the factory before being shipped. The turbomolecular pump becomes an integral component of the CIS Head, and the stored sensitivity factors are strictly valid when the CIS Analyzer is attached to a chamber. Please consult SRS or you local representative for information on the available pumping options.

Users who already own a pumping station, or who are putting together a custom or very specialized system might want to select their own pumps for the CIS Analyzer. The following paragraphs are designed to aid those users in the selection of a pumping system that is best suited for the CIS Analyzer. It is the user's responsibility to make sure that the pumping system ultimately chosen does not damage or limit the performance of the instrument.

For general information on vacuum system design and pump options consult :

- 1. Gerhard Lewin, "An Elementary Introduction To Vacuum Technique:, published by the Education Committee of the American Vacuum Society, 1987.
- 2. J. H. More et. al., "Building Scientific Apparatus", Chapter 3, Addison Wesley Publishing Co, Redwood City, CA., 1988.

#### **Pump Options**

*Turbo pump based systems are, by far, the best choice for quadrupole mass spectrometers.* They are reasonably priced and provide the largest list of desirable features: Small dimensions, high pumping speed, high compression ratio, low base pressure, minimal power and cooling requirements, short start up time, low noise, low contamination from backstreaming oils, low maintenance, and operation in any orientation are some of their advantages when compared to other options. Most turbo pumps operate at full pumping speed at inlet pressures above 1 mTorr . Turbo pumps compatible with highly corrosive gases are also available. Increased water pumping speeds can generally be achieved interposing a cryogenic water trap between the CIS analyzer and the turbo pump.

Modern hybrid turbomolecular pumps offer increased compression ratios and can be backed by oil-free diaphragm pumps providing ideal oil-free operation. This is the approach followed by SRS for its pumping systems, and the one recommended by the factory for all CIS Analyzers.

A good quality oil trap must always be placed between a turbo pump and the foreline pump, whenever an oil-based mechanical pump is utilized. Contamination from oils backstreaming through the vacuum pipes must be minimized at all cost in the CIS Analyzer. Oil-free pumping systems provide the best protection.

*Diffusion pumps should not be used in combination with the CIS Analyzer.* Even the best trapped diffusion pumps backstream oil into their vacuum chambers and should never be operated in the absence of a cold trap or baffle. They generally do not provide the base pressures required by the CIS analyzer, they are bulky and can only be operated in an upright position. With a few exceptions, they all require water cooling, and they consume large amounts of power during operation. Start-up times are considerably longer than in turbo-pumps (i.e. 5 to 10 minutes). Their price advantage is offset by the problems mentioned above, and usually disappears by the time the addition of an oil trap and maintenance costs are added to the purchase price.

*Ion pumps and cryo-pumps* are alternative oil-free pumping systems for the CIS analyzer. However, they are generally more expensive and less versatile than turbopumps. Their use as primary pumps is only recommended in specialized applications (i.e. where turbo pumps might not be adequate) or as auxiliary pumps (i.e. the pumps that do the work at night while the system is at base pressure). Cryopumps, which usually offer very large pumping speeds for water, have been succesfully used to "clean" water backgrounds and enhance low-level water detection limits of many commercially available, closed-source mass spectrometers.

A typical upgrade to the Pumping System includes the addition of a total pressure gauge dedicated to monitor the pressure on the quadrupole rods. Additional vacuum hardware is usually required to accommodate the extra gauge. Under normal operating conditions (i.e. gas pressure  $\approx 10^{-3}$  Torr in the process chamber, and a well matched pumping system) the pressure on the quadrupole rods should be approximately two decades lower than the chamber pressure (i.e.  $1-2\cdot10^{-5}$  Torr).

The following table is a compilation of the minimum requirements that the Pumping System must satisfy in order to be properly matched to the CIS Analyzer. Whenever applicable, a recommended spec is also provided.

Pumping System Requirements	Units	Minimum	Recommended
Pumping Speed (see Note 1)	Ls <sup>-1</sup>	40	70
Base pressure (see Note 2)	Torr	10 <sup>-9</sup>	10 <sup>-10</sup>
Inlet Flange	n.a.	CF 2.75" O.D.	CF 2.75" O.D.
Operating range (@ full pumping speed)	Torr	Base pressure to 10 <sup>-3</sup> Torr	Base pressure to 10 <sup>-3</sup> Torr
Start-up time	sec		<60
Operating position			any
Cooling requirements			Natural air convection or forced air
Forepump		Oil free	Oil Free

**Note1** The effective conductance at the ionizer's inlet (i.e. considering the length of tube and the two holes in series) is approximately 0.4 L.s<sup>-1</sup>. *This value indicates that a 100-fold pressure differential between the ionization volume and the quadrupole volume can be established simply connecting the CIS analyzer to a pumping station capable of delivering an effective pumping speed of at least 40 Ls<sup>-1</sup> at the filament and quadrupole rods.* These pumping speed levels can be accomplished with even some of the smallest turbomolecular pumps that are currently available. A large increase in effective pumping speed at the ionizer cannot be expected by increasing the size of the turbo pump above the recommended value of 70 Ls<sup>-1</sup>. Ultimately, the effective pumping speed is limited by the  $\approx$ 50 Ls<sup>-1</sup> conductance of the 1.5" OD piping that connects the CIS probe to the pump.

**Note 2** The CIS Analyzer is specifically designed for the direct measurement of minute trace gas impurities in process gases. Detection limits in the 100ppb level, corresponding to  $10^{-9}$  Torr impurities in a  $10^{-2}$  Torr process gas, are not unusual. Therefore, a  $10^{-9}$  Torr base pressure is a natural minimum requirement for the pumping system in order to minimize backgroung interferences in the mass spectra during ppm and sub-ppm measurements.

# Chapter 4 Electronics Control Unit

This chapter describes the most important features of the Electronics Control Unit of the CIS Analyzer.

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## Introduction

The SRS CIS Analyzer consists of a **CIS probe** and an Electronics Control Unit (**ECU**) which mounts directly on the probe's feedthru flange and contains all the electronics necessary to operate the instrument. The basic setup is shown in Figure 1.

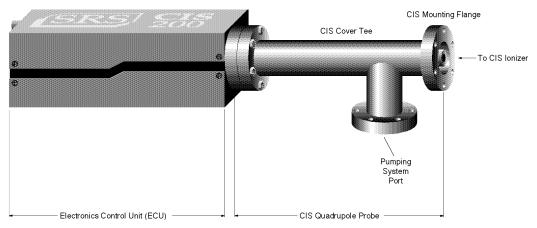


Figure 1 SRS CIS Analyzer components

The ECU is a densely packed box of electronics (3" x 4" x 9") that connects directly to the probe's feedthru-flange and also to a host computer. It includes several regulated power supplies, a built-in microprocessor, control firmware, and a standard RS232 communications port. It is powered by either an external 24VDC (@2.5Amps) power supply or an optional, built-in power module which plugs directly into an AC outlet.

#### Warning

The ECU does not have any serviceable parts and does not require any routine maintenance. All calibration procedures should be done using the RS232 interface and the CIS calibration command set.

During normal operation of the CIS Analyzer, regulated power supplies built into the ECU set the electron emission current, the voltage levels on the ionizer electrodes, and the high voltage across the electron multiplier. RF/DC levels for each mass are also set and regulated by the ECU, based on internal mass calibration parameters permanently stored in non-volatile memory. A built-in, logarithmic electrometer detects the ion currents collected by the FC, or electron multiplier, and converts them to voltage signals that are read by a built in A/D converter. The microprocessor automatically compares those signal voltages against an internal calibration and calculates the magnitude of the ion currents. The ion currents are then averaged as needed, offset corrected and transmitted to the host computer over RS232 for immediate display.

Several built-in checks constantly monitor the operation of the probe and its operating environment. For example, the filament emission, and the electron multiplier, are immediately turned off in the case of accidental overpressure.

Active LED's on the rear panel of the ECU provide constant feedback on the status of the

filament, electron multiplier, electronics system, probe and communications, and alert the user of any problems.

# **Front Panel**

The ECU mounts directly on the probe's feedthru flange. Its front panel is designed to rest flat against the back surface of the probe's flange, and it is not visible while the ECU is locked in place.

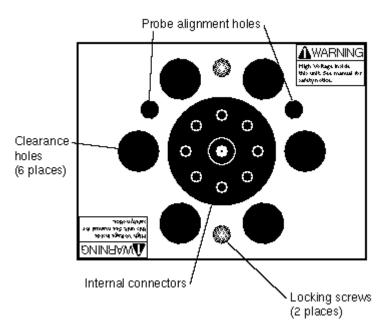


Figure 2 ECU Front Panel

The front panel of the ECU box has nine holes of three different sizes, and two locking screws.

The big hole in the center provides access to internal connectors that line up with the feedthru connectors of the probe. The six equally-spaced holes, surrounding the big hole, provide clearance for the six bolt heads on the back of the probe's feedthru flange. The two remaining holes (1/4" diameter) line up with the alignment rods of the probe and assure the correct alignment between the ECU and the Probe during installation. The locking screws line up with two threaded holes on the back of the feedthru flange, and are used to lock the ECU box in place.

### **Rear Panel**

The rear panel of the standard ECU Box has two connectors, two locking knobs, a cooling fan, and eight LED's.

Units with the optional, built-in power module (Option 02) also have a fused Power Entry Module with a built-in power switch.

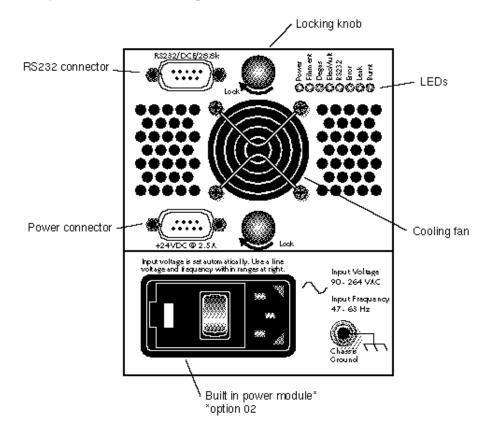
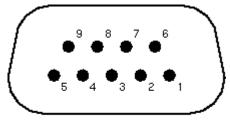


Figure 3 ECU Rear Panel

#### +24VDC@2.5A Connector

Use this connector to provide external power to the CIS Analyzer. The ECU electronics must be powered with a 24 V (+/- 2V) DC power supply @2.5Amps. The power supply must have a cable with a 9 pin, type D, female connector on the free end, wired as described in the "ECU- 24VDC power connector" diagram shown below.



Pin #	Voltage
1	+24 VDC
2	+24 VDC
3	Ground
4	Ground
5	Ground
6	+24 VDC
7	Ground
8	+24 VDC
9	Ground

Figure 4 ECU 24 VDC Power Connector

#### RS232/DCE/28.8k Connector

Use this connector to interface the CIS Analyzer to a computer. The RS232 interface connector of the CIS Analyzer is configured as a DCE (transmit on pin 3, receive on pin 2) with full RTS/CTS handshaking enabled. Use a straight through RS232 cable with 9-pin, type D connectors to connect the computer (usually DTE) to the ECU. The communication parameters are fixed at: 28,800 baud rate, 8 data bits, no parity, 2 stop bits.

#### LED's

Eight LED's provide constant feedback on the operation of the CIS Head. The function of the LED's is described later in this chapter.

#### Locking Knobs

Use the two knobs to lock the ECU box in place at the end of the ECU installation procedure. Do not overtighten the knobs. (Hand tighten only.)

#### Power entry module (Option 02)

Use this connector to power the CIS Head directly from an AC outlet. Use the three-wire power cord provided by SRS to connect the instrument directly to a properly grounded AC outlet. Use the built in switch to turn the unit on-off.

Refer to the first page of this manual for instructions on selecting the correct line voltage and fuse. The built-in power module (Option 02), has universal input (90-264 VAC, 47-63 Hz), and completely eliminates the need for an external 24VDC power supply connection.

#### **LED Functionality**

LED's on the rear panel of the ECU provide constant feedback on the status of the filament, electron multiplier, electronics system, probe and communications, and alert the user of any detected errors. This section describes in detail the function of each LED.

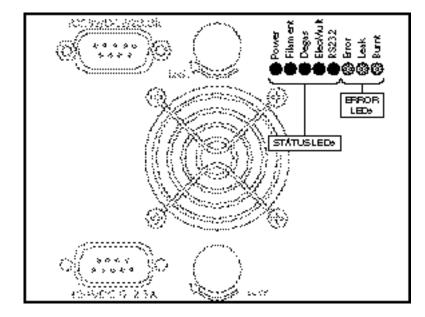


Figure 5 ECU LED's

#### STATUS (Green) LED's

**Power**: The Power LED is turned on whenever the CIS Analyzer is successfully powered up. When power is applied to the ECU, a firmware routine automatically checks the external voltage level and turns on the Power LED if the voltage is within the acceptable range of  $24 \pm 2$  V DC. The same test is performed in units with built-in power modules (Option 02) to check the voltage output of the internal switching power supply. If the check fails, the red Error LED is turned on instead to indicate the problem.

Note: A check on the external power supply can be performed at any time during operation using the diagnostic command EP?.

**Filament**: The Filament LED indicates the presence of electron emission current in the CIS ionizer. It is turned on when a finite emission current is requested by the user. It is turned off when the user requests a null emission current or when a filament error condition (i.e. overpressure, worn out or burnt filament) is detected and the filament emission is turned off.

Note: Filament error conditions are easily diagnosed with the help of the Leak and Burnt LED and the assortment of diagnostic checks built into the firmware and supported by the RGA Windows program.

Degas: The Degas LED is not used by the CIS Analyzer.

ElecMult: The ElecMult LED is turned on whenever the electron multiplier detector is

active (i.e. when a finite biasing voltage is applied across the electron multiplier). Several different mechanisms can turn off the electron multiplier and its LED: a null bias voltage request by the user, and an overpressure that shuts down the filament emission.

RS232: The RS232 LED reflects the activity on the RS232 Transmit and Receive lines.

#### ERROR (Red) LED's

**Error**: The Error LED indicates the presence of errors in the operation of the CIS Analyzer. Errors can originate from many different sources including hardware, communications, probe (i.e. bad filament) and operating environment (i.e. overpressure). Troubleshooting is simplified by an assortment of diagnostic commands built into the firmware, and fully supported by the RGA Windows program.

**Leak**: The Leak LED is automatically turned on whenever the filament heater is unable to establish a requested emission current. The most common reason for this problem is a serious leak in the vacuum system. In the event of an overpressure detection, the filament emission and the electron multiplier are immediately turned off, and the Error and Leak LED's are turned on to indicate the problem.

<u>Important:</u> The Leak LED will also be turned on if the filament is excessively worn down or damaged. However, this is a more rare event due to the long life of the filaments.

**Burnt**: The Burnt LED is turned on whenever the ECU fails to detect a filament. A burnt or missing filament are possible sources for this problem. The Error LED is also turned on to clearly signal the presence of a filament error.

### Electrometer

#### Detection limit vs. scan rate

A unique, temperature-compensated, logarithmic picoammeter built into the ECU box measures the ion currents collected by the Faraday cup (FC), or electron multiplier (CDEM). The output voltage of the electrometer is equal to the logarithm of the ion current so that several decades of signal can be read on the meter without any gain switching being necessary. The microprocessor automatically configures the electrometer and connects its input to the correct signal based on the type of detector being used.

The electrometer is completely autoranging and measures both positive and negative currents with the same accuracy and resolution. Its operating range covers current magnitudes between  $1.32 \cdot 10^{-7}$  and  $10^{-15}$  A.

The accuracy of the measurements is insured by an internal calibration procedure that calibrates the output of the electrometer against input current over its entire operating range, and stores a calibration table in the non-volatile memory of the CIS Head. *Instead of simply relying on the intrinsic logarithmic I-V behavior of the electrometer, a digital logarithmic interpolation algorithm calculates the currents from the calibration curve.* 

The I-V response of the electrometer can be recalibrated at any time through a single command (CL) supported by the RGA Windows software (<u>Calibrate Detector option in the Head menu</u>), making it possible to generate accurate readings under different operating conditions.

Excellent resolution is achieved using a 16 bit A/D converter to digitize the output of the electrometer. The bandwidth and detection limit of the logarithmic electrometer are fully programmable. This programmability is achieved biasing the electrometer with extra current from a microprocessor-controlled current source. The biasing current is added to the ion current and sets the gain and bandwidth of the electrometer during measurements. An increase in the biasing current results in lower I-V gain and larger bandwidth, but also means increased baseline noise due to the additional shot noise introduced by the extra current. During regular mass scans the CIS Analyzer automatically adjusts the scanning rate and averaging to match the bandwidth of the detector. The microprocessor calculates the total current flowing into the meter using the interpolation algorithm described above, and subtracts the biasing current from the total to provide the actual ion current values used for averaging. Obviously, the amount of biasing current must be chosen keeping in mind the strong interplay between bandwidth (i.e. acquisition rates) and baseline noise (i.e. minimum detectable partial pressures).

The single command NF (<u>Noise Floor</u>), supported by the RGA Windows program, is used to program the electrometer's response. Histogram scans, analog scans and singlemass measurements all share the same NF setting during measurements. A decrease in the Noise-Floor setting reduces the biasing current resulting in longer measurement times with cleaner baselines and lower detection limits. As mentioned above, the NF parameter value must be chosen keeping in mind the compromise between detection limit and acquisition speed.

When using the RGA Windows program to operate the instrument, the Scan Speed

parameter setting available in the Scan Parameter Setups of the Scan menu is used to set the NF parameter value in the CIS Head according to the equation: NF = ScanSpeed - 1.

The following table summarizes the performance of the electrometer during mass measurements as a function of the Scan Speed and NF settings. Please refer to this table to estimate minimum detectable partial pressures and scan rates for different scanning conditions.

Scan Speed	NF	Scan rate	Single mass meas.	Baseline noise <sup>(a)</sup>
parameter	parameter	(msec/amu)	time (msec)	(std.dev.)(Amps)
1	0	2000	2200	7e-15
2	1	1000	1100	1e-14
3	2	400	440	1.5e-14
4	3	200	220	2e-14
5	4	126	139	4e-14
6	5	45	50	1.2e-13
7	6	30	33	2.5e-13
8	7	15	16.5	5e-13

(a) The values in this column are the result of averaging a large number of data points from several different units. Use this values *for reference only*. Variations are to be expected from unit-to-unit, as a function of operating conditions and even as a function of time.

#### Adjusting the Zero of the Ion Detector

The zero of the ion detector is automatically readjusted at the beginning of each analog and histogram scan so that the baseline is always centered around zero. The zero can also be readjusted at any time with the command CA. In both zeroing procedures, the output of the electrometer is measured in the absence of input ion current and stored, as a current value, in the instrument's memory. The current value (called offset correction factor) is then automatically used by the firmware to offset-correct all ion currents measured under the same detector settings. The detector settings are the electrometer's noise floor parameter value and the type of detector (i.e. FC or CDEM) in use at the time the zeroing is performed. Offset correction factors for all the possible combinations of detector settings can be generated and accumulated in the head's memory. However, all offset correction factors are cleared after a recalibration (CL) of the electrometer is performed, and when the unit is turned off.

Please consult the CIS Command Set section for details on the CL and CA commands.

### **Mass Filter Power Supply**

All the necessary electronics required to power up the quadrupole mass filter during mass measurements are built into the ECU box. The RF/DC levels for each mass are set and regulated from the ECU, under microprocessor control, and based on internal calibration parameters permanently stored in non-volatile memory. The difference between the three CIS Analyzer models (CIS100, CIS200 and CIS300) is given by the maximum supply voltage available to the rods. For example, the maximum RF amplitude delivered to the probe in the CIS300 is 1284 V (2568 Vpp) and corresponds to ions with mass of 300 amu (8.56 Vpp/amu). The corresponding DC potential is a maximum of about 430 VDC for masses of 300 amu (+215 Vdc on one rod pair, and -215 Vdc on the other).

The frequency of operation of the mass filter is the same for all models and is fixed at 2.7648 MHz by an internal clock.

The rod structures in the mass filter are primarily capacitive, so to reduce the circuit drive requirements, the rod reactance is resonated with an inductance. The advantages of this resonant drive circuit is the primary reason for operation at a fixed frequency.

**Important:** The CIS Cover Tee (i.e. stainless steel tube) that encloses the quadrupole filter assembly contributes to the overall capacitance of the rod assembly, and should not be removed from the probe. Failure to operate with the CIS Cover Tee in place will push the driving circuit out of resonance, and will limit the operating mass range of the filter.

The internal calibration parameters used by the microprocessor to set the RF/DC levels during scans and measurements, are pre-programmed at the factory as part of the test and calibration procedures performed on the instrument before shipping. However, the parameters can be readjusted easily and whenever necessary using the Peak Tuning procedures described in the Tuning chapter.

# Maintenance and Service

- The ECU box does not have any serviceable parts and does not require any routine maintenance.
- Do not perform any unauthorized service, adjustment or modification of the instrument.
- Do not install any substitute parts.
- Contact the factory for instructions on how to return the instrument for authorized service and adjustment.

# Chapter 5 Programming the CIS Head

This chapter describes how to program the CIS Head from a host computer using the CIS Command Set and an RS232 Link.

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### Introduction

The CIS Analyzer comes standard with an RS232 communications port. A host computer interfaced to the instrument can easily configure, calibrate, diagnose and operate the quadrupole mass spectrometer using simple ASCII commands. The CIS head executes the commands in the order received and, when information is requested, data is quickly returned to the computer for analysis and display.

The fast exchange of data between the host computer and the CIS Analyzer provides dynamic information about the status of a vacuum process or system. The Command Set facilitates integration of all the spectrometer functions into any processing or diagnostic program.

# The RGA COM Utility

#### Intoduction

The RGA Com Utility is a simple Windows<sup>™</sup> OS communication program that allows you to communicate with the CIS Head directly by typing valid CIS commands on your keyboard. The program functions like any common terminal program where the typed characters are sent directly to the serial communications port, and any received characters are displayed immediately on the screen.

The RS-232 communication parameters of RGA Com are fixed to be compatible with the CIS Head. The only variable that may be selected is the communications port. A list of up to nine COM ports is available to connect to the CIS Head. RGA Com Fixed parameters:

- 28.8k Baud rate
- 8 Data bits
- No Parity
- 2 Stop Bits

#### Communicating with a Head

To connect to an SRS CIS Head do the following:

- 1. Start the RGA Com program.
- 2. Select the "Select Com Port..." command from the "COM port Setup" menu.
- 3. Select the Com port you want (usually COM2) from the list box and press OK.
- 4. Select the "Connect" command from the "COM port Setup" menu.
- 5. Begin typing commands, press "Enter" after each command to validate it.
- 6. When you are done select the "Disconnect" command and exit the program

#### Tip

• When you first connect to the head (step 5 above), send the ID? command to verify the connection with the CIS Head. This command will return the Model number, Serial number , and firmware version of the CIS Unit you are connected to.

#### Important

- If you make a typing error, the Error LED on the back plane of the ECU box will blink two or three times. Press "Enter" and retype the command
- The Backspace character is not processed by the CIS Head.
- When you are done using RGA Com you must disconnect the COM port or exit the program, otherwise no other Windows program can communicate with the open COM port.

# **RS232 Interface**

The RS232 interface connector of the CIS Analyzer is a standard 9 pin, type D, female connector configured as a **DCE** (transmit on pin 3, receive on pin 2) with full **RTS/CTS handshaking** enabled. The CTS signal (pin 8) is an output indicating that the CIS Unit is ready, while the RTS signal (pin 7) is an input that is used by the host computer to control the instrument's data transmission.

The communication parameters are fixed at: 28,800 baud rate, 8 databits, no parity, 2 stop bits.

**Cable connection:** Use a straight through RS232 cable with 9-pin, type D connectors to connect the computer (usually DTE) to the ECU. The DB9 to DB25 adapter (provided with the unit) will be needed if the computer has a 25 pin Type D connector.

**Important**: If you use your own RS232 cable check the connectors to make sure all the necessary handshaking lines are connected correctly.

# **LED Indicators**

To assist in programming, the CIS Head has two status LED's on the rear panel of the ECU box.

**RS232 LED:** RS232 Activity indicator that flashes whenever a character is received or transmitted over RS232.

**Error LED:** The Error LED flashes on/off a couple times when an error, such as an illegal command or parameter, has been detected.

### **Command Syntax**

The CIS commands are ASCII character strings consisting of a two letter (case insensitive) **command name**, a **parameter**, and a **carriage return** terminator.

**Note:** The carriage return character, decimal ASCII value=13, is represented throughout this manual with the symbol **<CR>**. All command strings must be terminated with this character in order to be acknowledged by the CIS Head.

#### Valid parameters are:

**Numbers**: Numbers are the most common type of parameter used to program the CIS Analyzer. Number parameters must be within a command-specific range and must not conflict with any pre-existing parameter values of the head. Only decimal format is accepted. The fractional parts are truncated to four decimal places before being stored in memory. The '+' sign is optional for positive numbers. A zero in front of the period is optional in fractional numbers with magnitude less than one.

**Asterisk "\*" (default**): A single asterisk indicates that the default parameter value (stored in the CIS head's permanent memory) is to be used for command execution. The asterisk must be strictly followed by a <CR>.

**Question mark "?"** (**Query**): Query commands are used to read error bytes and to confirm internal parameter values. The question mark must be strictly followed by a <CR> terminator. With a few exceptions, the data returned by most query commands is a string of ASCII characters terminated by a linefeed (<LF>, decimal ASCII value=10) and a <CR> terminator: string<LF><CR>.

No parameter: Some commands do not require a parameter.

The CIS Analyzer assumes a *potential* command-string has been received over RS232 when a <CR> character is detected in the serial interface. A **command handler** immediately analyzes the string, and if everything checks, the command is executed. However, if a problem is encountered a **communication error** is reported and no command execution takes place.

The CIS Analyzer has a 140 character input buffer and processes commands in the order received. If the buffer fills up, the instrument's processor holds off handshaking on the RS232 interface using the CTS line. Similarly, the CIS Head has a 32,000 character output buffer to store outputs until the host computer is ready to receive. If either buffer overflows, the entire buffer is cleared and the error is reported.

Linefeed characters (<LF>, decimal ASCII value=10) and single carriage returns (<CR>, decimal ASCII value=13)) transmitted from the host computer are ignored by the serial interface.

#### **Examples of command formats**

The following list of commands activates the ionizer and triggers a single analog scan from 1 to 100 amu at the default scan rate setting. Note that each one of the command

character strings must be strictly followed by a <CR> character in order to be detected by the spectrometer's command handler.

ID?	Identification Query.
MI1	Set initial scan mass to 1 amu.
MF100	Set final scan mass to 100 amu.
FL1.0	Turn on the filament to a 1.0 mA emission current.
NF*	Use default noise floor setting. (sets scan rate and averaging.)
AP?	Query the number of scan points to be received by the computer.
SC1	Trigger a single analog scan.

#### **Programming tips**

- Whenever possible, immediately follow a command that **sets** a parameter value with one that **queries** the parameter setting in the instrument's memory. For example, the second command in the list above should be followed by a MI?, and a number 1 should be echoed by the CIS Head.
- Whenever practical, check for errors at the end of command execution to make sure "everything went well".
- Even though the CIS head can store multiple commands in its receive buffer, it is highly recommended that the host computer wait for the response of a query before a new command (especially a new query) is transmitted to the CIS Analyzer. This simplifies the communications and also eliminates buffer overwrites.
- Commands that involve hardware control (such as turning the filament or the CDEM on) trigger diagnostic checks on the hardware as they are executed. Since the command execution time can be rather long, the CIS Head prompts the end of execution by sending back the value of the STATUS byte to the computer. Always check the value of the STATUS Byte returned for possible errors. In cases where a hardware error is reported retry the command one more time before declaring a hardware problem.
- With the exception of the ion current values, the data tramsmitted by the CIS Analyzer consist of ASCII character strings terminated by a <LF><CR> terminator. Ion signals are represented as integers in units of 10<sup>-16</sup> Amps, and transmitted directly in hex format (four byte integers, 2's complement format, Least Significant Byte first) for maximum data throughput.

### **Communication Errors**

Communication errors are signaled to the user flashing the Error LED a couple of times, setting Bit 0 of the STATUS error byte and setting the error-specific bits of the RS232\_ERR error byte .

Many different circumstances can result in a communication error being reported after a command string is received by the CIS Head. Some problems are detected early by the **command handler** and result in the command never being executed. Other errors are found later, during the actual command execution, and they result in the execution being immediately halted.

The following is a list of the communication errors that might be encountered during the analysis and execution of a command, including the specific ways in which they are reported:

#### **Command errors**

**Bad Command Name** : The first two characters in a command string must be letters (case insensitive) and must be part of the CIS Command Set, otherwise the Error LED is flashed, Bit 0 of RS232\_ERR and Bit 0 of STATUS are set, and no command is executed.

**Command-too-long error** : 14 characters are received over RS232 without a <CR> ever being detected. The Receive buffer is flushed, Bit 2 of RS232\_ERR and Bit 0 of STATUS are set, and no command is executed. *Note that the LED is not flashed for this error (only exception to the rule!)*.

#### **Parameter errors**

Bad-Parameter error: Many different possibilities lead to a bad-parameter error.

- The number parameter is out of the command's acceptable range.
- A "?" is not strictly followed by <CR> in a Query command.
- A "\*" is not strictly followed by <CR> in a Default command.
- A non-zero fractional part is found in a parameter that can only be an integer.
- No parameter is found in a command that strictly requires one.
- A parameter other than a "?" is found in a Query-only command.
- A default parameter value is used with a command that does not have one defined.

All Bad-Parameter errors are reported flashing the Error LED, and setting Bit 1 of RS232\_ERR and Bit 0 of STATUS. No command is executed.

**Parameter-conflict error**: When the parameter is in conflict with some other related parameter value which pre-existed in the CIS head, the Error LED is flashed, Bit 6 of RS232\_ERR and Bit 0 of STATUS are set and the command is not executed.

#### **Jumper Protection violation**

Some calibration related commands are subject to jumper protection. Jumper JP100 on the digital (i.e. top) board of the insrument's electronics box can be used to enable/disable some of the tuning features of the instrument. The jumper setting is checked before the command is executed and if calibration is disabled, the Error LED is

flashed, Bit 5 of RS232\_ERR and Bit 0 of STATUS are set, and the command is not executed. The jumper setting provides very solid protection against inadvertently tampering with important calibration parameters stored in the instrument's memory. Please refer to the "CIS Command Set" list to find out which calibration commands are jumper protected.

# **Troubleshooting Tools**

The communication interface includes a complete set of tools for troubleshooting communications between a host computer and the spectrometer's head during programming:

#### 1. Visual Clue

The first clue of a communication error can be obtained watching the Error LED while communicating with the head.

The ERROR\_LED is flashed on/off two times every time a communication error is detected.

#### 2. Error Queries

The presence of a communication error can also be detected queering the contents of the STATUS byte in the CIS head. The query command **ER**? returns the value of the STATUS Byte for analysis, and a communication error is present if Bit 0 of STATUS is set. The specific problem is determined querying the bits of RS232\_ERR byte with **EC**? which clears the RS232\_ERR byte and Bit 0 of STATUS at the end of its execution to provide a clean error-reporting slate.

Communication errors accumulate in the RS232\_ERR byte as they occur and the byte can only be cleared with the EC? Command or turning off the instrument.

**Note:** Please refer to the "Error Byte Definitions" section of this chapter for a complete listing of the error and status bytes of the CIS Analyzer. Also consult the "Error Reporting Commands" list for details on the **EC** and **ER** commands.

### **Programming the CIS Head**

This section describes the basic programming steps needed to configure, operate, and diagnose the CIS Analyzer. The emphasis is on general program implementation without going into specific details on the different commands that are mentioned.

Please consult the "CIS Command Set" section of this chapter to get more detailed information on the spectrometer's commands and their implementation.

#### Initializing the CIS Head

The CIS Head is ready to communicate a few seconds after it is turned on, right after all the built-in internal checks are performed. The **Initialization** commands are commonly used to test the quality of the RS232 interface, to reset the communication buffers, to scan the hardware for problems, and to reset the instrument to its default (factory preferred) parameter settings.

#### Initializing the CIS communications

The two commands most often used after connecting to the head are:

- ID? Identification Query. Echoes the ID string.
- IN0 Clear communication buffers. Echoes the STATUS Byte.

The ID? command is used to test the RS232 interface: The ID String, stored in the CIS Head, is returned to the computer and the integrity of the RS232 link is assured once the character string is received. The ID string also contains useful information such as model and serial number of the CIS head.

The INO command clears the RS232 buffers, runs a fresh set of tests on the ECU's hardware and sends back the STATUS Byte. Check the value of the STATUS byte for potential errors.

#### **Programming tips**

- Use the INO command every time there seems to be problems with the RS232 communications. The following procedure is recommended in those cases:
  - 1. Reset the buffers in the computer.
  - 2. Send a few carriage returns to clear the receive buffer.
  - 3. Send the IN0 command to reinitialize the instrument's buffers.
  - 4. Wait for the STATUS error byte to come back.
  - 5. Test STATUS for possible errors.

This trick is very useful to reset communications on both ends and is often implemented as a menu item.

The ID? command is used for a variety of purposes:
 -Make sure the CIS Analyzer is powered up and ready to go.
 -Check the quality of the serial connection to the host computer.
 -Check the user's communication software to make sure it is communicating

properly with the spectrometer.

-Check the serial numbers of the CIS heads connected to the computer's serial ports.

#### **Programming the Ionizer**

Positive ions are produced in the closed ionizer by bombarding process gas molecules with electrons derived from a heated filament. The operational parameters that affect the efficiency of the ionizer are: electron energy, ion energy, electron emission current, and extraction voltage. The CIS ionizer is typically operated under one of two basic set of parameters corresponding to the two Basic Operating Modes of the Analyzer. Please consult the Basic Operating Modes section in the General Operation chapter of this manual for details.

The **Ionizer Control** Commands program all the ionizer voltages and turn the filament on/off. The STATUS byte is transmitted at the end of their execution. **Important**: The Repeller plate and the extraction plate are only biased while the filament is emitting electrons. The anode tube is electrically biased at all times.

#### Setting the ionizer parameters

The following list of commands will set the electron energy to 70 eV, ion energy to high (8eV), extraction voltage to -50V and it will then turn the filament on with an emission current setting of 0.5 mA. It is a typical example of the set of commands sent to the CIS Head while programming the ionizer into the **RGA Mode** of operation.

- EE70 Electron Energy = 70 eV. Echoes the STATUS Byte.
- IE1 Ion Energy = high (8eV). Echoes the STATUS Byte.
- VF50 Extraction Voltage= -50V. Echoes the STATUS Byte.
- FL0.5 Filament emission = 0.5 mA and turn filament on. Echoes the STATUS Byte.

#### **Ionizer Programming Tips**

- It is possible to trigger scans and single mass measurements while the filament is not turned on.
- Make sure the pressure is under 10<sup>-2</sup> Torr before turning the filament on, specially if the filament wire is made out of W.
- The filament is tested frequently by the microprocessor while it is emitting electrons. Consult the Filament, Leak and Burnt LED's after activation for immediate status feedback.
- Wait for the STATUS byte to be returned between commands and check it every time for possible errors.
- In cases were a hardware error is reported retry the command one more time before declaring a hardware problem.
- Default parameter settings are available for all the Ionizer Control commands

• Use the FL0 command to turn off the filament.

#### **Programming the Detector**

Positive ions that successfully pass through the quadrupole filter are focused towards a detector that measures the ion currents directly (Faraday Cup, FC) or, using an optional electron multiplier (CDEM), measures an ion signal proportional to the ion current.

Use the **Detection Control** commands to choose the detector type (FC or CDEM), query the CDEM option, recalibrate the electrometer's I-V response and set the electrometer's averaging and bandwidth. Use the CA command to zero the ion detector at any time.

Important: Faraday Cup detection is the default setting when the CIS head is turned on.

Use the MO? command to determine whether an electron multiplier option (Option 01) is available in the CIS head being programmed. The CDEM is available if the response is 1 < LF > < CR >.

The HV command activates/deactivates the CDEM detector. Its parameter value represents the bias voltage applied across the multiplier, and determines the gain of the amplifier. The null parameter, HV0, deactivates the CDEM and reconnects the FC detector to the electrometer.

#### Note: The ElecMult LED reflects the status of the CDEM at all times.

A temperature compensated logarithmic picoammeter measures the ion currents collected by the FC or CDEM. The microprocessor automatically configures the electrometer and connects its input to the correct signal based on the type of detector being used. The sign of the electron multiplier currents is automatically reversed before the current value is sent out over RS232 so that the computer does not need to do any sign flipping on the currents received when the CDEM is activated. The accuracy of the ion current measurements is insured by an internal calibration procedure that calibrates the output of the electrometer against input current over its entire operating range. The I-V response can be recalibrated at any time with the CL command.

The bandwidth and detection limit of the electrometer are programmed with the NF (<u>Noise Floor</u>) command. A decrease in the Noise-Floor setting results in longer measurement times with cleaner baselines and lower detection limits. Histogram scans, analog scans, and single-mass measurements share the same NF setting. The CIS Head automatically adjusts the scanning rate and averaging based on the NF setting selected.

**Important:** The zero of the ion detector is automatically readjusted at the beginning of each analog and histogram scan so that the baseline is always centered around zero. The zero can also be readjusted at any time with the command CA. In both zeroing procedures, the output of the electrometer is measured in the absence of input ion current and stored, as a current value, in the spectrometer's memory. The current value (called offset correction factor) is then automatically used by the firmware to offset-correct all ion currents measured (i.e. including those from single mass measurements) under the same detector settings. The detector settings are the electrometer's noise floor parameter value and the type of detector (i.e. FC or CDEM) in use at the time the zeroing is performed. Offset correction factors for all the possible combinations of detector settings

can be generated and accumulated in the instrument's memory. However, all offset correction factors are cleared after a recalibration (CL) of the electrometer is performed, and when the unit is turned off.

Please consult the CIS Command Set section for details on the CA and CL commands.

#### **Detector Programming example**

The following list of commands starts by checking the CIS head to make sure there is a multiplier installed: A CDEM is present if a 1<LF><CR> response is sent back to the computer. After the test (and assuming the CDEM option was detected), a voltage of -1400V is set across the multiplier, and the noise floor setting is programmed for minimum averaging and maximum scan rate. The detector is now fully configured for CDEM measurements with -1400 V of CDEM bias voltage.

MO?	Multiplier Option Query.			
Check query re	Check query response: 1 <lf><cr> confirms CDEM option.</cr></lf>			
HV1400	High Voltage $CDEM = 1400V$ . Echoes the STATUS Byte.			
NF7	Noise Floor = 7. Echoes the STATUS Byte.			

#### Notes:

Append a CA command at the end of this list to readjust the zero of the ion detector measurements under the present detector settings. The correction factor will then be used by all measurements (i.e. single mass) under this same detector settings to correct against zero drifts. Use the HV0 command to go back to FC detection mode.

#### **Detector Programming Tips**

- Use the ElecMult LED to visually inspect the status of the CDEM during programming.
- Use the HV? command to query the type of detector that is currently active. The CDEM is active if a non-zero voltage setting is returned to the computer.
- Check the value of STATUS returned after the HV command execution: Bit 3 provides immediate information about the success of the command.
- The HV command checks for the presence of a CDEM option in the CIS Head before execution starts and an error is reported if no CDEM is available. (Please see EM? command for details.)
- It is possible to activate the CDEM if the filament is not emitting electrons, but the CDEM will be deactivated by any overpressure that shuts-down the filament.
- The correct operation of the electrometer can be tested using the Error Reporting command ED?.
- The scan rates and signal-to-noise ratios for the different NF settings of the electrometer are listed in a table in the Electrometer section of the "Electronics Control Unit" chapter.

- When using the RGA Windows program to operate the RGA, the Scan Speed parameter setting available in the Scan Parameter Setups of the Scan menu is used to set the NF parameter value in the CIS Head according to the equation: NF = ScanSpeed 1.
- Use the CA command to readjust the zero of the detector every time the detector settings are changed (i.e. after changing the NF parameter setting or changing detector type). This is particularly important if the new settings have not been used in a long time or since the unit was turned on or recalibrated with the CL command.
- Follow all recommended procedures for the operation of the CDEM. Consult the Maintenance chapter of this manual for complete CDEM Care and Handling information.

#### Setting up Analog Scans

Analog scanning is the most basic data acquisition mode of the CIS Analyzer as a quadrupole mass spectrometer. During analog scanning the quadrupole mass spectrometer is stepped at fixed mass increments through a pre-specified mass-range. The ion current is measured after each mass-increment step and transmitted to the host computer over RS232.

Analog scans are triggered with the SC command. The scan parameter can be set for single, multiple and continuous scanning operation. The mass range for the scan is set in advance with the commands MI (Initial Mass) and MF (Final Mass) and the massincrements are fixed with the command SA. Scan rate and detection limits are preprogrammed by the NF (Noise Floor) setting. A current value is transmitted for MI and after each mass increment through MF for a total of (MF-MI )\*SA+1 measurements (See AP query command). For maximum data throughput, the ion currents measured are represented as integers in units of 0.1 fA, and transmitted directly in Hex format (four byte integers with 2's complement format and Least significant byte first).

The detector settings (i.e. detector type and electrometer's noise-floor setting) to be used during the scans must be selected in advance with the NF and HV commands. The zero of the ion detector is automatically readjusted at the beginning of each analog scan so that the baseline is always centered around zero. (The offset correction factor calculated and stored in memory, will then be used to correct all currents measured under the same detector settings)

The internal scan parameters used by the firmware to step the RF during the scan, are checked and corrected at the beginning of the scan to make sure that the correct RF levels (i.e. as specified by the last Peak Tuning procedure) are programmed on the RF rods as a function of mass.

#### **Important:**

- An extra current value, in addition to the values corresponding to the mass steps, is sent at the end of all analog and histogram scans. This extra current value is unrelated to the scan and does not need to be saved or displayed; however, it must be taken into account whenever analog scanning is incorporated into custom software.
- The detector's zero and the internal scan parameters are checked and corrected at the beginning of each scan resulting in a slight delay before the scan actually starts.
- The measurements are performed with the detector setting that is active at the time the scan is triggered.

#### Setting up an analog scan

The following list of commands sets a scanning range of 10 to 150 amu, the fastest scan rate (minimum averaging), 0.1 amu steps, and, after checking the number of currents to be measured per scan (not including extra value sent at the end), it triggers 10 analog scans. Each one of the 10 scans transmits 1401 ion current values plus one extra current that is not displayed. Each current is 4 bytes long.

MI10	Initial mass $= 10$ amu.
MF150	Final mass $= 150$ amu.
NF7	Fastest scan rate selected.
SA10	Steps/amu = 10.
AP?	Analog Points query. The number 1401 is echoed. Add one for extra current.
SC10	Analog Scan trigger: 10 scans are generated and transmitted.

#### **Analog Scan Programming Tips**

- It is good programming practice to follow each command that sets a parameter with a query of the parameter setting. For example, the first command in the list should be followed by a MI?, and a number 10 should be echoed by the CIS Head.
- Notice that MI and MF are shared by analog and histogram scans.
- Any command sent to the spectrometer during scanning will immediately halt the scanning action and clear the instrument's transmit buffer. Remember to also clear the computer's receive buffer to reset the communications. The command responsible for stopping the scan will be executed!
- The CIS Head has the ability to store a complete scan in its output buffer. The scan remains in memory until all the data is transmitted out to the host computer over RS232. As a result of the high acquisition rate, there might be a delay between the time at which the data is collected by the CIS head and the time at which a complete spectrum is displayed by the host computer. The time lag between data acquisition and display depends on a large number of factors including the scan rate (NF setting), the host computer's processing speed, and the amount of handshaking

activity over the RS232 lines. As computers get faster, they will be able to catch up with the data acquisition speeds and this problem will no longer need any consideration.

- Before a new scan starts the microprocessor checks the instrument's internal memory to make sure that no data from any previous scans is pending to be transmitted. If data is still pending, the spectrometer must finish transmitting it before the new scan can start. This process may result in a delay from the time the scan trigger is received to the time it actually starts. Using the SC1 command and waiting until the whole scan data stream is transmitted back to the host computer will minimize the problems that are associated to this feature.
- Perform a complete **Peak Tuning** procedure on the CIS Head if the peaks in the spectrum do not appear at their correct mass values (Consult the Peak Tuning section in the Tuning chapter for details).

#### Setting up Histogram Scans

A Histogram (Bar Mode) Scan consists of a succession of individual single mass measurements over a pre-specified mass range. A single value is used to represent the peak heights at each integer mass within the range. Histogram scanning is one of the most commonly used modes of operation for the CIS Analyzer. Its two main advantages are a faster scan rate than analog scans, and a reduced amount of data being exchanged and stored.

Histogram scans are triggered with the HS command. The scan parameter can be set for single, multiple and continuous scanning operation. The mass range for the scan is set in advance with the commands MI (Initial Mass) and MF (Final Mass). A current value is transmitted for each integer mass value between MI and MF for a total of (MF-MI +1) measurements (See HP command). For maximum data throughput, ion currents are represented as integers in units of  $10^{-16}$  Amps, and transmitted directly in hex format (four byte integers, 2's complement format, Least Significant Byte first).

The detector settings (i.e. detector type and electrometer's noise-floor setting) to be used during the scans must be selected in advance with the NF and HV commands. The zero of the ion detector is automatically readjusted at the beginning of each histogram scan so that the baseline is always centered around zero. The offset correction factor calculated and stored in memory, will then be automatically used to correct all currents measured under the same detector settings.

The internal scan parameters used by the firmware to step the RF during the scan, are checked and corrected at the beginning of the scan to make sure that the correct RF levels (i.e. as specified by the last Peak Tuning procedure) are programmed on the RF rods as a function of mass.

#### **Important:**

• An extra current value, in addition to the values corresponding to the mass steps, is sent at the end of all analog and histogram scans. This extra current value is unrelated to the scan and does not need to be saved or displayed; however, it

must be taken into account whenever histogram scanning is incorporated into custom software.

- The detector's zero and the internal scan parameters are checked and corrected at the beginning of each scan resulting in a slight delay before the scan actually starts.
- The measurements are performed with the detector that is active at the time the scan is triggered.

#### Setting up a histogram scan

The following list of commands sets a scanning range of 1 to 50 amu at the slowest scan rate, and, after checking the number of currents to be transmitted per scan (not including extra value transmitted at the end), it triggers a single histogram scan Fifty ion currents and one extra current are transmitted to the computer per scan. Each current is 4 bytes long.

- MI1 Initial mass=1 amu.
- MF50 Final mass=50 amu
- NF0 Slowest scan rate selected.
- HP? Histogram Points query. The number 50 is echoed. Add one for extra current that is not displayed.
- HS1 Histogram Scan trigger: 1 scan is collected and transmitted.

#### Histogram Scan Programming Tips

- It is good programming practice to follow each command that sets a parameter with a query of the parameter setting. For example, the first command in the list should be followed by a MI?, and a number 1 should be echoed by the CIS Head.
- Notice that MI and MF are shared by analog and histogram scans.
- Any command sent to the CIS Analyzer during scanning will immediately halt the scanning action and clear the transmit buffer. Remember to also clear the computer's receive buffer to reset the communications. The command responsible for stopping the scan is executed!
- It is good practice to perform an analog scan before triggering a large set of histograms to assure the correct peak tuning (i.e. correct peak locations and widths) of the quadrupole mass filter. Perform a complete Peak Tuning Procedure as described in the Tuning chapter of this manual if significant shifts in the peak locations are observed.
- The CIS Analyzer has the ability to store a complete scan in its output buffer. The scan remains in memory until all the data is transmitted out to the host computer over RS232. As a result of the high acquisition rate, there might be a delay between the time at which the data is collected and the time at which a complete spectrum is displayed by the host computer. The time lag between data acquisition and display depends on a large number of factors including the scan rate (i.e. NF setting), the host computer's processing speed, and the amount of handshaking activity over the RS232 line. As computers get faster, they will be able to catch up with the

spectrometer's data acquisition speed and this problem will no longer need any consideration.

• Before a new scan starts, the microprocessor checks the internal memory to make sure that no data from a previous scan is pending to be transmitted. If data is still pending, the spectrometer must first finish transmitting the stored data before the new scan can start. This process may result in a delay from the time the scan trigger is received to the time it actually starts. Using the HS1 command and waiting until the whole scan data stream is transmitted back to the host computer will minimize the problems that are associated to those delays.

#### **Single Mass Measurements**

Single mass measurements are triggered with the MR command. The parameter is the **integer** mass number (mass-to-charge ratio in amu units) at which the measurement is performed.

The type of detector and noise-floor settings must be selected in advance with the NF and HV commands.

The precision and duration of the measurement are totally determined by the NF parameter value. The scan rates and signal-to-noise ratios for the different NF settings of the electrometer are listed in a table in the Electrometer section of the "Electronics Control Unit" chapter. The command execution time includes some initial time spent waiting for the Quadrupole Mass Filter and the electrometer response to settle.

As usual, a compromise must be made between signal-to-noise and measurement time.

The ion current is expressed in the usual format: 4 byte long, 2's complement integer in units of  $10^{-16}$  A, with Least Significant Byte transmitted first.

#### **Important:**

- During a Single Mass Measurement the spectrometer performs a "Miniscan" around the mass requested, and the maximum current value measured is sent out over **RS232**. The scanning procedure, referred to as **Peak-Locking**, is designed to measure peak currents for individual masses in a mass spectrum without being affected by drifts in the mass scale calibration. The Miniscan covers a 0.6 amu range centered at the mass requested, and selects the maximum current from 7 individual measurements performed at 0.1 amu mass increments.
- The detector settings (i.e. type of detector and noise-floor setting) to be used during the measurement must be selected in advance with the NF and HV commands; otherwise, the measurements are performed with the detector settings that are present at the time the scan is triggered.
- The ion detector can be zeroed, prior to the measurements, performing an analog or histogram scan or using the CA command, under the same detector settings (An offset-correction factor is calculated and stored in memory that it is then automatically used by the Miniscan procedure to correct all ion currents measured). The CA command will also update the internal scan parameters to assure that the

correct RF levels (i.e. as specified by the last Peak Tuning procedure) are programmed on the RF rods as a function of mass during the Miniscan.

#### Single Mass Measurement example

The following list of commands can be used to perform a single mass measurement at 28 amu. First, the electron multiplier is turned off and the FC is connected to the electrometer, the noise floor is set to the maximum averaging (best signal-to-noise ratio and longest measurement time), the zero of the detector is readjusted, and the measurement is triggered. The single current value returned by this command is the maximum current in the 28 +/-0.3 amu region of the mass spectrum, and must be corrected with a sensitivity factor to obtain the actual partial pressure reading at mass 28.

**Important**: The RF/DC on the QMF are left at 28.3 amu at the end of this measurement, and the MR0 command can be used to turn them off.

Important: The CA command turns off the RF/DC at the end of its execution.

- HV0 High Voltage CDEM = 0 Volts, FC detection enabled.
- NF0 Maximum averaging (slowest scan rate) selected.
- CA Readjust the zero of the detector for clean baseline and update the internal scan parameters.
- MR28 Single measurement triggered @ 28 amu. A single current value is returned and the RF/DC are left at 28.3 amu.

Collect the 4 byte current value returned.(Use sensitivity factor to convert it to pressure value.)

MR0 Turn off the RF/DC on the QMF rods when done measuring.

#### Single Mass Measurement programming tips

- Single mass measurements are very commonly used in program loops where several different masses are monitored in a merry-go-round fashion and their partial pressures displayed as a function of time.
- The outputs provided by a set of single mass measurements are often used in process control programs to control alarms, analog and digital outputs, and relays. For example:
  - \* The output of a D/A converter can be linearly related to the readings obtained at a certain mass.
  - \* A relay switch can be closed whenever a specific mass concentration goes above a certain level.
  - \* A digital output can be set high when a mass peak goes under a minimum acceptable partial pressure value.
- For best accuracy of results, it is best to perform all consecutive mass measurements in a set with the same type of detector and at the same noise floor (NF) setting. Fixed detector settings eliminate settling time problems in the electrometer and in the CDEM's HV power supply.

- It is good practice to perform an analog scan before triggering a long set of measurements to assure the correct tuning (i.e. correct peak locations and widths) of the quadrupole mass filter. Perform a complete Peak Tuning Procedure as described in the Tuning chapter of this manual if shifts in the peak locations are observed.
- The RF/DC voltages are not turned off at the end of a measurement. Use the MR0 command at the end of a set of measurements and before quitting a program to make sure the RF/DC voltages are deactivated.

#### Storing information in the CIS Head

In order to make the CIS Analyzer a stand-alone mass spectrometer several Parameter Storage commands are available to store head-specific information in the instrument's non-volatile memory.

A **Parameter Storage Table**, capable of storing up to 10 different numeric values (i.e. elements), is part of the non-volatile memory of the CIS Head. The command pair [XA,XV] is used to update or query the elements of that table. XA (where A is for Address) is used first to point at any one element within the table. XV(where V is for Value) can then be used to query or update the value of the element previously pointed to by XA.

The numeric storage available through XA and XV is often used to store a variety of instrument specific calibration data directly into the CIS Head, making the instrument's calibration independent from the host computer that controls it.

**Important:** RGA Windows reserves the use of the first three members of the table (XA1, 2 and 3) for the storage of sensitivity factors for the three Basic Operating Modes of the CIS Analyzer: **RGA, CIS-70 and CIS-35** modes. Do not modify those memory locations if you plan to use RGA Windows to control the CIS Analyzer. The three sensitivity factors are measured at the factory for N2 @ 28 amu, and stored in units of 10<sup>-6</sup>A/Torr before the unit is shipped out. Please consult the General Operation Chapter of this manual for details on the Basic Operating Modes of the CIS Analyzer.

**Important:** The numeric values stored in the Parameter Storage Table are not used internally by the CIS Head in any way during operation. They can only be used by the software programs that operate the CIS Head from the host computer.

There is also two additional storage commands, MV and MG that are used together to store CDEM specific information in the non-volatile memory of the CIS Head:

MV: CDEM High Voltage setting that corresponds to the gain stored by MG. See MV command.

MG: CDEM gain at the HV setting stored in MV. See MG command.

RGA Windows uses the stored MV value to bias the CDEM when the multiplier is turned on, and the MG value as the corresponding gain while the multiplier is activated.

#### **Important:**

- The parameter values are not used internally by the CIS Head to correct for sensitivity or gain, they are simply stored so they can be read and used by any computer connected to the spectrometer.
- MV and MG are only available in instruments with the CDEM option (Option 01) installed (See MO command for details) and usually store a calibrated pair of [gain, high voltage] values for the CDEM.

#### **Programming example**

The following list shows a typical application of the storage commands to save head specific information in the CIS Head. The first two lines store a partial pressure sensitivity factor of 10.0microA/Torr into the first location of the storage table (i.e. typically the RGA Mode Sensitivity for  $N_2$ ). The next two lines store a partial pressure sensitivity factor of 1.0microA/Torr into the second location of the storage table (i.e. typically the CIS-70 Mode Sensitivity for  $N_2$ ). The following two lines store a partial pressure sensitivity factor of 0.5microA/Torr into the third location of the storage table (i.e. typically the CIS-35 Mode Sensitivity for  $N_2$ ). The last two lines store a [1400V | 10,000 gain] calibrated pair for the CDEM.

XA1	Select the first location of the parameter table
XV10.0	Store the measured RGA Mode sensitivity in microA/Torr.
XA2	Select the second location of the parameter table
XV1.0	Store the measured CIS-70 Mode sensitivity in microA/Torr.
XA3	Select the third location of the parameter table
XV0.5	Store the calculated CIS-35 Mode sensitivity in microA/Torr.
MV1400	CDEM HV setting (for MG gain): 1400 V.
MG10	CDEM gain (@MV setting)=10,000x.

#### **Programming the Quadrupole Mass Filter**

The ML (Mass Lock) command activates the quadrupole mass filter (QMF) and centers its pass-band at any mass number within the available range. It is included for the convenience of users who wish to use the spectrometer as a mass filter. The QMF is parked at the mass requested but no actual measurements take place. The command's parameter can be a real number and the step resolution is 1/256 amu.

The command execution involves two steps:

- 1. The RF/DC levels corresponding to the mass requested are calculated and set on the QMF rods based on the mass axis calibration parameter values specified by the last Peak Tuning procedure.
- 2. A firmware driven algorithm is then automatically enabled to stabilize the voltage output of the RF Driver's controller against temperature fluctuations.

The net result is **very stable RF/DC levels** that are highly insensitive to the operating conditions of the CIS Head.

**Important:** The RF/DC stabilization algorithm (Step 2 above) remains active as long as no new commands are sent to the CIS Head. Once a new command is received, stabilization stops, and the new command is executed.

Use the ML0 command to turn off the RF/DC bias when finished performing measurements and before quitting the program controlling the spectrometer.

#### QMF programming example

ML28	Activate the mass filter and center its pass band at 28 amu
ML42.52	Activate the mass filter and center its pass band at 42.52 amu.
ML0	Deactivate the QMF. (same as MR0 command)

#### **QMF** Programming tips

- Take advantage of the stabilization feature of the ML command whenever possible. For example: Do not send any new commands to the CIS Head once the QMF has been set to the specified mass value, or otherwise recall the ML command, whenever practical, to refresh the QMF RF/DC settings.
- A typical application of the ML command involves monitoring a single mass concentration looking directly at the linear output of the CDEM anode with a boxcar or transient digitizer. All SRS mass spectrometers can be configured at the factory to provide direct access to the analog output current of the electron multiplier. Ask SRS or your local representative about the Option03 hardware enhancement.
- Use the ML0 command to turn off the RF/DC bias when finished performing measurements and before quitting the program controlling the instrument.
- It is good practice to perform an analog scan before using the ML command to assure the correct tuning (i.e. correct peak locations and widths) of the quadrupole mass filter. Perform a complete Peak Tuning Procedure as described in the Tuning chapter of this manual if shifts in the peak locations are observed.

#### Error Checking the CIS Analyzer

Several firmware-driven **checks** automatically test the CIS Head when the unit is turned on, and continuously monitor the internal workings of the instrument. A "Background Filament Protection Mode" is activated when the filament is turned on to protect the delicate filament (and CDEM) from accidental overpressures. Several commands can be used to trigger hardware tests on the ECU. Any one of the internal checks just described can detect and report errors, and it is the responsibility of the user to constantly monitor the CIS Head for error reports.

There are two ways to detect the presence of errors in the CIS Analyzer:

1. Visually: Inspecting the red LED's on the rear panel of the ECU.

The Error LED is turned on whenever a hardware problem is detected, and it flashes twice if a communications error is generated during programming.

The Burnt and Leak LED's indicate specific filament problems and are turned on, in addition to the Error LED, whenever the ionizer's emission is internally shut down or not established as requested.

2. **Error Queries**: Queering the **Error Bytes** with the **Error Reporting** commands. The "Error Byte Definitions" section of this chapter describes the different error bytes used to store the results of the internal checks. The "Error Reporting Commands" query the error bytes and are used to quickly diagnose problems.

**Important**: **RGA Windows** supports all the Error Reporting commands and routinely monitors the Error Bytes. Detected errors are immediately reported to the user and identified based on Error Codes. See the Troubleshooting chapter for recommended solutions to the problems.

The STATUS error byte is the gateway to error diagnosis:

# Important: The CIS Analyzer is free of detected errors as long as the STATUS byte is clear (no bits set).

Each bit of the STATUS byte reflects the result of a different type of **internal check**. Each internal check involves **several** different **tests** on a component of the CIS Head (See the Troubleshooting Chapter for details). The results of the specific tests are stored in check-specific **error bytes**. All error bytes can be queried by the computer, using the Error Reporting commands, to find out which bits are set.

STATUS Bit	Internal Check <sup>(a)</sup>	ERROR Byte <sup>(c)</sup>	Query <sup>(d)</sup>
7	not used	N.A.	N.A.
6	24V External P/S <sup>(b)</sup>	PS_ERR	EP?
5	Electrometer <sup>(b)</sup>	DET_ERR	ED?
4	Quadrupole Mass Filter <sup>(b)</sup>	QMF_ERR	EQ?
3	Electron Multiplier	CEM_ERR	EM?
2	not used	N.A.	N.A.
1	Filament	FIL_ERR	EF?
0	Communications	RS232_ERR	EC?

(a) Head component checked. Each check involves several tests on the component.

(b) Internal checks automatically performed upon a power-on-reset

(c) Error bytes that store the results of the tests for each type of internal check.

(d) The Error Reporting command that queries the error byte.

The Error LED is immediately turned on if any one of the bits 1-7 of STATUS is set. Bit 0 of STATUS reports communications errors and the Error LED is only flashed twice when the bit is set.

The STATUS Byte should be queried regularly by the programming software (ER? command.)

Commands that involve hardware control (such as Ionizer Control commands) do diagnostic checks on the hardware as they are executed. Since the command execution time can be rather long, the CIS Head prompts the end of execution by sending back the value of the STATUS byte to the computer. *Always check the STATUS Byte value returned for possible errors*.

If one or more bits of the STATUS byte are found set, the specific error bytes (PS\_ERR, DET\_ERR, QMF\_ERR, CEM\_ERR, FIL\_ERR, and RS232\_ERR) must be queried individually to diagnose the problem.

#### Error diagnosis examples

#### Example #1:

The CIS Analyzer is powered up and, after the automatic internal checking is over, the Power LED is turned off and the Error LED is turned on. The problem is immediately diagnosed visually as a failure in the 24V External P/S check. A multimeter could be used to check the output of the external power supply and determine the nature of the problem; however, the computer could also be used to diagnose the problem using the query commands:

The ER query command returns a STATUS Byte with bit 6 set indicating a 24V P/S problem. The EP query command is then used to query PS\_ERR. Bit 6 of PS\_ERR is found set (Error Code: PS6) indicating that the P/S output is less than the acceptable minimum of 22 V.

This error is also immediately reported by the RGA Windows software as an Error Code: PS6, and the Troubleshooting section can be consulted to solve the problem.

#### Example #2:

The CIS Analyzer is idling between scans, waiting for commands from the computer, when suddenly an inlet-valve is accidentally opened and the process chamber is pressurized. The Background Filament Protection mode immediately detects the problem and turns off the filament. The change is easily detected because the Filament LED turns off, and the Error and Leak LED's simultaneously turn on.

A visual inspection of the red LED's is all that is needed in this case to detect and diagnose the problem; however, a computer could do the same diagnosis with the help of the query commands:

The ER? command, used to monitor the STATUS byte regularly, returns a non-zero byte value. Further analysis, shows that bit 1 is set indicating a filament problem. The EF? command is then used to read in the value of FIL\_ERR. Bit 6 is found set indicating that the filament was unable to set the requested emission current and had to be shut down

(Error Code: FL6). The problem is quickly diagnosed as an overpressure using the Troubleshooting chapter directions.

The error is also immediately reported by the RGA Windows software as Error Code: FL6, and the Troubleshooting section can be consulted to solve the problem.

#### Example #3:

The CIS Analyzer is turned on and, after all the internal checks are performed, the green Power LED and the Error LED are turned on. The red LED signals the operator that a problem was detected. A 24V P/S error is not expected since the Power LED is on. The ER? command returns a STATUS byte with bit 4 set, pointing to a quadrupole mass filter problem. The command EQ? is used to read in the QMF\_ERR byte. Bit 7 is found set indicating that the RF driver is unable to set the maximum RF value available to the quadrupole mass filter (Error code RF7 in Troubleshooting Chapter). A quick visual inspection of the CIS head shows that the ECU is not pushed all the way into the probe and no actual connection exists between the rods and the RF driver.

This error is also immediately reported by the RGA Windows software as Error Code: RF7, and the Troubleshooting section can be consulted to solve the problem.

#### Example #4:

The user notes that the Error LED flashes every time the ID? command is sent to the CIS Head and the ID string is never returned (i.e. receive timeout). The STATUS byte returned by the ER command has the bit 0 set as expected for a communications problem. The specific communications problem is diagnosed using the EC? command. Bit 0 of RS232\_ERR is found set indicating that a bad command is being detected by the CIS Analyzer. The user checks the communications program and finds out that, due to a typing error, the IM? string is being sent instead of ID?. The problem is easily corrected.

#### **Error Checking Programming tips**

- Keep a close eye on the Error LED's while programming the CIS Analyzer. Diagnose problems as soon as they are detected.
- Even though a lot of internal checks are performed by the microprocessor, not all possible internal error conditions are accounted for.
- The values of the Error Bytes often change after an Error Byte query command is executed. Some query commands update the byte value after performing a fresh test on the hardware, while others clear error bits, after they are read, to provide a clean error-reporting slate. Please see the Error Reporting Commands list for details.
- It is good programming practice to check the value of the STATUS byte whenever it is returned at the end of command execution.
- In cases were a hardware error is reported at the end of a command attempt the command one more time before declaring a hardware problem.

### **CIS Command Set**

This section lists and describes the commands in the CIS Command Set. The commands are separated into several lists, based on their functions. They are each identified by a **header** that describes the **command's syntax** (with the acceptable parameter values), the **command's function**, and the information returned (**Echo**) to the computer during execution.

The CIS commands are ASCII character strings consisting of a two letter (case insensitive) **command name**, a **parameter**, and a **carriage return** terminator.

**Note:** The carriage return character, decimal ASCII value=13, is represented throughout this manual with the symbol **<CR>**. All command strings must be terminated with this character in order to be acknowledged by the CIS Head.

The two letter mnemonic in each command sequence specifies the command name. The rest of the sequence consists of parameters. Three types of parameters are allowed: numbers, asterisk (default param), and question mark (query). Not all commands accept all types of parameters, and some need no parameters at all.

In general, number parameters can be real or integer; however, some commands will only accept integers. Commands that accept only integers are recognized because the numbers used to specify their parameter range do not include a fractional part.

The maximum mass number recognized by the CIS Analyzer is model dependent and is represented by **M\_MAX**: 100 for the CIS100, 200 for the CIS200, and 300 for the CIS300.

With a few exceptions, the data returned by most query commands is a string of ASCII characters terminated by a linefeed (<LF>, decimal ASCII value=10) and a <CR> terminator: string<LF><CR>.

Ion currents are represented as integers in units of  $10^{-16}$  Amps, and transmitted directly in Hex format: four byte integers, 2's complement format, Least Significant Byte first.

### **Initialization Commands**

#### ID?

**Description**: Identification query.

Echo: ID string.

Use to identify the CIS Head connected to the host computer. The instrument returns the ID string (ASCII format): SRSCIS###VER#.##SN#####<LF><CR>

The three string parameters, in the exact format shown above, correspond to:

- 1. Model number (=M\_MAX): 100 for CIS100, 200 for CIS200 and 300 for the CIS300.
- 2. Firmware version (for example: 0.10).
- 3. Serial Number of the unit (5 digit format).

The ID command is used for a variety of purposes:

- Make sure the CIS Analyzer is powered up and ready to go.
- Check the quality of the RS232 connection to the host computer.
- Check the user's communication software to make sure it is communicating properly with the CIS Head.
- Check the serial numbers of the CIS heads connected to the computer's serial ports. The unique serial number attached to each head can be used for identification purposes avoiding the problems caused by cable swapping in the RS232 connections under multiplexed operation, or head swapping in the vacuum system.

Parameters: Only one possible format:

#### ID?

#### Error checking:

Only a query format is accepted. Anything else results in a Bad-parameter error.

#### IN0, IN1, IN2

**Description**: Initialization command

Echo: STATUS Byte

Initialize the CIS Analyzer to a known state.

Three different levels of initialization are available.

Command excecution times vary depending on the pre-existing ionizer conditions. The end of the command excecution is prompted to the host computer returning the STATUS byte over RS232.

#### **Parameters:**

**IN0:** Initialize communications and check the ECU hardware.

- The input and output data buffers are emptied (all communications are disabled while this happens).
- Bit 0 of STATUS and RS232\_ERR are cleared
- A fresh check of the ECU hardware, including the 24V P/S, the Electrometer and the QMF RF P/S, is performed and the necessary error bytes are updated.

**IN1:** Reset the CIS Head to its factory default settings.

In addition to the above, the CIS Analyzer is reprogrammed to its default (i.e. Factory preferred) values:

- Default parameter settings are selected for: MI (1), MF (M\_MAX), SA (10), NF (4), IE (1), EE (70), and VF (50).
- The filament's electron emission setting is left unmodified and the ionizer is biased to default voltages if necessary.

**IN2**: Activate Standby mode.

In addition to the above:

• The filament and the CDEM are turned off.

# **Error Checking:**

The command accepts no default or query parameter values. The absence of a parameter is treated as a bad-parameter error.

# **Ionizer Control Commands**

# EEparam, param: 15 - 105, \*, ?

**Description**: Electron Energy (eV).

Echo: STATUS error byte or query response.

Set the Electron Impact Ionization Energy of the ionizer. The parameter represents the desired electron ionization energy in units of eV.

If the filament is emitting electrons at the time the command is invoked, the repeller voltage is immediately reconfigured to provide the desired electron energy, while the ion energy and electron emission currents remain unafected. If the filament is off, the new electron energy setting is stored in memory for the next time the filament's emission is activated

Command excecution times vary depending on the pre-existing ionizer conditions. The end of the command excecution is prompted to the host computer by sending out the STATUS byte over RS232.

**Note:** The Electron Impact Ionization Energy is set to the default value when the unit is turned on.

**Important:** The repeller plate and the extraction plate are only biased while the filament is emitting electrons.

#### **Parameters:**

**EEparam, param: 15-105:** The parameter represents the electron impact ionization energy in eV.

EE\*: The default Electron Energy value is used to run the command.

Default: 70 (eV)

**EE?:** Query. The electron energy parameter setting is returned over RS232 in ASCII format.

#### **Error checking:**

Number parameters must be within the accepted range and must be integers. The absence of a parameter (i. e. EE) is treated as a parameter-error.

# FLparam, param: 0.00 - 3.50, \*, ?

**Description**: Electron emission current (mA).

**Echo:** STATUS error byte or query response.

Set the electron emission current level in the ionizer. The parameter represents the desired electron emission current in units of mA.

When a finite emission current is requested, the CIS Head biases the ionizer's repeller and extraction plates, and activates the filament's heater until the requested electron current is established.

A null parameter value, turns off the filament and grounds the ionizer's repeller and extraction plates.

Command excecution times vary depending on the pre-existing ionizer conditions. The end of the command excecution is prompted to the host computer sending out the STATUS byte over RS232.

The Filament LED reflects the ionizer's emission status at all times.

A firmware-driven "**Filament Protection Mode**" monitors the performance of the filament while it is emitting electrons and if a problem is detected at any time, the heater is immediately shut down and the problem is reported through the error bytes and the error LED's (see below).

# **WARNING:** The pressure in the process chamber must be under 10<sup>-2</sup> Torr before turning the filament on!

#### **Important:**

- The repeller and extraction plates are only biased while the filament is emitting electrons.
- In order to protect the filament, the emission current is defaulted to zero when the CIS Analyzer is turned on.
- The CDEM is turned off by any overpressure that also shuts down the filament.

# **Parameters**:

**FL0.00:** The filament is turned off and the repeller and the extraction plates are grounded.

**FLparam, param: 0.02-3.50**: The parameter is the requested Electron Emission Current in units of mA. The repeller and extraction plates are biased, and the filament's heater is activated until the requested electron emission current is achieved.

FL\*: The command is excecuted using the default parameter value.

#### Default: 0.05 mA

**FL?**: Returns over RS232 the value of Electron Emission Current (in mA) actually flowing through the ionizer.

**Important:** The number returned by the query command will always be very close, but rarely identical, to the current value set with the FLparam command.

The difference observed will never exceed +/-0.02 mA. The value returned is the actual electron current circulating through the ionizer as internally calculated by the CIS head. The discrepancy is due to the finite resolution of the digital-to-analog converters used to program the ionizer's emission current.

## **Error Checking:**

The absence of a parameter (i. e. FL) is treated as a bad-parameter error. The STATUS byte is echoed to the host computer at the end of the command excecution, and it should be checked to determine whether the requested electron emission current was succesfully established. If Bit 1 of STATUS is found set, FIL\_ERR should be immediately checked to identify the specific problem (See EF? command in the Error Reporting Commands list). It is good practice to attempt the command a second time before declaring a hardware problem. Once it has been set, FIL\_ERR can only be cleared after succesfully turning on the filament.

#### IEparam, param: 0,1, \*, ?

**Description**: Ion Energy (eV).

Echo: STATUS error byte or query response.

Set the Ion Energy to one of two possible levels: Low (4eV) or High(8eV). The parameter represents the ion energy level: 0 for Low and 1 for High.

Since the axis of the quadrupole mass filter is at ground, the ion energy (in eV) is equal to the anode tube's bias voltage (in Volts).

**Important**: The anode tube is always biased regardless of the filament's emission status.

Upon reset the anode bias level is set to the default value.

#### **Parameters**:

**IE0** Low ion energy: 4 eV.

**IE1** High ion energy: 8 eV.

**IE**\*: The default Ion Energy parameter value is used to run the command.

Default:  $1 \Rightarrow$  high ion energy: 8 eV

**IE?:** Query. The value of the ion energy parameter is returned over RS232 in ASCII format. *Note that it is the parameter value and not the actual voltage level that is returned.* 

#### **Error checking:**

Number parameters can be 0 or 1 only, and must be integers. The absence of a parameter (i. e. IE) is treated as a bad-parameter error.

# VFparam, param: 0 - 150, \*, ? Description: Extraction plate voltage (Volts)

Echo: STATUS Error Byte and query response.

Set the extraction plate voltage in the ionizer. The parameter represents the magnitude of the biasing voltage (negative) in units of volts.

The ions formed inside the anode tube are extracted into the quadrupole mass filter by the negative attractive potential of the Extraction Plate. The plate serves the double purpose of drawing the ions away from the ionization volume, and keeping the ionizing electrons away from the quadrupole mass filter. Electron leakage into the filter is only detectable at low mass settings (1 to 10 amu) and can easily be eliminated biasing the extraction plate more negative than the repeller plate.

Careful adjustment of the voltage results in optimum coupling of the ion beam into the QMF and maximum sensitivity.

If the filament is emitting electrons at the time the command is invoked, the extraction voltage is immediately reprogrammed, while the ion energy, electron energy and electron emission currents remain unaffected. If the filament is off, the new extraction voltage value is stored in memory for the next time the filament's emission is activated.

Command excecution times vary depending on the pre-existing ionizer conditions. The end of the command excecution is prompted to the host computer sending out the STATUS byte over RS232.

# **Parameters:**

**VFparam, param: 0-150**: The parameter represents the magnitude of the extraction plate bias potential in Volts (*The actual bias voltage is negative*). The STATUS byte is transmitted at the end of command execution.

**VF\***: The default extraction plate biasing voltage value is used to excecute the command. Default: 40 (V).

**VF?:** Query the extraction plate biasing voltage setting.

# **Error checking:**

Number parameters must be integers within the accepted range. The absence of a parameter (i. e. VF) is treated as a bad-parameter error.

# **Detection Control Commands**

CA

Description: Calibrate All.

Echo: STATUS Error Byte.

Readjust the zero of the ion detector under the present detector settings, and correct the internal scan parameters against small temperature fluctuations to assure that the correct RF voltages (i.e. as specified by the last Peak Tuning procedure) are programmed on the QMF rods as a function of mass.

In order to readjust the zero of its ion detector, the CIS Analyzer measures the output of its electrometer in the absence of input ion current and under the present detector settings (i.e. noise floor and detector type). The current value obtained is stored in internal memory as an offset correction factor that is automatically used by the firmware to correct all ion currents measured (i.e. including those from single mass measurements) under the same detector settings.

#### **Important:**

- Offset correction factors for **all** the possible combinations of detector settings can be accumulated in the head's memory.
- All offset correction factors are automatically cleared after a complete recalibration (CL) of the electrometer is performed, and also when the unit is turned off.
- Use the CA command every time the detector settings are changed to values that have not been used in a long time or that were not used since the unit was turned on or recalibrated with the CL command.
- The zero of the ion detector is also automatically readjusted at the beginning of each analog and histogram scan so that the baseline is always centered around zero.

In order to correct the mass axis against peak position shifts caused by smalltemperature fluctuations, the CIS Analyzer characterizes the voltage output of the RF driver's control circuit, and **recalculates its internal scan parameters** to assure that the correct RF levels (i.e. as specified by the last Peak Tuning procedure) are programmed on the QMF rods as a function of mass.

**Important:** Use a complete Peak Tuning Procedure to correct the mass axis calibration when large temperature fluctuations are suspected. The calibration procedure triggered by the CA command cannot correct the mass axis against large temperature drifts that affect the response of the RF Driver to its controlling voltage, or the relationship between mass and RF levels in the QMF itself (i.e. due to changes in the QMF's physical dimensions).

# Notes:

- The mass axis correction procedure can also be triggered at any time using the RS and RI commands with no parameters (See Tuning commands).
- The mass axis correction procedure is also automatically performed at the beginning of all analog and histogram scans. However, no correction is performed at the beginning of single mass measurements since the extra checking would significantly extend the time it would take the measurement to be completed.
- Please see the Peak Tuning section of the Tuning chapter for more details on mass axis calibration requirements under small and large temperature fluctuations in the CIS Head.
- The CA command turns off the RF/DC voltages at the end of execution.

Parameters: Only one possible command format is allowed

CA

#### **Error Checking:**

An attempt to pass any parameter with CA results in a bad-parameter error being reported.

CL

**Description**: Calibrate Electrometer's I-V response.

Echo: STATUS error byte.

Perform a complete calibration of the electrometer's I-V response.

The output of the electrometer is calibrated against input current over its entire operating range, and a calibration table is stored in the non-volatile memory of the CIS Head. Instead of simply relying on the intrinsic logarithmic I-V behavior of the electrometer, a digital logarithmic interpolation algorithm calculates the currents from the calibration curve during measurements.

The procedure takes several seconds (twice as long when the CDEM option is installed) and its completion is signaled to the host computer by returning the STATUS byte value.

# Important:

- The electrometer is designed for maximum long term stability, however, it is good practice to recalibrate it periodically, particularly in the presence of a large change in the operating temperature of the CIS Head.
- All offset correction factors previously stored in memory are cleared after a complete calibration of the electrometer is performed (see CA command for more information).

Parameters: Only one possible command format is allowed CL

#### **Error Checking:**

An attempt to pass any parameter with CL results in a bad-parameter error being reported.

#### HVparam, param: 0 - 2490, \*, ?

**Description**: Electron Multiplier High Voltage Bias setting.

Echo: STATUS error byte or Query Response.

Set a negative high voltage across the electron multiplier (CDEM), connect the CDEM signal output to the electrometer and reconfigure the electrometer and current measurement algorithms to handle negative electron currents. The parameter value represents the magnitude of the bias voltage in units of Volts.

Warning: This command only works in heads with the CDEM option (Opt01) installed (See MO command.)

Since the CDEM turns ion signals into amplified electron currents, the CIS Head automatically reconfigures the electrometer to measure negative currents when a finite biasing voltage is selected. The current measurement algorithm is also automatically reconfigured so that the **magnitude** of the currents measured is transmitted over RS232. The host computer can then handle the amplified signals the same way it did with the positive ion currents (i.e. it does not need to flip the sign of the currents received).

The HV\_LED reflects the status of the CDEM bias at all times.

Since it takes time for the CDEM and for the electrometer to settle, completion of the command is prompted to the host computer transmitting the STATUS byte value over RS232.

#### **Important:**

- Faraday Cup detection is the default detector setting upon a power-on reset of the CIS head.
- The voltage set by this command is the output of the high voltage power supply. A 1MOhm resistor is placed between that output and the CDEM's cone. CDEM's typically have an internal resistance in the order of 100-2500 MOhm, and consequently the voltage measured on the CDEM will always have a magnitude smaller than the value returned by a HV? command (i.e. the CDEM becomes part of a voltage divider in conjunction with the 1MOhm resistor).
- It is good practice to readjust the Zero of the ion detector every time the type of detector (FC or CDEM) is changed. This is particularly important if the new detector settings have not been used in a long time or since the unit was

turned on or recalibrated with the CL command. See the CA command for details and more recommendations.

#### **Parameters**:

**HV0:** Use this parameter value to "turn off" the Electron Multiplier and enable Faraday Cup (FC) Detection. The following steps are taken:

- The biasing voltage of the CDEM is set to Zero (i.e. no ion collection and no gain).
- The electrometer is connected to the FC signal output and reconfigured to measure positive currents.
- The firmware algorithms are reconfigured to transmit the **magnitude** of the positive currents measured.
- The HV\_LED is turned off.
- The STATUS Byte is sent out to prompt the end of the command

**HVparam, param: 10-2490:** The parameter is the magnitude of the CDEM bias voltage requested (the actual biasing voltage is negative). The following steps are taken:

- The requested biasing voltage is applied to the CDEM.
- The electrometer is connected to the CDEM anode and reconfigured to measure negative currents.
- The firmware algorithms are reconfigured to transmit the **magnitude** of the negative currents measured.
- The HV\_LED is turned on.
- The STATUS Byte is sent out to prompt the end of the command

**HV\*:** The command is excecuted with the default parameter value.

Default: 1400 (Volts)

HV?: Returns the magnitude of the biasing voltage on the CDEM.

The number returned by the query command is the actual voltage available at the output of the HV driver and will always be very close but rarely identical to the one requested with the HV param command.

#### **Error Checking:**

The CDEM option (Option 01) must be available in the CIS head receiving the command or a bad-command error is reported (see MO command for details).

Number parameters must be within the accepted range, and must be integers.

No parameter (i. e. HV) is treated as a bad-parameter error.

Bit3 of STATUS (transmitted at the end of the command) reports errors in the excecution of the command. CEM\_ERR byte must be consulted to find out the specific problem encountered (See EM? in Error Reporting command list).

#### MO?

**Description**: Electron Multiplier Option Query.

Echo: Query Response.

Query the electron multiplier (CDEM) option in the CIS Head being programmed.

The CIS Analyzer is provided standard with a Faraday Cup detector. For increased sensitivity and faster scan rates an optional CDEM is offered. The MO? command can be used to determine whether Electron Multiplier detection is an option in the CIS Analyzer being programmed.

Note: See also EM? command in the Error Reporting commands list for an alternative way to query the CDEM option.

#### **Parameters**:

Query is the only possible format: MO?

The value returned over RS232 in ASCII format has two possible values:

0 <lf><cr></cr></lf>	No CDEM option is available.
1 <lf><cr></cr></lf>	CDEM option is available.

#### **Error checking:**

Command must have a query format or a bad parameter error is reported.

#### NFparam, param: 0 - 7, \*, ?

**Description**: Electrometer's Noise-floor setting.

Echo: Query Response

Set the rate and detection limit for ion current measurements.

A decrease in the Noise-Floor setting results in cleaner baselines and lower detection limits during scans and measurements, but also means longer measurement and scanning times due to the reduced bandwidth of the electrometer and increased averaging.

The NF parameter must be chosen keeping in mind the strong interplay between detection limit and acquisition speed.

Histogram scans, analog scans and single-mass measurements all share the same value of NF setting during measurements.

**Important**: The NF parameter is set to its default value when the CIS Analyzer is turned on.

It is good practice to readjust the Zero of the ion detector every time the electometer's noise floor setting is changed. This is particularly important if the new detector settings have not been used in a long time or since the unit was turned on or recalibrated with the CL command. See the CA command for details and more recommendations.

**RGA Windows note:** When using the **RGA Windows** program to operate the spectrometer, the **Scan Speed** parameter setting available in the Scan Parameter Setups of the Scan menu is used to set the NF parameter value in the CIS Head according to the equation: **NF** = **ScanSpeed - 1**.

#### **Parameters:**

NFparam, param: 0-7: The parameter represents the noise-floor level desired.

Lower parameter values correspond to lower baseline noise, better detection limits and increased measurement times.

Please refer to the Electrometer section in the Electronics Control Unit chapter to obtain detailed information about detection limits and bandwidth values as a function of NF settings.

NF\*: The noise-floor is set to its default value.

Default value: 2.

NF?: Returns the noise-floor setting currently used by the electrometer .

#### **Error checking:**

Number parameters must be within the specified range, and be integers. The absence of a parameter (i. e. NF) is treated as an error.

# Scan and Measurement Commands

AP?

**Description**: Analog Scan Points Query.

Echo: Query Response.

Query the total number of ion currents that will be measured and transmitted during an analog scan under the current scan conditions.

**Important:** The query response does not include the extra current (4 bytes) that is transmitted at the end of all analog scans (Please see SC command for details).

The number of points (i.e. ion currents) retuned over RS232 is calculated based on the MI, MF and SA parameter values. Number of points = (MF - MI) \* SA +1. The first point corresponds to the mass MI and the other (MF-MI)\*SA are from scanning to MF with SA measurements (steps) per amu. Each point transmitted represents an ion current and as such corresponds to 4 bytes being received by the host computer.

The AP query is used to verify that the CIS Head and the host computer agree on the number of bytes that will be exchanged over RS232 during the analog scan.

The total number of bytes sent out to the host computer during an analog scan is obtained multiplying by four the number returned by the AP query.

Parameters: This command is a query, and can only have one parameter format:

#### AP?

# Error checking:

The only acceptable parameter is a question mark. The absence of a parameter (i. e. AP) is treated as an error.

# HP?

**Description**: Histogram Scan Points Query.

Echo: Query Response

Query the number of ion currents that will be measured and transmitted during a histogram scan under the current scan conditions.

**Important:** The query response does not include the extra current (4 bytes) that is transmitted at the end of all histogram scans. (Please see HS command for details).

The number of points (ion currents) retuned over RS232 is calculated based on the MI and MF parameter values. Number of points = MF - MI + 1. Each point transmitted represents an ion current and as such corresponds to 4 bytes being received by the host computer.

The HP query is used to verify that the CIS Head and the host computer agree on the number of bytes that will be exchanged over RS232 during the histogram scan.

The total number of bytes sent out to the host computer during an histogram scan is obtained multiplying by four the number returned by the HP query.

Parameters: This command is a query, and can only have one parameter format:

#### HP?

#### **Error checking:**

The only acceptable parameter is a question mark. The absence of a parameter (i. e. HP) is treated as a Bad-Parameter error.

#### HSparam, param:0 - 255,\* Description: Histogram Scan Trigger.

Echo: Ion Currents.

Excecute one or multiple Histogram Scans under the present scan conditions. The scan parameter can be set for single, multiple and continuous scanning operation.

A Histogram Scan consists of as a succession of individual mass measurements (see MR command) over a pre-specified mass range.

The mass range for the scan is set in advance with the commands MI (Initial Mass) and MF (Final Mass). The type of detector and noise-floor settings to be used during the scan must be selected in advance with the NF and HV commands. A current value is transmitted for each integer mass value between MI and MF for a total of (MF-MI +1) measurements (See HP command).

For maximum data throughput, ion currents are represented as integers in units of  $10^{-16}$  Amps, and transmitted directly in hex format (four byte integers, 2's complement format, Least Significant Byte first).

#### **Important:**

• Any command received by the CIS Head in the middle of a scan will immediately stop the scan, halt transmission and clear the transmit buffer.

- The detector's zero and the internal scan parameters are checked and corrected at the beginning of each scan resulting in a slight delay before the scan actually starts.
- An extra current value (4 bytes), unrelated to the scan, is transmitted out to the host computer at the end of the scan (Please see HP Command). The current value needs to be accounted for, but it does not need to be stored or displayed.
- The measurements are performed with the detector that is active at the time the scan is triggered.

#### **Parameters:**

**HS**: Continuous scanning mode. The CIS Analyzer produces a continuous string of histogram scans.

A new command must be sent to the CIS Head in order to stop the scanning activity. Once the command is received, the scan is immediately stopped, all transmission is halted and the transmit buffer is flushed (*all remaining data is lost*). The command which stopped the scan is executed right after the buffer is flushed.

**HS0**: Do nothing command. Commonly used to interrupt continuous scanning mode.

**HSparam, param:1-255:** Multiple scans. The number of scans specified by the parameter is executed. Scanning is immediately stopped when a new command is received as in the case of continuous scanning.

HS\*: The default parameter value is used for multiple scan excecution.

Default parameter value: 1 (single scan)

#### Error checking:

Number parameters must be within the specified range, and be integers. No query format is allowed

# **Programming Tips:**

- Any command received by the CIS Head while scanning will immediately stop the data acquisition and clear the transmit buffer. Remember to also clear the computer's receive buffer to reset the communications. The command which stopped the scan is executed right after the CIS Head buffer is flushed.
- It is good practice to perform an analog scan before triggering a large set of histograms to assure the correct tuning (i.e. correct peak locations and widths) of the quadrupole mass spectrometer. Perform a complete Peak

Tuning Procedure as described in the Tuning chapter of this manual if shifts in the peak locations are observed.

- The CIS Analyzer has the ability to store a complete scan in its output buffer. The scan remains in memory until all the data is transmitted out to the host computer over RS232. As a result of the high acquisition rate there might be a delay between the time at which the data is collected and the time at which a complete spectrum is displayed by the host computer. The time lag between data acquisition and display depends on a large number of factors including the scan rate (NF setting), the host computer's processing speed, and the amount of handshaking activity over the RS232 lines.
- Before a new scan starts the CIS Head checks its internal memory to make sure that no data from any previous scan is pending to be transmitted. If data is still pending, the CIS Head must finish transmitting it before the new scan can start. This process may result in a delay from the time the scan trigger is received to the time it actually starts. Using the HS1 command and waiting until the whole scan data stream is transmitted back to the host computer will minimize the problems that are associated to this feature.

#### MFparam, param: 1 - M\_MAX, \*, ? Description: Final M

**Description**: <u>Final Mass</u> (amu) of mass spectra (Analog and Histogram).

Echo: Query Response

Set the Final Mass value (in amu) for Analog and Histogram scans.

The last ion current transmitted during an Analog or Histogram scan corresponds the mass-to-charge ratio specified by the MF parameter.

**Important**: Note that the final mass setting is shared by both histogram and analog scans, and must be an integer number.

Parameters: Three possibilities :

**MFparam, param: 1 - M\_MAX:** The parameter represents the final scan mass in amu units.

The upper mass limit depends on the SRS CIS model number: M\_MAX=100 for CIS100, 200 for CIS200 and 300 for the CIS300 units.

MF\*: The final mass value is set to its default value.

Default value: M\_MAX=100 for CIS100, 200 for CIS200 and 300 for the CIS300.

**MF**?: Query the value of final mass to be measured during scans.

#### **Error checking:**

Number parameters must be within the specified range, and be integers.

The mass value set by MF must always be greater than or equal to the initial mass setting of MI or else a parameter-conflict communications error is generated.

The absence of a parameter (i.e. MF) is treated as a bad-parameter error.

```
MIparam, param: 1 - M_MAX, *, ?
```

**Description**: Initial Mass (amu) of mass spectra (Analog and Histogram).

Echo: Query Response.

Set the Initial Mass value (in amu) for Analog and Histogram scans.

The first ion current transmitted during an analog or histogram scan corresponds the mass-to-charge ratio specified by the MI parameter.

Note that the initial mass setting is shared by both Histogram and Analog scans, and must be an integer number.

#### **Parameters:**

**MIparam, param: 1-M\_MAX:** The parameter represents the initial scan mass in amu units.

The upper mass limit depends on the SRS CIS model number: M\_MAX=100 for CIS100, 200 for CIS200 and 300 for the CIS300.

MI\*: The initial mass value is set to its default value.

Default value: 1.

MI?: Returns the value of initial mass to be measured during scans.

#### **Error checking:**

Number parameters must be within the specified range, and be integers. The mass value set by MI must always be less than or equal to the final mass setting MF or else a parameter-conflict communications error is generated. The absence of a param (i.e. MI) generates a bad parameter error.

#### MRparam, param:0 - M\_MAX

Description: Single Mass Measurement

Echo: Ion Current

Excecute a single ion current measurement at the specified mass setting. The parameter is the **integer** mass number (mass-to-charge ratio in amu units) at which the measurement is performed.

The type of detector and noise-floor settings to be used by the measurement must be selected in advance with the NF and HV commands.

The precision and duration of the measurement are totally determined by the NF parameter value. The scan rates and signal-to-noise ratios for the different NF settings of the electrometer are listed in a table in the Electrometer section of the Electronic Control Unit chapter. The command execution time includes some initial time spent waiting for the Quadrupole Mass Filter and the electrometer response to settle. As usual, a compromise must be made between signal-to-noise and measurement time.

The ion current is expressed in the usual format: 4 byte long, 2's complement integer in units of  $10^{-16}$  A, with Least Significant Byte transmitted first.

# **Important:**

- During a Single Mass Measurement the CIS Head performs a "Miniscan" around the mass requested, and the maximum current value measured is sent out over RS232. The scanning procedure, referred to as Peak-Locking, is designed to measure peak currents for individual masses in a mass spectrum without being affected by drifts in the mass-axis calibration. The Miniscan covers a 0.6 amu range centered at the mass requested, and selects the maximum current from 7 individual measurements performed at 0.1 amu mass increments.
- The detector settings (i.e. type of detector and noise-floor setting) to be used during the measurement must be selected in advance with the NF and HV commands, otherwise, the measurements are performed with the detector settings at the time the scan is triggered.
- The ion detector can be zeroed, prior to the measurements, performing an analog or histogram scan or using the CA command, under the same detector settings. (An offset-correction factor is calculated and stored in memory that it is then automatically used by the Miniscan procedure to correct all ion currents measured). The CA command will also update the internal scan parameters to assure that the correct RF levels (i.e. as specified by the last Peak Tuning procedure) are programmed on the RF rods as a function of mass during the Miniscan.

#### **Parameters:**

**MR0:** The RF/DC voltages are completely shut down and no measurement is performed (no ion current is transmitted back to the host computer).

Use this command format at the end of a set of single mass measurements to make sure the RF/DC are completely turned off.

**MRparam, param:1 - M\_MAX :** A Miniscan is performed around the mass number selected by the parameter and the maximum ion current is sent out over RS232.

The upper mass limit depends on the SRS CIS model number: M\_MAX=100 for CIS100, 200 for CIS200 and 300 for the CIS300.

# **Error Checking:**

The command does not accept query or default parameters.

# **Programming tips:**

- Single mass measurements are commonly performed in sets where several different masses are monitored sequencially and in a merry-go-round fashion. For best accuracy of results, it is best to perform the consecutive mass measurements in a set with the same type of detector and at the same noise floor (NF) setting. Fixed detector settings eliminate settling time problems in the electrometer and in the CDEM's HV power supply.
- It is good practice to perform an analog scan before triggering a long set of measurements to assure the correct tuning (i.e. correct peak locations and widths) of the quadrupole mass filter. Perform a complete Peak Tuning Procedure as described in the Tuning chapter of this manual if shifts in the peak locations are observed.
- The RF/DC voltages are left on at the end of a single mass measurement. Use the MR0 command at the end of a set of measurements and before quitting a program to make sure the RF/DC voltages are left off.

# SAparam, param: 10 - 25, \*, ?

**Description**: <u>Steps per amu of analog scan</u>.

Echo: Query Response.

Set the number of steps excecuted per amu of analog scan. The parameter specifies the number of steps-per-amu.

During an analog scan the quadrupole mass filter is stepped at fixed mass increments through the mass range specified by the MI and MF commands. An ion current is measured after each step and transmitted to the host computer over RS232. SA programs the number of steps excecuted by the CIS Head per amu of analog scan. The fixed mass-increment corresponding to each analog scan step is equal to the inverse of the SA parameter value. Since the CIS Analyzer is usually operated at unit mass resolution throughout its available mass range, the parameter value can also be interpreted as the approximate number of ion currents to be collected for each mass peak in the spectrum.

# **Parameters:**

**SAparam, param: 10-25:** The parameter specifies the number of steps-peramu collected during analog scans.

SA\*: The number of points per amu value is set to its default value.

Default: 10

**SA?**: Query. Returns the SA parameter value currently in use by the analog scans.

#### **Error checking:**

Number parameters must be integers and within the specified range. The absence of a parameter (i. e. SA) is treated as an error.

# SC[param], param: 0 - 255, \*

**Description**: Analog Scan Trigger.

Echo: Ion Currents

Excecute one or multiple analog scans under the present scan conditions.

The scan parameter can be set for single, multiple and continuous scanning operation.

Analog scanning is the most basic data acquisition mode of the CIS Analyzer. During analog scanning the quadrupole mass spectrometer is stepped at fixed mass increments through a pre-specified mass-range. The ion current is measured after each mass-increment step and transmitted to the host computer over RS232. The mass range for the scan is set in advance with the commands MI (Initial Mass) and MF (Final Mass) and the mass-increments are fixed with the command SA. Scan rate and detection limits are pre-programmed by the NF (Noise Floor) setting. A current value is transmitted for MI and after each mass increment through MF for a total of (MF-MI )\*SA+1 measurements (See AP query command).

The ion currents are represented as integers in units of 0.1 fA, and transmitted directly in Hex format (four byte integers with 2's complement format and Least significant byte first).

The type of detector and noise-floor settings to be used during the scans must be selected in advance with the NF and HV commands.

#### **Important:**

- Any command received by the CIS Head in the middle of a scan will immediately stop the scan, halt transmission and clear the transmit buffer.
- The detector's zero and the internal scan parameters are checked and corrected at the beginning of each scan resulting in a slight delay before the scan actually starts.
- An extra current value, unrelated to the scan, is transmitted out to the host computer at the end of each analog scan (Please see AP and TP Commands). The current value does not need to be porocessed or displayed, but it needs to be accounted for by the software communicating with the CIS Head.

• Unless otherwise specified, the measurements are performed with the detector settings that are present at the time the scan is triggered.

#### **Parameters:**

**SC**: Continuous scanning mode. The CIS Head produces a continuous string of analog scans.

A new command must be sent to the spectrometer in order to stop the scanning activity. Once the command is received, the scan is immediately stopped, all transmission is halted and the transmit buffer is flushed (*all remaining data is lost*). The stopping command is executed after the scanning ends.

**SC0**: Do nothing command. Commonly used to interrupt continuous scanning mode.

**SCparam, param: 1 - 255:** Multiple scans. The number of scans specified by the parameter is executed. Scanning is immediately stopped when a new command is received as in the case of continuous scanning.

SC\*: The default parameter value is used for multiple scan excecution.

Default parameter value: 1 (single scan)

# Error checking:

Number parameters must be integers and within the specified range. No query format is allowed for the parameter.

# **Programming Tips:**

- Any command received by the CIS Head while scanning will immediately stop the scan process and clear the transmit buffer. Clear the computer's receive buffer to reset the communications. The new command which stopped the scan is excecuted.
- The CIS Head has the ability to store a complete scan in its output buffer. The scan remains in memory until all the data is transmitted out to the host computer over RS232. As a result of the high data acquisition rate there might be a delay between the time at which the data is collected and the time at which a complete spectrum is displayed by the host computer. The time lag between data acquisition and display depends on a large number of factors including the scan rate (NF setting), the host computer's processing speed, and the amount of handshaking activity over the RS232 lines.
- Before a new scan starts the CIS Head checks its internal memory to make sure that no data from any previous scan is pending to be transmitted. If data is still pending, the instrument must finish transmitting it before a new scan can start. This process may result in a delay from the time the scan trigger is received to the time it actually starts. Using the SC1 command and waiting until the whole scan data stream is transmitted back to the host computer will minimize the problems that are associated to this feature.

• Perform a complete **Peak Tuning** procedure on the CIS Head if the peaks in the spectrum do not appear at their correct mass values (See Peak Tuning section in the Tuning chapter for procedures).

# **Parameter Storage Commands**

# MGparam, param: 0.0000 - 2000.0000,?

**Description**: Electron Multiplier Gain Storage.

Echo: Query Response.

Store a value of electron multiplier (CDEM) Gain, expressed in units of thousands, in the non-volatile memory of the CIS head.

The command is typically used together with the MV instruction to store calibrated sets of [High Voltage and Gain] for the Electron Multiplier.

**Important**: The gain is not used internally by the CIS Analyzer to correct the ion currents measured with the Electron Multiplier, it is simply stored so it can be read and used by any host computer connected to the instrument. For example, RGA Windows automatically divides the ion current readings obtained with the CDEM detector by the gain value value returned by MG?.

As expected, this command is only available in heads with a CDEM option (Option 01) installed (See MO command for details).

#### **Parameters:**

**MGparam, param: 0.0000-2000.0000:** The parameter, representing the CDEM gain in units of thousands, is stored in the non-volatile memory of the head.

MG?: Electron Multiplier Gain parameter query.

#### **Error checking:**

The absence of a parameter (i. e. MG) is treated as an error. No default value is available. A bad-command communications error is reported when this command is invoked in a unit with no CDEM option installed (See MO command).

#### MVparam, param: 0 - 2490,?

Description: Electron Multiplier Bias Voltage Storage.

Echo: Query Response

Store a value of Electron Multiplier (CDEM) Bias Voltage, in units of Volts, in the non-volatile memory of the CIS head.

The command is typically used together with the MG instruction to store calibrated sets of [Bias Voltage and Gain] for the Electron Multiplier.

**Important:** The voltage value is not used internally by the CIS Head to set the bias voltage of the Electron Multiplier, it is simply stored so it can be read and

used by a host computer. For example, RGA Windows uses the High Voltage value returned by MV? to set the bias voltage in the CDEM.

As expected, this command is only available in heads with a CDEM option installed (See MO command for details).

#### **Parameters:**

**MVparam, param: 0-2490:** The parameter, interpreted as a CDEM bias voltage in units of Volts, is stored in the non-volatile memory of the head.

**MV?:** CDEM Bias voltage query.

#### **Error checking:**

The absence of a parameter (i. e. MV) is treated as an error. No default value is available. A bad-command communications error is reported when this command is invoked in a unit with no CDEM option installed.

# XAparam, param:0-10, ?

**Description**: Parameter Address.

Echo: Query Response

Select a specific element within the Parameter Storage Table.

A Parameter Storage Table, capable of storing up to 10 different numeric values (i.e. elements), is part of the non-volatile memory of the CIS Head. The command pair [XA,XV] is used to update or query the elements of that table. XA (where A is for Address) is used first to point at any one element within the table. XV(where V is for Value) can then be used to query or update the value of the element previously pointed to by XA.

The numeric storage available through XA and XV is often used to store a variety of instrument specific calibration data directly into the CIS Head, making the instrument's calibration independent from the host computer that controls it.

**Important:** RGA Windows reserves the use of the first three members of the table (XA1, 2 and 3) for the storage of sensitivity factors for the three Basic Operating Modes of the CIS Analyzer: RGA, CIS-70 and CIS-35 modes. Do not modify those memory locations if you plan to use RGA Windows to control the CIS Analyzer. The three sensitivity factors are measured at the factory for N2 @ 28 amu, and stored in units of  $10^{-6}$ A/Torr before the unit is shipped out. Please consult the General Operation Chapter of this manual for details on the Basic Operating Modes of the CIS Analyzer.

**Important:** The numeric values stored in the Parameter Storage Table are not used internally by the CIS Head in any way during operation. They can only be used by the software programs that operate the CIS Head from the host computer.

#### **Parameters:**

**XA0:** Clear the pointer, so that XV does not access any member of the Parameter Storage Table.

XA0 followed by XV? returns a zero.

**XAparam, param:1-10:** Point to a specific member of the Parameter Storage table so that XV can get to it..

For example, XA4 followed by XV? returns the value of the fourth member of the Parameter Storage Table.

**XA?:** Query the pointer value. Use to verify which member of the table XV will interact with.

#### Error checking:

Parameter must be within specified range and must be an integer. No default value is available.

# XVparam, param:0.0000 - +/-99999.9999, ?

**Description**: Parameter Value.

Echo: Query Response

Query or modify an element of the Parameter Storage Table, at the table location previously specified with the XA command.

Important: Use XA to select the correct table element in advance.

A Parameter Storage Table, capable of storing up to 10 different numeric values (i.e. elements), is part of the non-volatile memory of the CIS Head. The command pair [XA,XV] is used to update or query the elements of that table. XA (where A is for Address) is used first to point at any one element within the table. XV(where V is for Value) can then be used to query or update the value of the element previously pointed to by XA.

The numeric storage available through XA and XV is often used to store a variety of instrument specific calibration data directly into the CIS Head, making the instrument's calibration independent from the host computer that controls it.

**Important:** RGA Windows reserves the use of the first three members of the table (XA1, 2 and 3) for the storage of sensitivity factors for the three Basic Operating Modes of the CIS Analyzer: RGA, CIS-70 and CIS-35 modes. Do not modify those memory locations if you plan to use RGA Windows to control the CIS Analyzer. The three sensitivity factors are measured at the factory for N2 @ 28 amu, and stored in units of  $10^{-6}$ A/Torr before the unit is shipped out. Please consult the General Operation Chapter of this manual for details on the Basic Operating Modes of the CIS Analyzer.

**Important:** The numeric values stored in the Parameter Storage Table are not used internally by the CIS Head in any way during operation. They can only be used by the software programs that operate the CIS Head from the host computer.

# **Parameters:**

**XV0-**+/-**99999.9999:** The parameter is the numeric value that is programmed into the Parameter Storage Table, at the location previously specified with the XA command.

The XA pointer is erased at the end of execution, so that an XA command needs to be strictly used before every XVparam action.

**XV?:** Query the table value previously pointed to by XA.

The XA pointer is erased at the end of the query, so that an XA command needs to be strictly used before every XV?

# **Error checking:**

Parameter must be within specified range. No default value is available.

# **Mass Filter Control Commands**

# MLparam, param: 0.0000 - M\_MAX

**Description**: Mass Lock

Echo: none

Activate the quadrupole mass filter (QMF) and center its pass-band at the mass value specified by the parameter. The QMF is parked at the mass requested but no ion current measurements take place.

The parameter is a real number and the mass increments are limited to a minimum value of 1/256 amu.

The command excecution involves two steps:

- 1. The RF/DC levels corresponding to the mass requested are calculated and set on the QMF rods based on the mass axis calibration parameter values specified by the last Peak Tuning procedure.
- 2. The firmware stabilizes the voltage output of the RF Driver's controller against temperature fluctuations. The result is very stable RF/DC levels that are highly insensitive to the operating conditions of the CIS Head.

**Important:** The RF/DC stabilization algorithm (Step 2 above) remains active as long as no new commands are detected by the CIS Head. Once a new command is received, stabilization stops, and the new command is executed.

Use the ML0 command to turn off the RF/DC bias when finished performing measurements and before quitting the program controlling the spectrometer.

# **Parameters:**

ML0.0000: The RF/DC voltages are completely turned off.

MLparam, param: 0.0040 - M\_MAX: The parameter represents the mass setting for the QMF in amu units. The minimum mass increment value is 1/256 amu.

The upper mass limit depends on the CIS model number: M\_MAX=100 for CIS100, 200 for CIS200 and 300 for CIS300.

# **Error checking:**

Parameters must be numbers within the specified range. The absence of a parameter (i.e. ML) generates a bad parameter error.

# **Error Reporting Commands**

EC?

**Description**: RS232\_ERR Byte Query

Echo: RS232\_ERR Byte.

Query the value of the RS232\_ERR byte. The value of the RS232\_ERR byte is sent to the computer in ASCII format and with a <LF><CR> terminator. RS232\_ERR and bit 0 of STATUS are then cleared to provide a clean error reporting slate.

**Important:** See "Troubleshooting Tools" in the Programming chapter for more details on the use of this query.

Parameters: This command is a query, and can only have one parameter format:

#### EC?

### **Error checking:**

The only acceptable parameter is a question mark. The absence of a parameter (i. e. EC) is treated as a bad-parameter error.

ED?

**Description**: DET\_ERR Byte Query

Echo: DET\_ERR Byte.

Query the value of DET\_ERR and update its value after running a fresh check on the Electrometer (Bit5 of STATUS and the DET\_ERR byte are updated based on the tests results). The DET\_ERR byte value is returned to the computer in ASCII format and with a <LF><CR> terminator. No errors are present as long as the byte value is zero.

Consult the Error Byte Definitions section in this chapter for detatils on the different error bytes of the CIS Analyzer.

Consult the Troubleshooting chapter of this manual for possible causes and solutions to any problems reported.

Always try the query a second time before declaring a hardware problem.

**Important**: The electrometer is not affected by this test, and the detector is restored to its pre-test configuration once the command excecution is over.

Parameters: This command is a query, and can only have one parameter format:

ED?

# Error checking:

The only acceptable parameter is a question mark. The absence of a parameter (i. e. ED) is treated as a bad-parameter error.

EF?	<b>Description</b> : FIL_ERR Byte Query
	Echo: FIL_ERR Byte.
	Query the value of FIL_ERR.
	The FIL_ERR byte value is returned to the computer in ASCII format and with a <lf> terminator.</lf>
	FIL_ERR can only be modified by the "Filament Protection Mode" which constantly monitors the filament while it is emitting electrons. No errors are present as long as the byte value is zero. A non-zero FIL_ERR byte is only cleared after the filament's heater succesfully (i.e. no detected errors) establishes a finite electron emission current.
	Consult the Error Byte Definitions section of this chapter for detatils on the different error bytes of the CIS Analyzer.
	Consult the Troubleshooting chapter of this manual for possible causes and solutions to any problems reported.
	<b>Parameters:</b> This command is a query, and can only have one parameter format:
	EF?
	<b>Error checking:</b> The only acceptable parameter is a question mark. The absence of a parameter (i. e. EF) is treated as a bad-parameter error.
EM?	<b>Description</b> : CEM_ERR byte Query
	Echo: CEM_ERR Byte.
	Query the value of CEM_ERR.
	If no electron multiplier (CDEM) is available, the absence of the CDEM is reported setting bit 7 of CEM_ERR before the byte value is transmitted.
	In all cases, the CEM_ERR byte value is sent to the computer in ASCII format with a <lf><cr> terminator. CEM_ERR and Bit 3 of STATUS are then cleared.</cr></lf>
	This query command can be used to determine whether the CDEM option is installed in the CIS Head being programmed: A CDEM option is available if Bit 7 of CEM_ERR is cleared when the byte is queried (See also MO command).

Parameters: This command is a query, and can only have one parameter format:

#### EM?

#### **Error checking:**

The only acceptable parameter is a question mark. The absence of a parameter (i. e. EM) is treated as a bad-parameter error.

EP?

**Description**: PS\_ERR Byte Query.

Echo: PS\_ERR Byte.

Query the value of PS\_ERR and update its value after running a fresh check on the 24V External Power Supply (Bit6 of STATUS and the PS\_ERR byte are updated based on the tests results). The PS\_ERR byte value is returned to the computer in ASCII format and with a <LF><CR> terminator. No errors are present as long as the byte value is zero.

Consult the Error Byte Definitions section of this chapter for detatils on the different error bytes of the CIS Analyzer.

Consult the Troubleshooting chapter of this manual for possible causes and solutions to any problems reported.

Always try the query a second time before declaring a hardware problem.

Parameters: This command is a query, and can only have one parameter format:

#### EP?

#### **Error checking:**

The only acceptable parameter is a question mark. The absence of a parameter (i. e. EP) is treated as a bad-parameter error.

Q?

**Description**: QMF\_ERR Byte Query.

Echo: QMF\_ERROR Byte.

Query the value of QMF\_ERR and update its value after running a fresh check on the Quadrupole Mass Filter's RF Power Supply (Bit4 of STATUS and the QMF\_ERR byte are updated based on the tests results). The QMF\_ERR byte value is returned to the computer in ASCII format and with a <LF><CR> terminator. No errors are present as long as the byte value is zero.

Consult the Error Byte Definitions section of this chapter for details on the different error bytes of the CIS Analyzer.

#### EQ?

Consult the Troubleshooting chapter of this manual for possible causes and solutions to any problems reported.

Always try the query a second time before declaring a hardware problem.

Parameters: This command is a query, and can only have one parameter format:

#### EQ?

#### Error checking:

The only acceptable parameter is a question mark. The absence of a parameter (i. e. EQ) is treated as a bad-parameter error.

**Description**: STATUS Byte Query.

Echo: STATUS Byte.

Query the value of the STATUS Error byte.

The STATUS byte value is returned to the computer in ASCII format and with a <LF> terminator. No errors are present as long as the byte value is zero.

If one or more bits of the STATUS byte are found set, the specific error bytes (PS\_ERR, DET\_ERR, QMF\_ERR, CEM\_ERR, FIL\_ERR, and RS232\_ERR) must be queried individually to diagnose the problem.

Consult the Error Byte Definitions section of this chapter for detatils on the different error bytes of the CIS Analyzer.

Consult the Troubleshooting chapter of this manual for possible causes and solutions to any problems reported.

**Important**: Since internal checks are constantly being performed within the CIS Head, the STATUS Byte should be queried regularly by the programming software to detect any possible problems.

Parameters: This command is a query, and can only have one parameter format:

#### ER?

#### **Error checking:**

The only acceptable parameter is a question mark. The absence of a parameter (i. e. ER) is treated as a bad-parameter error.

# **Tuning Commands**

#### CE?

**Description**: Calibration Enable Query.

Echo: JP100 setting.

Query the Calibration Enable/Disable jumper (JP100) status. An internal jumper (JP100) on the digital (i.e. top) electronics board of the ECU box can be configured by the end-user to enable/disable the modification of the peak tuning parameters. The CE query command returns the JP100 setting in ASCII format with a <LF><CR> terminator. The two options for the query response are:

- 0. JP100 jumper is in the Calibration Disabled position. Peak Tuning Parameters protected by the JP100 jumper.
- 1. JP100 jumper is in the Calibration Enabled position. Peak Tuning Parameters not protected by the JP100 jumper.

Parameters: Only one possible command format is allowed

CE?

#### **Error Checking:**

The only acceptable parameter is a question mark. The absence of a parameter (i. e. EC) is treated as a bad-parameter error.

Dlparam, param: 0 - 255, \*, ? Description: DI Parameter adjust (Peak Width Tuning command). JP100 Jumper protected.

Echo: Query Response.

Program the value of DI during the Peak Width Tuning Procedure. The parameter (one of four peak tuning parameters) represents the DI value, in bit units.

**Warning:** Please read the Peak Tuning Section of the Tuning Chapter before using this command.

The CIS Head adjusts the DC levels of the quadrupole filter during measurements so that constant mass resolution is automatically available throughout the entire mass range of the spectrometer. The bulk of the DC voltage is supplied by a DC power supply with an output linearly related to the RF amplitude. The rest of the DC voltage (DC\_Tweek) is provided by the output of an 8 bit digital-to-analog converter (DAC). The firmware uses two Peak Tuning Parameters : DI (Intercept) and DS (Slope), stored in the non-volatile memory of the CIS Head, to calculate the 8 bit settings of the DAC according to the linear equation:

DAC8 (m) = DS  $\cdot$  m + DI

where m is the mass in amu, and DAC8(m) is the 8 bit setting at that mass.

# The purpose of the Peak Width Tuning Procedure is to determine the values of DI and DS so that all the peaks in an analog spectrum have the desired peak width (typically 1 amu).

The DI command is used to program the value of the DI peak tuning parameter during the Peak Width Tuning Procedure. The value is saved in the non-volatile memory of the CIS Head and used by the firmware to generate DAC8(m) during measurements.

**Note to Supervisors**: A calibration disable jumper (JP100) available on the circuit board can be used by a supervisor to block any attempt to modify the value of the DI parameter. Supervisors may use this feature to prevent accidental changes in the calibration parameters by inexperienced operators. Setting JP100 will disable peak tuning of the Head.

#### **Parameters:**

**DIparam, param: 0 - 255:** If Calibration is enabled by the jumper JP100 (See CE Command) the value of DI in non-volatile memory is updated and the internal scan parameters used to program the DC during scans and single mass measurements are updated accordingly.

**DI**\*: Use this format to replace the peak tuning parameter with the original factory setting for DI. The factory value is retrieved from memory, and used as the new parameter value to excecute the command as above

DI?: Query. Returns over RS232 the value of DI currently stored in memory.

#### **Error checking:**

The absence of a parameter (i. e. DI) is treated as an error in the parameter. This parameter is protected by an internal jumper (JP100) and a Protection-Violation error will result if the jumper is in the Calibration Disable mode (See CE command).

```
DSparam, param: -0.8500 - +0.8500, *, ?
```

**Description**: DS Parameter adjust (Peak Width Tuning command). JP100 Jumper protected.

Echo: Query Response.

#### **Important:**

CIS300 DS range: -0.850 - +0.850 CIS200 DS range: -1.2750 - +1.2750 CIS100 DS range: -2.5500 - +2.5500

Program the value of DS during the Peak Width Tuning Procedure. The parameter (one of four peak tuning parameters) represents the DS value, in units of bits/amu.

**Warning:** Please read the Peak Tuning Section of the Tuning Chapter before using this command.

The CIS Head adjusts the DC levels of the quadrupole filter during measurements so that constant mass resolution is automatically available throughout the entire mass range of the spectrometer. The bulk of the DC voltage is supplied by a DC power supply whose output is linearly related to the RF amplitude. The rest of the DC voltage (DC\_Tweek) is provided by the output of an 8 bit digital-to-analog converter (DAC). The firmware uses two Peak Tuning Parameters : DI (Intercept) and DS (Slope), stored in the non-volatile memory of the CIS Head, to calculate the 8 bit settings of the DAC according to the linear equation:

DAC8 (m) = DS  $\cdot$  m + DI

where m is the mass in amu, and DAC8(m) is the 8 bit setting at that mass.

The purpose of the Peak Width Tuning Procedure is to determine the values of DI and DS so that all the peaks in an analog spectrum have the desired peak width (typically 1 amu).

The DS command is used to program the value of the DS peak tuning parameter during the Peak Width Tuning Procedure. The value is saved in the non-volatile memory of the CIS Head and used by the firmware to generate DAC8(m) during measurements.

**Note to Supervisors**: A calibration disable jumper (JP100) available on the circuit board can be used by a supervisor to block any attempt to modify the value of the DI parameter. Supervisors may use this feature to prevent accidental changes in the calibration parameters by inexperienced operators. Setting JP100 will disable peak tuning of the CIS Head.

#### **Parameters:**

**DSparam, param: -0.8500 - +0.8500:** If Calibration is enabled by the jumper JP100 (See CE Command) the value of DS in non-volatile memory is updated and the internal scan parameters used to program the DC during scans and single mass measurements are updated accordingly. CIS100 DS range: -2.5500 - +2.5500, CIS200 DS range: -1.2750 - +1.2750

**DS\***: Use this format to replace the peak tuning parameter with the original factory setting for DS. The factory value is retrieved from memory, and used as the new parameter value to excecute the command as above.

**DS**?: Query. Returns the parameter value currently saved in memory over RS232.

#### **Error checking:**

The absence of a parameter (i. e. DS) is treated as an error in the parameter.

This parameter is protected by an internal jumper (JP100) and a Protection-Violation error will result if the jumper is in the Calibration Disabled mode (See CE command).

#### RIparam, param: -86.0000 - +86.0000, \*, ?, none

**Description**: RF\_Driver output @ 0 amu (Peak Position Tuning command). JP100 Jumper protected.

Echo: Query Response.

**Warning:** Please read the Peak Tuning Section of the Tuning Chapter before using this command.

Program the output of the RF\_Driver @ 0 amu during a Peak Posion Tuning Procedure. The parameter (one of four peak tuning parameters) represents the voltage output selected for the RF\_Driver @ 0 amu, in mV.

The magnitude of the RF determines the mass-to-charge ratio of the ions that can pass through a quadrupole mass filter without striking the rods (i.e with stable oscillations). A linear relationship between mass and RF amplitude is one of the most attractive features of these type of filters.

The regulated output of the RF source that powers the quadrupole rods is controlled by, and linearly related to, the voltage output of an RF\_Driver circuit. A linear relation exists between the output of the RF\_Driver, the RF amplitude on the rods, and the mass setting of the filter. The purpose of a Peak Position Tuning Procedure is to determine the voltages that the RF Driver must output at 0 and 128 amu so that all the peaks in an analog spectrum appear in the right position. The RI command is used to program the voltage output of the RF\_Driver @ 0 amu during the Peak Position Tuning Procedure. The value is saved in the non-volatile memory of the CIS Head and used by the firmware to generate the internal scan parameters used to step the RF during scans and single mass measurements .

**Note to Supervisors**: A calibration disable jumper (JP100) available on the circuit board can be used by a supervisor to block any attempt to modify the value of the DI parameter. Supervisors may use this feature to prevent accidental changes in the calibration parameters by inexperienced operators. Setting JP100 will disable peak tuning of the CIS Head.

# **Parameters**:

**RIparam, param: -86.0000 - +86.0000:** If Calibration is enabled by the JP100 jumper (See CE Command), the parameter is saved into the non-volatile memory of the CIS Head and the internal scan parameters used to step the RF during scans and single mass measurements are updated accordingly.

**RI**\*: Use this format to replace the peak tuning parameter with the original factory setting for the RF\_Driver output @ 0 amu. The factory value is retrieved from memory, and used as the new parameter value to excecute the command as above.

RI?: Query. Returns over RS232 the parameter value currently saved in memory.

**RI:** Uses the current parameter value to recalculate the internal scan parameters used to step the RF during scans and single mass measurements. This is often used to compensate against small temperature drifts in the mass scale, caused by drifts in the output of the RF\_Driver.

#### **Error checking:**

The absence of a parameter (i. e. RI) is treated as an error in the parameter.

This parameter is protected by an internal calibration jumper (JP100) and a Protectionviolation error will result if the jumper is in the Calibration Disabled mode (see CE command).

# RSparam, param: 600.0000 - 1600.0000, \*, ?, none Description: RF\_Driver output @ 128 amu (Peak Position Tuning command). JP100 Jumper protected.

Echo: Query Response.

**Warning:** Please read the Peak Tuning Section of the Tuning Chapter before using this command.

Program the output of the RF\_Driver @ 128 amu during a Peak Position Tuning Procedure. The parameter (one of four peak tuning parameters) represents the voltage output selected for the RF\_Driver @ 128 amu, in mV.

The magnitude of the RF determines the mass-to-charge ratio of the ions that can pass through a quadrupole mass filter without striking the rods (i.e with stable oscillations). A linear relationship between mass and RF amplitude is one of the most attractive features of these type of filters.

The regulated output of the RF source that powers the quadrupole rods is controlled by, and linearly related to, the voltage output of an RF\_Driver circuit. Clearly, a linear relation exists between the output of the RF\_Driver, the RF amplitude on the rods, and the mass setting of the filter. The purpose of a Peak Position Tuning Procedure is to determine the voltages that the RF Driver must output at 0 and 128 amu so that all the peaks in an analog spectrum appear in the right position. The RS command is used to program the voltage output of the RF\_Driver @ 128 amu during the Peak Position Tuning Procedure. The value is saved in the non-volatile memory of the CIS Head and used by the firmware to generate the internal scan parameters used to step the RF during scans and single mass measurements.

**Note to Supervisors**: A calibration disable jumper (JP100) available on the circuit board can be used by a supervisor to block any attempt to modify the value of the DI parameter. Supervisors may use this feature to prevent accidental changes in the calibration parameters by inexperienced operators. Setting JP100 will disable peak tuning of the Head.

# **Parameters**:

**RSparam, param: 600.0000 - 1600.0000:** If Calibration is enabled by the JP100 jumper (See CE Command), the parameter is saved into the non-volatile memory of the CIS Head and the internal scan parameters used to step the RF during scans and single mass measurements are updated accordingly.

**RS\***: Use this format to replace the peak tuning parameter with the original factory setting for the RF\_Driver output @ 128 amu. The factory value is retrieved from memory, and used as the new parameter value to excecute the command as above.

**RS**?: Query. Returns over RS232 the parameter value currently saved in memory.

**RS:** Uses the current parameter value to recalculate the internal scan parameters used to step the RF during scans and single mass measurements. This is often used to compensate against small temperature drifts in the mass scale, caused by drifts in the output of the RF\_Driver.

# **Error checking:**

The absence of a parameter (i. e. RS) is treated as an error in the parameter.

This parameter is protected by an internal calibration jumper (JP100) and a Protectionviolation error will result if the jumper is in the Calibration Disabled mode (see CE command).

# **Error Byte Definitions**

The **Error Bytes** described in this section store the results of the firmware-driven checks built into the CIS Head. Use the Error Reporting commands to query the value of the bytes.

Important: No errors are present as long as all bits in the Error Bytes are cleared.

The **RGA Windows** software supports all the Error Reporting commands and reports the errors detected based on their Error Codes.

The Troubleshooting chapter identifies the different problems based on their Error Codes and suggests possible causes and solutions.

Bit	Internal Check <sup>(a)</sup>	ERROR Byte <sup>(c)</sup>	Query <sup>(d)</sup>
7	not used	N.A.	N.A.
6	24V External P/S <sup>(b)</sup>	PS_ERR	EP?
5	Electrometer <sup>(b)</sup>	DET_ERR	ED?
4	Quadrupole Mass Filter <sup>(b)</sup>	QMF_ERR	EQ?
3	Electron Multiplier	CEM_ERR	EM?
2	not used	N.A.	N.A.
1	Filament	FIL_ERR	EF?
0	Communications	RS232_ERR	EC?

(a) CIS Head component checked. Each check involves several tests on the component.

(b) Checks automatically performed upon a power-on-reset

(c) Error bytes that store the results of the tests for each type of internal check.

(d) Error Reporting command that queries the error byte.

**STATUS Error Byte**: General Status byte. Each bit corresponds to a different type of internal check.

Bit	Description	Error Code
7	External 24V P/S error: Voltage >26V.	PS7
6	External 24V P/S error: Voltage <22V.	PS6
5	Not used	
4	Not Used	
3	Not Used	
2	Not Used	
1	Not Used	
0	Not Used	

**PS\_ERR Error Byte:** 24V P/S Error Byte.

Bit	Description	Error Code
7	ADC16 Test failure.	DET7
6	DETECT fails to read +5nA input current	DET6
5	DETECT fails to read -5 nA input current	DET5
4	COMPENSATE fails to read +5nA input current	DET4
3	COMPENSATE fails to read -5nA input current	DET3
2	Not Used	
1	OP-AMP Input Offset Voltage out of range	DET1
0	Not Used	

**DET\_ERR Error Byte:** Electrometer Error Byte.

Bit	Description	Error Code
7	RF_CT exceeds (V_EXT- 2V) at M_MAX	RF7
6	Primary current exceeds 2.0A	RF6
5	Not used	
4	Power supply in current limited mode.	RF4
3	Not Used	
2	Not Used	
1	Not Used	
0	Not Used	

**QMF\_ERR Error Byte:** Quadrupole Mass Filter RF P/S Error Byte.

Bit	Description	Error Code
7	No Electron Multiplier Option installed	EM7.
6	Not Used	
5	Not Used	
4	Not Used	
3	Not Used	
2	Not Used	
1	Not Used	
0	Not Used	

**CEM\_ERR Error Byte:** Electron Multiplier Error Byte.

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Bit	Description	Error Code
7	No filament detected.	FL7
6	Unable to set the requested emission current.	FL6
5	Vacuum Chamber pressure too high.	FL5
4	Not used	
3	Not used	
2	Not used	
1	Not used	
0	Single filament operation.	FL0

FIL\_ERR Error Byte: Filament Error Byte.

Bit	Description
7	Not used
6	Parameter conflict
5	Jumper protection violation
4	Transmit buffer overwrite
3	OVERWRITE in receiving
2	Command-too-long.
1	Bad Parameter received
0	Bad command received

RS232\_ERR Error Byte: Communications Error Byte

# Chapter 6 Tuning

This chapter describes the tuning procedures needed to calibrate the CIS head and assure accurate qualitative and quantitative measurements.

## WARNING!

The Tuning procedures described in this chapter should be performed by qualified personnel only. A mistuned CIS Head will provide Erroneous Readings until it is properly retuned.

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# Introduction

Accurate qualitative and quantitative partial pressure measurements can only be assured by proper tuning of the CIS Head.

Correct calibration of the mass scale is essential during qualitative analysis for the correct assignment of mass numbers to the different peaks.

The mass resolution of the quadrupole mass filter,  $\Delta m_{10\%}$ , must be kept at or under 1 amu to avoid severe overlap between adjacent peaks. Changes in  $\Delta m_{10\%}$  during the measurements (caused by aging, severe contamination and large temperature changes) will cause variations in the sensitivity of the instrument and the shapes of the fragmentation patterns of the molecules, seriously affecting all quantitative measurements.

For careful quantitative analysis, it is important that the sensitivity of the instrument be determined for every gas which may be a component of the system. The sensitivity factors must be obtained under the same operating conditions that will be used during general partial pressure analysis since they depend on many instrumental parameters, including: ionization energy, emission current, mass filter setting, type of detector, etc. For example, the **RGA**, **CIS-70** and **CIS-35** modes of operation will all have different sensitivity factors for the same gas, since they correspond to three different sets of ionizer conditions. Sensitivity factors change as a factor of time due to aging and periodic recalibration is necessary.

The gain of the electron multiplier is mass dependent and needs to be determined prior to performing measurements with the device. The gain characteristics of the multiplier change with time and periodic recalibrations are very important.

The following sections of this chapter describe several tuning procedures designed to assure that all the calibration conditions described above are satisfied prior to a set of partial pressure measurements. All tuning procedures can be executed from RGA Windows using a set of Tuning Commands built into the program. The On-line Help files provide all the information necessary to set up and execute the tuning commands. Users writing their own programs can implement the procedures themselves, using the CIS Command Set and the instructions of this chapter.

All tuning procedures require the ability to introduce pure gases (or a mixture of gases of known composition) into the vacuum system, and a way to measure or calculate pressures. Auxiliary calibrated pressure gauges are required to measure the pressure of the calibration gases. Most gauges that perform indirect pressure measurements are calibrated for a single gas ( $N_2$  or Ar) and a table of conversion factors will be needed to convert the readings for other gases.

When only a few choices of calibration gases are available choose those that are most likely to be encountered during measurements (i.e. usual major components of the residual gas environment of the chamber). Obviously, the correct gases will be needed for sensitivity factor and fragmentation factor determinations.

**Important**: Tuning should only be attempted after the unit has been warmed up (with the filament on and under typical operating conditions) for at least one complete hour.

# **Tuning Options**

The different tuning procedures, including the corresponding RGA Windows commands, are listed in the following table:

Tuning Procedure	RGA Windows Cmd.	Options
	(a)	
Peak Tuning	Peak Tuning	Peak Position
		Peak Width
Sensitivity Tuning	Sensitivity Tuning	Operating
		Modes
Electron Multiplier	Channel Electron	Gain
Tuning	Multiplier	adjustment
_	_	

(a) Head menu command options.

Consult the RGA Windows User's Manual and the RGA On-line Help files included in the program disks for information on the execution of the Tuning Commands built into the program.

# **Peak Tuning Procedure**

## Introduction

During a mass scan, the peaks for the different gas species must be displayed at their correct mass-to-charge ratio location and their peak widths must be less or equal than 1 amu at 10% of peak height. The correct location of the peaks is essential for accurate qualitative analysis, and unity resolution ( $\Delta m_{10\%}=1$  amu) minimizes the overlap between adjacent peaks (i.e. improves the abundance sensitivity of the spectrometer). The location of the mass peaks and their widths can vary with time due to aging of the head. Changes in resolution are particularly serious since they affect the sensitivity of the instrument and introduce errors in the partial pressure measurements.

The Peak Tuning procedures described in this section allow the user to calibrate the mass scale and the resolution,  $\Delta m_{10\%}$ , of the mass spectrometer. The CIS Analyzer has a very solid design and this type of tuning procedures should rarely be needed.

## WARNINGS

The peak tuning procedures should be performed by qualified personnel only. A mistuned CIS Head will give Erroneous Readings until it is retuned properly.

Peak Tuning should only be attempted after the unit has been warmed up (with the filament on and under typical operating conditions) for at least a one hour.

**Peak Tuning** requires a mixture of gases whose mass spectra is well known. In general, a two gas mixture, one with **low mass** peaks and one with **high mass** peaks, is sufficient. The sample is introduced into the vacuum, and the quadrupole mass filter parameters, referred to as Peak Tuning parameters, are adjusted based on the sample analog spectra. The mass scale is adjusted so that <u>all</u> peaks are displayed at their correct mass-to-charge values, and the peak widths,  $\Delta m_{10\%}$ , are adjusted to unity (or smaller) values. The two tuning procedures are referred to as **Peak Position and Peak Width Tuning**, respectively.

<u>Note</u>: A mixture of He, Ar, Kr and Xe inert gases is used at SRS to Peak Tune the CIS Analyzers. The resolution is adjusted to 1 amu and peak tuning parameters are saved into the CIS Head before shipping. The inert gases cover a broad mass range, and they do not interact with the CIS probe (i.e. they do not contribute to its aging.) Perfluorotributylamine is commonly added to the calibration gas to test the mass axis calibration above 200 amu in CIS300 units.

Peak tuning can be easily performed with **RGA Windows** using the Peak Tuning command of the Head Menu. The program provides password protection for locking out the Peak Tuning Parameters so that casual users cannot alter the mass scale calibration or the spectrometer's resolution.

An extra copy of the tuning parameters determined at the factory for the mass filter is saved in the CIS Head before shipping, and those values can be retrieved at any time in case they are necessary. Consult the RGA Windows User's Manual or the On-line Help Files included with the program for details.

**Note to Supervisors:** A calibration disable jumper (JP100) can be configured to block any attempt to change the value of the mass filter settings in the CIS Head. The jumper is located on the top electronics board of the ECU box, next to the microprocessor chip (i.e. biggest component on the board), and its two settings are clearly indicated as CAL DIS and CAL EN. **Supervisors may use this feature to prevent accidental changes in the calibration parameters by inexperienced operators. Peak tuning is completely disabled when the jumper is configured to the CAL DIS setting.** 

**Tip:** Virtually every vacuum system will have detectable amounts of hydrogen (2 amu), water (18 amu), carbon monoxide (28 amu) and carbon dioxide (44 amu). Become familiar with these species and their fragmentation patterns, and use their peaks to quickly check the correct performance of the instrument (i.e. mass scale calibration and mass resolution) while operating the instrument.

#### **General Procedure**

Peak tuning is a simple procedure that requires the introduction of two known gases into the vacuum system. A low mass gas (1-20 amu recommended) is used to adjust the low end of the mass axis, a high mass gas, with a mass-to-charge ratio close to the upper limit of the instrument's mass range, is used to adjust the high end of the mass scale.

Several analog scans are performed on the sample and the peak positions and widths are checked and adjusted as necessary. Changes in resolution affect the sensitivity of the analyzer, and a Sensistivity tuning procedure should always be performed at the end of the peak tuning process.

The entire procedure can be carried out with the help of the Peak Tuning command (Head Menu) of RGA Windows. The program guides the user through the calibration procedure and automatically updates the Peak Tuning Parameters in the CIS Head based on the results of the calibration.

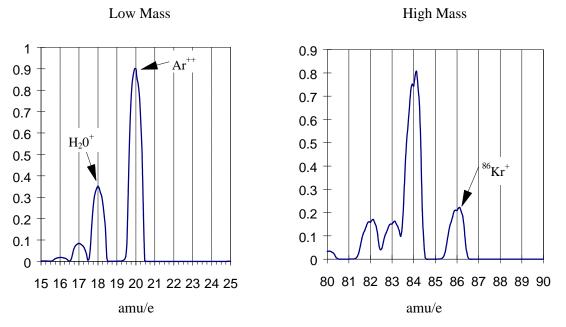
The overall adjustment procedure is very simple and must follow the steps described below:

- 1. Low mass peak position adjustment
- 2. High mass peak position adjustment
- 3. Repeat 1 & 2 in that order one or two more times until no more changes in peak positions are observed.
- 4. Low mass resolution adjustment
- 5. High mass resolution adjustment
- 6. Repeat 4 &5 in that order until no more changes in peak width are observed.

7. Repeat 1 & 2 in that order one or two more times in the case steps 4 & 5 caused changes in peak positions.

**Important**: Collect a fresh analog scan for each step. The peak positions are adjusted such that all peaks throughout the scanning range of the CIS Analyzer fall within +/- 0.25 amu of their known mass-to-charge ratio (This is required to make sure that the **Peak-locking** algorithm used for single mass measurements always finds the mass peak within its search window). The peak width,  $\Delta m_{10\%}$ , must be a constant, and less than 1 amu throughout the whole scan range.

**Example**: The following figure shows the result of peak tuning the CIS Analyzer based on the <sup>18</sup>H<sub>2</sub>O<sup>+</sup> (low mass= 18 amu) and <sup>86</sup>Kr<sup>+</sup> (high mass=86 amu) calibration peaks. All peaks are at their correct mass settings and show absolute resolution values of about 0.9 amu amu. Also note a 20 amu peak in the low mass spectrum corresponding to <sup>40</sup>Ar<sup>++</sup> at 20 (i.e. 40/2) amu.



Users writing their own computer code can write Peak Tuning Commands for their own programs using the Tuning Commands of the CIS Command Set and the instructions of the following two sections.

#### **Peak Position Tuning Algorithms**

The magnitude of the RF determines the **mass-to-charge ratio** of the ions that can pass through a quadrupole mass filter without striking the rods (i.e. with stable oscillations). A linear relationship between mass and RF amplitude is one of the most attractive features of these type of filters. The regulated output of the RF source that powers the CIS Analyzer's quadruple rods is controlled by, and linearly related to, the voltage output of an RF Driver circuit. The RF Driver uses an 18 bit digital-to-analog converter and some additional electronics to program its output voltages.

The purpose of the Peak Position Tuning Procedure is to determine the voltages that the RF Driver must output at 0 and 128 amu so that all the peaks in an analog spectrum

appear at the right place in the mass axis.

The calibrated voltage settings, in mV, are saved in the non-volatile memory of the CIS Head (RI and RS commands respectively) and used by the firmware to generate the internal scan parameters used to step the RF during scans and single mass measurements. Please consult the Tuning Commands List in the Programming chapter of this manual for details on the RS and RI commands.

As described above, the peak position tuning procedure requires the introduction of two known gases into the vacuum system. A **low mass** gas (1-20 amu recommended) is used to adjust the low mass end of the mass axis, a **high mass** gas, with a mass-to-charge ratio close to the upper limit of the scanning range of the CIS Analyzer, is used to adjust the high mass end of the mass scale. Several analog scans are performed, and the values of RI (RF Driver output @ 0 amu) and RS (RF Driver output @ 128 amu) are adjusted until all mass peaks appear at the correct position in the mass scale. An increase in RI causes the low end of the analog spectrum to displace towards lower masses (A small effect is seen at the high masses). An increase in RS results in the spacing between peaks in a scan to decrease (with the largest effect seen at the high mass end).

The formulae used to correct the calibration parameters during peak position adjustment are presented next:

Low Mass Peak Position Adjustment: To displace a low mass peak by a distance  $\Delta m$  amu in the mass axis the value of RI must be modified from its original value RI<sub>0</sub> according to:

$$RI = RI_0 - \Delta m^{-}(RS / 128)$$

Notes:

- The new RI value must fall within the acceptable parameter range of the RI command.
- This change mostly affects the position of the peaks at the low end of the spectrum.
- A decrease in RI shifts the low mass peaks to the right (peaks show up at higher masses), an increase in RI shifts the same peaks to the left (peaks show up at lower masses).

**High Mass Peak Position Adjustment**: To displace a high mass peak,  $m_0$ , by a distance  $\Delta m$  amu in the mass axis modify the value of RS from its original value RS<sub>0</sub> to:

$$RS = RS_0 [m_0/(m_0+\Delta m)]$$

Notes:

- The new RS value must fall within the acceptable parameter range of the RS command.
- Modifying RS affects the spacing between peaks in the spectrum. An increase in RS results in the peaks getting closer together, and a decrease in RS results in the peaks getting further apart from each other.
- The effect is more significant at the higher masses and should have very little effect on the position of the low mass peaks (that is why you do this adjustment second).

- The lower the mass-to-charge ratio of the low mass gas the less this adjustment will effect the position of its peak.
- See that a decrease in RS results in the high mass peaks moving to higher masses (right shift), and vice versa.

**Iterations:** In most cases it will be necessary to repeat the two position adjustments one or two more times until both low and high mass peaks show up at their known positions.

#### **Peak Width Tuning Algorithms**

Constant absolute resolution  $(\Delta m_{10\%})$  in a quadrupole mass filter requires DC voltages linearly related to the mass, with a slight negative offset at low masses (i.e. negative intercept).

The CIS Head dynamically adjusts the DC levels of the quadrupole filter during measurements so that constant mass resolution is automatically available throughout the entire mass range of the spectrometer. The bulk of the DC voltage is supplied by a DC power supply whose output is linearly related to the RF amplitude. The rest of the DC voltage (DC\_Tweek) is provided by the output of an 8 bit digital-to-analog converter (DAC). The firmware uses two Peak Tuning Parameters : DI (Intercept) and DS (Slope), stored in the non-volatile memory of the CIS Head, to calculate the 8 bit settings of the DAC according to the linear equation:

DAC8 (m) = DS m + DI (DC\_Tweek (m) = (DAC8(m) - 128) 19.6 mV)

where m is the mass in amu, and DAC8(m) is the 8 bit setting at that mass.

The purpose of the Peak Width Tuning Procedure is to determine the values of DI and DS so that all the peaks in an analog spectrum have the desired peak width (typically  $\Delta m_{10\%}=1$  amu). The calibrated parameters are saved in the non-volatile memory of the CIS Head (DI and DS commands, respectively) and used by the firmware to generate the internal scan parameters used to adjust DC\_Tweek during scans and single mass measurements. Please consult the Tuning Commands List in the Programming chapter of this manual for details on the DS and DI commands.

<u>Note</u>: The sensitivity of the peak widths to the DC\_TWEEK voltage can accurately be approximated to: -1 amu per 550mV (28 bits). In other words, a 550 mV increase in DC\_Tweek voltage causes a 1 amu decrease in the width of any peak. The DC\_Tweek voltages span from -2.5 to +2.5 V with increments of 19.6 mV per bit (255 total bits). This corresponds to a peak width adjustment range of  $\pm$ -4.5 amu and minimum increments of 0.036 amu per bit.

As described above, the peak width tuning procedure requires the introduction of two known gases into the vacuum system. A **low mass** gas (1-20 amu recommended) is used to adjust peak widths at the low end of the mass scale, a **high mass** gas, with a mass-to-charge ratio close to the upper limit of the scanning range of the CIS Analyzer, is used to adjust the widths at the high end of the mass scale. Several analog scans are performed on the sample and the values of DI (nominally 128) and DS (nominally 0) are adjusted until all peaks show the correct target width.

The formulae used to correct the calibration parameters during peak position adjustment are described next:

**Low Mass Peak Width Adjustment**: To increase the low mass peak width by an amount  $\Delta$ m amus modify the value of DI from its original value, DI<sub>0</sub>, to:

 $DI = DI_0 - \Delta m * 28$ 

Notes:

- The new DI value must fall within the acceptable parameter range of the DI command.
- A change in DI affects the width of all the peaks in the spectrum.
- A decrease in DI results in broader peaks at a rate of 0.036 amu per bit removed.

**High Mass Peak Width Adjustment**: To modify the high mass peak width by an amount  $\Delta$ m amus, modify the value of DS from its original value DS<sub>0</sub> to:

$$DS = DS_0 - 28 * (\Delta m) / m_0$$

Notes:

- The new DS value must fall within the acceptable parameter range of the DS command.
- Modifying DS affects the width of peaks at the higher end of the spectrum. An increase in DS results in the peaks getting sharper, and a decrease in DS results in the peaks getting broader
- The effect is more significant at the higher masses and that is why we do this adjustment second after the width has already been modified by the change in DI.
- If the mass-to-charge ratio of the low mass gas is real low this adjustment will have a small effect on the width of its peak.

**Iterations:** In most cases it will be necessary to repeat the two width adjustments one or two more times until both low and high mass peaks show the desired widths.

**Note:** Changes in the peak positions will also be observed, at a rate of -0.40 amu per amu of increase in peak width, and a Peak Position Tuning procedure will be needed to put the peak positions back where they belong.

**Important**: The Peak Width Tuning Procedure can be used to adjust the resolution of the CIS Analyzer to the ultimate theoretical limit of the mass filter. See the Quadrupole Mass Filter section in the Quadrupole Probe chapter of this manual for details on that limit.

#### Temperature effects on the mass scale calibration

As the temperature of the CIS Head changes two different effects affect the calibration of the mass scale:

- Small temperature changes: Drift in the voltage output of the RF Driver that controls the RF power supply can cause the mass peaks to shift their position in the mass spectrum. In order to correct against this effect, the RF driver output is checked at the beginning of each analog and histogram scan at 0 and 128 amu. The internal calibration parameters, used by the firmware to step the RF during scans, are updated so that the control levels specified by the RI (0 amu) and RS (128 amu) parameter values are correctly set at the present temperature.
- Large temperature changes: The sensitivity of the RF power supply to its controlling voltages might be affected or, more fundamentally, the relationship between mass and RF levels in the filter might change ( for example, if the QMF changes its physical dimensions). In this case a Peak Tuning procedure will be necessary to reestablish the mass axis scale.

## **Sensitivity Tuning Procedure**

All quantitative calculations performed with the CIS Analyzer rely on the assumption that there is a linear relationship between the partial pressure and the corresponding ion current signals from the different gases. Each gas ionizes differently, and its ions make it through the mass filter with different efficiencies. As a result the proportionality constant relating the ion current of a gas to its partial pressure is very dependent on the specific gas.

The **partial pressure sensitivity** of the CIS analyzer to a gas g,  $S_g$ , is defined as the ratio of the change  $(H-H_0)$  in principal mass peak height to the corresponding change  $(P-P_0)$  in total pressure due to a change in partial pressure of the particular gas species.  $H_0$  and  $P_0$  are background values.  $S_g = (H-H_0) / (P-P_0)$ The units of  $S_g$  are of ion current per unit pressure (amp/Torr, for example).

The sensitivity of the instrument changes with time due to aging of the head, and is a strong function of the operating conditions of the instrument. Careful quantitative analysis requires that the sensitivity factor,  $S_g$ , be determined for every gas which may be a component gas in the system being analyzed. The sensitivity factors must be obtained under the same operating conditions that will be used during general partial pressure analysis since they depend on many instrumental parameters, including: ionization energy, emission current, mass filter setting, type of detector, etc. For example, the **RGA**, **CIS-70** and **CIS-35** modes of operation will use different sensitivity factors for the same gas, since they correspond to three different sets of ionizer conditions.

**Important:** In order to separate the gain of the electron multiplier from the intrinsic sensitivity of the CIS head, the sensitivity factors are always defined for Faraday Cup detection. A separate Electron Multiplier Gain Factor, is used to correct the ion signals when the electron multiplier is active. See the Electron Multiplier Tuning section for details.

Regardless of the operating mode, the **partial pressure sensitivity of the CIS Analyzer** to a particular gas is determined following the general steps listed below.

- 1. Refresh the calibration of the electrometer using the Calibrate Detector command (Head Menu) of the RGA Windows program or the CL command of the CIS command set.
- 2. Start with the vacuum system at base pressure (preferably under  $10^{-7}$  Torr).
- 3. Select Faraday cup detection and choose the desired operational parameters for the instrument. Note: In general, this involves choosing one of the three basic operating modes for the CIS Analyzer: RGA, CIS-35 or CIS-70.

- 4. Perform a few analog scans on the chamber to assure that the contributions from all other gases to your measurements can be neglected. Check the quality of the analog spectra, and peak tune the instrument if needed.
- 5. Introduce the pure calibration gas into the vacuum system, at a known or calculable pressure. Recommended pressures are 10<sup>-5</sup> Torr for the RGA Mode and 10<sup>-4</sup> Torr for the CIS Modes.
- 6. Run a few analog or histogram scans on the sample gas to assure the purity of the calibration gas.
- 7. Measure the ion current signal for the principal mass peak of the calibration gas (i.e. usually the parent molecule peak) using the Faraday cup detector. Extract the peak value from spectral scans or measure it directly using the single mass measurement mode of the CIS Analyzer.
- 8. The ratio of this ion current signal to the pressure of the gas is the partial pressure sensitivity factor for the present mode of operation.
- 9. The sensitivity factor, in units of microA/Torr, can be saved into the non-volatile memory of the CIS Head using the (XA, XV) command pair. An individual sensitivity factor can be saved for each one of the three modes of operation of the CIS Analyzer. The parameter value saved is automatically used by RGA Windows as a conversion factor between the ion currents received from the head and the partial pressure units selected by the user. See the CIS Command Set listing for details on the XA and XV commands.

**Important**: The sensitivity factors calculated in this fashion are highly dependent on the ionizer's operating conditions. Change the instrument parameter settings to those of your measurements prior to the calibration if necessary.

**Note**: The underlying assumption when using sensitivity factors in quantitative calculations is that there is a linear relation between the pressure and the corresponding ion current signals of the gases. This assumption is only strictly correct as long as the pressures in the CIS Head are kept below ceratin values:  $10^{-4}$  Torr for RGA Mode and  $10^{-3}$  Torr CIS Mode. Deviations from linearity are to be expected above the recommended maximum pressure values due to space charge effects in the ionizer and ion-neutral scattering interactions in the filter. A more thorough check of the sensitivity factors involves measuring the ion signals over several orders of magnitude of partial pressure to determine the range over which a linear relationship exists. The sensitivity factor for the gas is calculated as the slope of the "signal vs. pressure" response over the linear range.

**RGA Windows** automatically uses partial pressure sensitivity factors stored in the nonvolatile memory of the instrument's head as conversion factors between the ion currents received from the head and the pressure units selected by the user. A Sensitivity Tuning command in the Head menu automates the sensitivity tuning procedure described above and allows the user to recalibrate or change the sensitivity factors very easily. The procedure also prompts the user to select the desired mode of operation before the current measurements are performed. Consult the RGA Windows User's Manual and the On-line Help Files for details on the tuning commands of the program.

The sensitivity factors are always calculated with the Faraday Cup detector. A separate Electron Multiplier Gain Factor, stored in the non-volatile memory of the CIS Head, is used by the RGA Windows program to correct the ion signals when the electron multiplier is turned on (i.e. all data acquired while the electron multiplier is on gets divided by the gain automatically before it is displayed by the program). See the Electron Multiplier Tuning section of this chapter for details.

# **Electron Multiplier Tuning Procedure**

Accurate quantitative measurements with the electron multiplier detector require the determination of the CDEM gain for all the ion peaks being measured. Frequent recalibrations are recommended to correct against aging of the device.

The gain of the electron multiplier (CDEM) in the CIS Analyzer is defined relative to the Faraday Cup output (which is assumed to be mass independent). It can be programmed anywhere from 1 to  $10^7$  adjusting the high voltage applied across the device, is highly mass dependent, and changes with time due to aging.

The electron multiplier gain is easily calibrated since it is possible to measure the same ion current with and without the CDEM. The common method of calibrating the electron multiplier gain for a given mass peak is to measure the peak intensity with the Faraday cup, and then repeat the same measurement with the electron multiplier without changing anything else. The gain of the multiplier is the ratio of the multiplier output current to the Faraday cup output current. (Note that there is no need to change the sign of the electron multiplier signal prior to the division since the firmware automatically reverses its sign before transmitting the value.)

The CIS Head can store a single set of [High Voltage, Gain] values for the electron multiplier in its non-volatile memory. RGA Windows uses the voltage value to bias the CDEM and the gain value to divide the ion currents when the CDEM is turned on. See the HV, MV and MG commands in the CIS Command set for details on the command-level implementation of this procedure.

RGA Windows can automatically program the gain of the electron multiplier for any mass using the automatic Electron Multiplier Gain Adjustment function of the Electron Multiplier command (Head Menu). Select a gain value between 10 and 1,000,000, choose a mass value for partial pressure measurements , and the RGA Windows program automatically calculates the required high voltage setting when the Adjust button of the Electron Multiplier Window is pressed. The HV and gain settings are saved into the CIS Head and used every time the CDEM is turned on. All data acquired while the CDEM is on gets automatically divided by the gain before it is displayed.

# Chapter 7 Maintenance

This chapter describes how to maintain the components of the quadrupole probe. The ECU does not have any serviceable parts and should not require any routine maintenance.

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# Warnings!

- The service information in this chapter is for the use of Qualified Service Personnel. To avoid shock, <u>do not perform any procedures in this chapter unless you are qualified to do so</u>.
- Read and follow all "Safety and Precaution" warnings before servicing the product.
- Dangerous voltages, capable of causing injury or death, are present in this instrument. Use extreme caution whenever servicing any of its parts.
- Carefully follow the instructions in this chapter. Failure to do so might result in serious personal injury and damage to the instrument.
- Do not substitute parts or modify the instrument. Because of the danger of introducing additional hazards, do not install substitute parts or perform any unauthorized modification to the product. Do not use the product if it has unauthorized modifications. Return the product to SRS for service and repair to ensure that safety features are maintained.
- Use only SRS supplied replacement parts.
- Disconnect the ECU box from the probe before servicing any of the probe components.
- Turn off the emission and wait for at least 30 minutes before removing a probe from a vacuum chamber. Serious burns can occur if the probe components are handled too soon.
- Use proper vacuum procedures when handling the probe.
- Avoid contaminating the probe:
  - Work in a clean, dust free area. A clean room compatible environment is best.
  - Do not talk or breath on any of the parts.
  - Wear gloves!
  - Use clean tools during service procedures!
  - Wear face masks, hair covers and no facial make-up.
- Protect the integrity of the vacuum seals:
  - Avoid scratching the metal seals.
  - Do not use nonmetal seals.
- Verify that the vacuum port is electrically grounded before attempting installation of the CIS Head to the vacuum system.

• Stanford Research Systems does not guarantee that the cleaning procedures described in this chapter will completely remove contamination from the probe. In some cases (i.e. depending on the vacuum composition) replacement of the parts might be the only solution to a contamination problem.

## **Probe Bakeout**

Bakeout of the CIS probe is recommended in the following cases:

- 1. After installation of the probe in the vacuum chamber.
- 2. After any prolonged exposure of the probe assembly to open air.
- 2. When background contamination is present in the mass spectra.
- 3. When the performance of the CIS is degraded due to excessive contamination.

When a new part is introduced into a high vacuum system or after the vacuum chamber has been vented up to air, outgassing of molecules (particularly  $H_2O$ ) from the walls usually sets the time it takes the pumping station to achieve the desired base pressure. A bakeout accelerates the outgassing rates and results in reduced pump-down times.

A thorough bakeout helps clean the entire probe and usually results in reduced contribution of the quadrupole sensor to the background signals. Routine overnight bakeouts are required for CIS Analyzers used in the detection of trace level impurities, and are essential for applications requiring the determination of ppm levels of water. For example, the detection of 1ppm of water in 1 mTorr of process gas is best achieved in the pressence of background water levels in the order of  $10^{-9}$  Torr. Such water pressures can only be achieved overnight after a complete probe bakeout.

As the instrument is used, deposits form on the ionizer parts and on the filter components. The performance of the spectrometer is affected by the build-up of electrostatic charge on the contaminated surfaces. The symptoms of the problem are: decreased sensitivity, decreased resolution and, in severe cases, unusual peak shapes. A periodic bakeout helps keep the quadrupole probe clean and minimizes this problem.

#### Warnings

- To avoid fire risk use a good quality heater with a built-in temperature limit switch. High performance, custom-designed heating jackets can be ordered from a number of manufacturers. Consult SRS or your local representative for details.
- To avoid overheating the components use a reliable temperature controller to regulate the bakeout temperature.
- Do not leave the system unattended for long periods of time.
- To avoid damage to the electronic components detach the ECU from the probe during bakeout.
- Do not operate the instrument during bakeouts.
- To avoid burns cover the heater with heavy insulation during bakeout, and do not touch the probe until it cools down after bakeout is over.
- Use only metal gasket seals during bakeout since rubber gaskets might melt!

## Procedure

- 1. The quadrupole probe must be mounted on the vacuum system and at base pressure. Note: If necessary, use an isolation valve to separate the CIS Analyzer from the vacuum process chamber during routine bakeouts.
- 2. Turn off the CIS and disconnect the ECU from the probe.
- 3. Wrap a heating tape or heating jacket around the entire probe and cover with fiberglass insulation if necessary. Make sure the entire probe, including flanges and auxiliary valves, is evenly covered. Note: Isolation and/or Pressure Reducing valves placed between the chamber and the probe must be baked out along with the rest of the quadrupole probe. Follow the manufacturer's instructions regarding maximum bakeout temperatures for the different valves.
- 4. Bake the quadrupole probe to at least 200°C for several hours (i.e. overnight is usually ideal).
- 5. After bakeout wait for the probe to cool down to room temperature before mounting the ECU back on its feedthru flange.
- 6. Check for the correct operation of the probe and run a complete peak tuning procedure if necessary.

# **Ionizer Replacement**

As the CIS Analyzer is used, deposits form on the ionizer parts and the sensitivity of the sensor is degraded. Once the sensitivity of the spectrometer is significantly affected by this buildup it is necessary to completely replace the ionizer. All components of the ionizer should be replaced together at once. The replacement procedure is simple and should only take a few minutes. Use the exploded view of the CIS Ionizer included in the Probe Assembly chapter of this manual for extra help during the entire replacement procedure.

It is recommended that the filament be replaced as well during this procedure since some damage is usually inevitable during handling of its fine wire.

## Equipment

- Ionizer Replacement kit (O100RIC): consisting of anode tube, filament posts, repeller plate, extraction plate, alumina rings, spare screws and e-clips and filament replacement kit (O100RFW or O100RFT, depending on filament material).
- One standard OFHC copper gasket for 2.75" CF Flange. New and Clean!
- Precision flat-head screwdriver: 2.5 mm head.
- Needle nose pliers.
- Latex gloves/powder free.
- Clean, dust-free work area.

#### Procedure

- 1. **Read all warnings** at the beginning of this chapter before attempting to service the probe. Make sure the correct filament spares were ordered with the Ionizer Replacement Kit.
- 2. Set up in advance a clean dust-free working area where to carry out this procedure.
- 3. Turn off the CIS Analyzer and disconnect the ECU from the probe.
- 4. Wait for the probe to cool down for at least 30 minutes after the emission is turned off. Severe burns can result if the probe is handled too soon.
- 5. Turn off the pumping system and pressurize the quadrupole chamber to atmospheric pressure levels.
- 6. Without disconnecting the CIS Cover Tee from the vacuum port, remove the six bolts from the feedthru flange at the end of the probe and slide the entire quadrupole assembly out of the vacuum tube (Note the rotational orientation of the Feedthru Flange before removing the probe assembly from the vacuum system so that the probe can be reassembled in the exact same way at the end of the procedure. Mark the side of the flanges with a permanent marker if necessary.) The ionizer, the filter,

and the detector are now fully exposed and easily accessible. The alumina seal (SRS# 7-00787) remains attached to the CIS Port held in place by a retaining ring. Make sure the crescent washer (SRS Part# 0-778) is still attached to the anode tube. Otherwise, use long pliers or a long wire to remove the washer from the inside of the CIS Cover Tee.

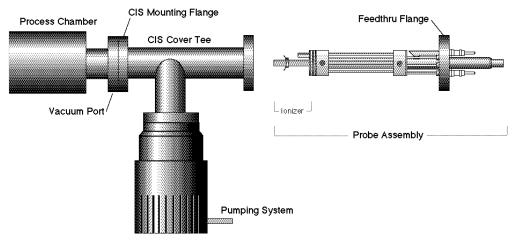


Figure 1 Probe Removal for Ionizer Replacement

- 7. Carry the probe to a clean, dust free area immediately. **Avoid contamination** using handling procedures compatible with high vacuum requirements.
- 8. Hold the probe in a upright position and do a thorough visual inspection of the unit. Check for loose, damaged, misaligned and contaminated components. This is the best time to fix any problems that might be detected.
- 9. Using the clean, flat-head screwdriver disconnect the repeller plate from its filament post, fully exposing the filament and the anode slit. Be careful not to damage the delicate filament if you plan to use it again!
- 10. Remove the filament following the steps and precautions described in the "Filament Replacement" section of this chapter.
- 11. Remove the short alignment rod located right in front of the anode slit (i.e. between the two filament mount baseplates). This rod will not be replaced. Save it.
- 12. Next, remove the two filament mounts. Two short alignment posts are separated from the baseplates during this step. Save them in a safe place so they can be reattached during reassembly.
- 13. The top alumina ring is now free to be removed from the ionizer, exposing the anode's baseplate. Remove the two screws that hold the anode's plate against the bottom alumina ring and pull out the tube assembly. The short alignment rod will be removed during this step, but it does not need to be replaced (Save it!).
- 14. The bottom alumina ring is now free to be removed from the ionizer, exposing the extraction plate. Remove the single screw that holds the plate against the quadrupole's alumina spacer and pull out the plate.

- 15. Use the new parts from the "Ionizer Replacement Kit" to replace the ionizer components. Remove the old e-clips from the loose alignment rods before they are reattached to the plates. The new components are clean and vacuum compatible. Avoid contamination! Replace all components at once! Discard the old ones!
- 16. Use a fresh screw from the ionizer replacement kit to mount the new extraction plate on the alumina spacer.
- 17. Slide the bottom alumina ring into place and attach the new anode tube assembly right on top of it. Do not forget to reattach the short alignment rod to its bottom plate. Use the new screws to fasten its bottom plate to the alignment and connecting rods.
- 18. Once the anode tube is secured in place with two new e-clips, insert the top alumina ring and reattach the filament mounts. Make sure that the alignment rods are back in place and that the filament posts are correctly lined up relative to the anode slit.
- 19. At this point there should be an empty hole that goes all the way through the alumina rings. Place the last alignment rod into it, and use a fresh e-clip to secure it in place.
- 20. Replace the filament following the steps described in the "Filament Replacement" section of this chapter. It is highly recommended that a new filament be used for this step since damage to the delicate wire is usually unavoidable during removal.
- 21. Attach the new repeller plate to the filament post using a fresh screw and inspect visually the entire ionizer assembly to assure the correct alignment of its parts. Once satisfied, mount the probe back on the vacuum system. The anode tube should easily slide into the alumina seal. Check the correct rotational orientation of the feedtrhu flange (i.e. same as it was before disassembly) before tightening the flange bolts.
- 22. A bakeout of the probe is highly recommended before operating the CIS Analyzer again.
- 23. Return the sensitivity of the CIS Analyzer, following the instructions in the Tuning chapter, before using the unit for any quantitative measurements.

# **Filament Replacement**

The filament eventually wears out and needs to be replaced. There is no need to send the CIS Probe back to the factory for this service. The replacement procedure is simple and can be completed in a few minutes by qualified personnel.

**Two kinds of filament materials are available for the CIS Ionizer**: W (standard, O100RFW Replacement Kit) and ThO<sub>2</sub> Ir (optional, O100RFT Replacement Kit). Make sure the correct filament spares are available before the replacement procedure is started.

The filament is very delicate and should be handled with extreme care. The thoria coating in  $ThO_2$  Ir filaments is very delicate and can easily be damaged if the filament is mishandled. W wire becomes brittle over time and often breaks when it is handled. Please read the following handling recommendations before opening the filament's box.

## Handling and care of the filament

- Do not bend, or twist the filament .
- Do not scratch the coating in ThO<sub>2</sub> Ir filaments.
- Do not handle the filament directly with your fingers, use fine tweezers, and hold it by the square tabs only.
- Do not attempt to clean the filament.
- Use only SRS supplied filaments as replacement.

## Equipment

- Filament Replacement kit: consisting of a set of filaments and spare screws.
- One standard OFHC copper gasket for 2.75" CF Flange. New and Clean!
- Precision flat-head screwdriver: 2.5 mm head.
- Needle nose pliers.
- Latex gloves/powder free.
- Clean, dust-free work area.

## Procedure

- 1. **Read all warnings** at the beginning of this chapter before attempting to service the probe.
- 2. Set up in advance a clean, dust-free working area where to carry out this procedure.
- 3. Turn off the CIS and disconnect the ECU from the probe.
- 4. Wait for the probe to cool down for at least 30 minutes after the emission is turned off. Severe burns can result if the probe is handled too soon.
- 5. Turn off the pumping system and pressurize the quadrupole chamber to atmospheric pressure levels.

6. Without disconnecting the CIS Cover Tee from the vacuum port, remove the six bolts from the feedthru flange at the end of the probe and slide the entire quadrupole assembly out of the vacuum tube (Note the rotational orientation of the Feedthru Flange before removing the probe assembly from the vacuum system so that the probe can be reattached in the exact same way it was at the end of the procedure. Mark the side of the flanges with a permanent marker if necessary.) The ionizer, the filter, and the detector are now fully exposed and easily accessible. The alumina seal (SRS# 7-00787) remains attached to the CIS Port held in place by a retaining ring. Make sure the crescent washer (SRS Part#0-778) is still attached to the anode tube. Otherwise, use long pliers or a long wire to remove the washer from the inside of the CIS Cover Tee.

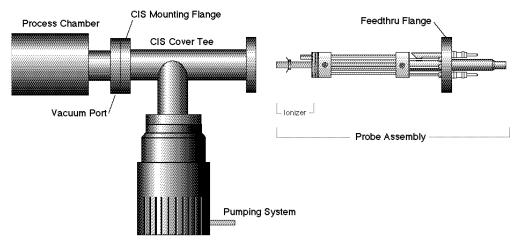


Figure 2 Probe Removal for Filament Replacement

- 7. Immediately carry the probe to a clean, dust-free area and secure it in an upright position. **Avoid contamination** using handling procedures compatible with high vacuum requirements.
- 8. Using the clean, flat-head screwdriver to disconnect the repeller plate from its filament post, fully exposing the filament and the anode slit.
- 9. Before touching the filament, visually inspect the entire ionizer. Check for loose, damaged, misaligned and contaminated components. Some discoloration centered around the anode slit is normal. If problems are detected, a complete "Ionizer Replacement" procedure should be carried out at this time. Otherwise, continue with the filament replacement procedure as follows.
- 10. Inspect the filament, and become acquainted with the way in which it is mounted on the filament posts. Regardless of its composition, the filament consists of a straight wire with a stainless steel square washer (i.e. mounting tab) spot-welded to each end. Precision machined slots define the exact placement of the filament tabs on the posts and a stainless steel screws hold the wire in place.
- 11. Remove the two screws that hold the filament in place and pull the filament (with its tabs) out the ionizer assembly. Discard the old filament.

- 12. Open the filament box and pick up a new filament by one of its tabs with the clean tweezers.
- 13. Very carefully mount the filament on the mounting posts so that the square washers fall into the slots. The filament wire should line up with the anode slit.
- 14. Fasten the filament in place using two new screws. The filament wire is pressed between the post and the square washer.
- 15. Visually inspect the filament alignment and do any adjustments that might be necessary. *The filament should be perfectly straight and centered on the anode slit.* Misalignments result in reduced ionization efficiency and need to be corrected before the probe is mounted back on the vacuum system.
- 16. Reattach the repeller plate to the filament post using a fresh screw and inspect visually the entire ionizer assembly to assure the correct alignment of its parts. Once satisfied, mount the probe back on the vacuum system. The anode tube should easily slide into the alumina seal. Check the correct rotational orientation of the feedtrhu flange (i.e. same as it was before disassembly) before tightening the flange bolts.
- 17. A bakeout of the probe is highly recommended before operating the CIS Analyzer again.
- 18. Retune the sensitivity of the CIS Analyzer, following the instructions in the Tuning chapter, before using the unit for any quantitative measurements.

# **CDEM Handling and Care**

Continuous Dynode Electron Multipliers (CDEM) have a history of high performance and dependability in mass spectrometry applications. By following the simple recommendations described below the user should achieve a long useful lifetime from these detectors.

### Handling and mounting

Handling and mounting of the CDEM should only be performed in a clean vacuum fashion:

- Work on a clean dust-free area. Avoid dust, lint and any kind of particulate matter. •
- Wear talc-free rubber gloves or finger cots. ٠
- Use properly degreased tools. •

Avoid excessive shock, such as from dropping onto a hard surface (Remember that CDEM's are made out of glass).

## **Operating pressure**

The recommended operating pressure is  $2 \cdot 10^{-5}$  or less. The lower the pressure during any operation, the longer the lifetime of the CDEM.

Never apply voltage to a CDEM at pressures above  $10^{-4}$  Torr.

#### **Operating temperature**

The safe operating temperature of a CDEM is less than 100°C.

#### **Bakeout temperature**

The bakeout temperature must be less than 300°C with no voltage applied across the unit.

#### **Operating voltage**

The maximum voltage applied to any CDEM should not exceed 3500V. The CIS Analyzer High Voltage power supply is limited to a 2500V output.

Typical operating range is 1100-2500V. In general, the lower the average signal current drawn from the detector and the lower the operating voltage, the longer the lifetime that will be realized.

#### Initial pump-down

To best maintain the high gain and low dark current properties of electron multipliers it is very important to follow the steps described in the **CDEM Pre-conditioning** section of this chapter during the initial pump-down of the electron multiplier. The procedure serves to degas the detector and stabilize the gain. Failure to follow the recommended steps before powering up the multiplier might result in gain degradation by as much as a factor of ten.

#### Contamination

The active surface of the CDEM can be repeatedly exposed to air without degradation; however, it should not be contaminated with dust, lint or other particles. Organic

substances present in the vacuum system tend to accumulate on the active surface of the multiplier and lead to slow performance degradation (due to reduced secondary emission efficiency).

Oil contamination is a serious problem, and can result in catastrophic destruction of the multiplier: use liquid nitrogen traps with diffusion pumps (particularly for slicone oil based pumps), and molecular sieves traps with mechanical roughing pumps whenever possible.

If the multiplier becomes contaminated it must be cleaned immediately! (See **CDEM Refreshment** procedure in this chapter)

## Storage

CDEM's can be stored indefinitely in a clean dry container such as an air or dry nitrogen-filled "dry box". Double bagging with clean, dust-free zip locked bags also works well as a temporary alternative

# **CDEM Pre-conditioning**

Use the following steps on the first pump-down and initial operation of a new CDEM:

- 1. Pump overnight prior to initial application of voltage.
- 2. Begin operation at the lowest voltage possible, working up to the voltage required to produce observable peaks.
- 3. Limit the initial operation to trace peaks with gradual increase in abundance levels over the first two hours of operation of a new CDEM.

All CDEM's undergo an initial cleanup phase during which absorbed gases are removed from the active surface. If during this time the detector is exposed to high input currents permanent gain degradation by as much as a factor of ten may result. The steps described above help to control initial outgassing and are performed in order to precondition the CDEM. Optimum sensitivity and longer lifetime will result if the recommendations are followed.

# **CDEM** Refreshment

A CDEM contaminated with organic impurities (i.e. pump oil) can sometimes be refreshed following the cleaning procedure described in this section. The CDEM should show a gain improvement after the cleaning.

#### Warning

Stanford Research Systems does not guarantee that this procedure will improve the performance of the CDEM. Use this method as a last resort only.

#### **Materials**

- Ultrasonic cleaner
- Isopropyl alcohol, Electronic grade or better.
- 1000 mL beaker
- Oil-free, dry nitrogen
- Petri dish.
- Clean oven ( higher than 100°C setting).

#### Warning

The fumes from isopropyl alcohol can be dangerous to health if inhaled and are highly flammable. Work in well ventilated areas and away from flames.

#### Warning

Read and follow all directions and warnings of the ultrasonic cleaner regarding the use of organic solvents for cleaning.

#### Procedure

- 1. Fill the 1000mL beaker with isopropyl alcohol.
- 2. Gently place the CDEM into the alcohol.
- 3. Insert the beaker containing the CDEM into the ultrasonic cleaner for 10 minutes
- 4. Remove the CDEM from the isopropyl alcohol and allow it to drain for a few minutes
- 5. Depending on the contamination level, repeat steps 1-4 as needed.
- 6. Blow-dry the CDEM with oil-free dry nitrogen.
- 7. Place the CDEM in a clean Petri dish and dry at 100°C for about one hour.

## **CDEM** Replacement

There is no need to send the CIS Probe back to the factory for this service. The replacement procedure is very simple and can be completed in a few minutes by qualified personnel.

Gain degradation limits the **lifetime** of all electron multipliers. Eventually the gain drops to unacceptable values and the multiplier needs to be replaced. As a rule of thumb, **the CDEM should be replaced when the required gains can no longer be achieved by increasing the bias voltage.** 

#### Warnings

The CDEM is very delicate and should be handled with extreme care. Handling and mounting of the CDEM should only be performed in a clean vacuum fashion:

- \* Work on a clean dust-free area. Avoid dust, lint and any kind of particulate matter.
- \* Wear talc-free rubber gloves or finger cots.
- \* Use properly degreased tools.
- \* Avoid excessive shock, such as from dropping onto a hard surface (Remember that the CDEM is made out of glass).
- \* Use only SRS supplied electron multipliers for replacement.

#### Equipment

- Multiplier Replacement kit: consisting of new CDEM with mounting clamp in place and spare screws.
- One standard OFHC copper gasket for 2.75" CF Flange. New and Clean!
- Precision flat-head screwdriver: 2.5mm head.
- Needle nose pliers.
- Latex gloves/powder free.
- Clean, dust-free work area.

#### Procedure

- 1. **Read all warnings** at the beginning of this chapter before attempting to service the probe.
- 2. Set up in advance a clean dust-free working area where to carry out this procedure.
- 3. Turn off the instrument and disconnect the ECU from the probe.
- 4. Wait for the probe to cool down for at least 30 minutes after the emission is turned off. Severe burns can result if the probe is handled too soon.

- 5. Turn off the pumping system and pressurize the quadrupole chamber to atmospheric pressure levels.
- 6. Without disconnecting the CIS Cover Tee from the vacuum port, remove the six bolts from the feedthru flange at the end of the probe and slide the entire quadrupole assembly out of the vacuum tube (Note the rotational orientation of the Feedthru Flange before removing the probe assembly from the vacuum system so that the probe can be reattached in the exact same way it was at the end of the procedure. Mark the side of the flanges with a permanent marker if necessary.) The ionizer, the filter, and the detector are now fully exposed and easily accessible. The alumina seal (SRS# 7-00787) remains attached to the CIS Port held in place by a c-clip.

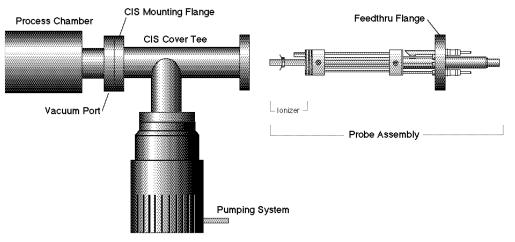


Figure 3 Probe Removal for CDEM Replacement

- 7. Carry the probe to a clean, dust free area immediately. **Avoid contamination** using handling procedures compatible with high vacuum/clean room requirements.
- 8. Hold the probe in a secure upright position and do a thorough visual inspection of the unit. Check for loose, damaged, misaligned and severely contaminated components.

Carefully inspect the ionizer and, if any problems are detected, perform a filament and/or ionizer replacement following the procedures described earlier in this chapter. Inspect the CDEM for signs of arcing and contamination. A blue tinge is an indication of an electrical arc while brown indicates contamination. A "burned" black color may indicate both arcing and contamination have occurred. If excessive contamination is evident take the necessary steps to identify and reduce contamination sources in the vacuum system.

#### 9. CDEM Removal:

Note the position of all parts and their orientation before disassembly! The CDEM consists of a straight, four-channel tube made out of glass, with a cone of the same material attached to the front end. It is held upright, next to the FC, and away from the axis of the analyzer, by a **clamp** and a **clip**. The **clamp** wraps around the multiplier tube, and is connected to a neighboring rod. It assures proper alignment of the cone and provides the electrical connection to the High Voltage power supply. A hole on the side of the FC Shield allows space for mounting the cone very close to the FC top. The **clip** is spot welded to the side of the FC shield and holds the lower end of the multiplier's tube at ground. Chrome electrical coatings, deposited at both ends of the multiplier provide the necessary electrical contacts. A plate (**CDEM Anode**) mounted at the exit of the CDEM collects the secondary electrons. The whole assembly is self-aligning.

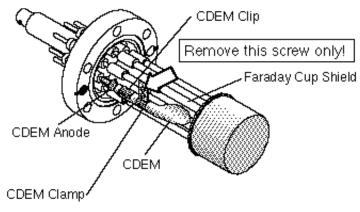


Figure 4 CDEM Replacement

- 10. Using the clean flat head screwdriver, remove the small screw that fastens the clamp to the HV rod and rotate the entire multiplier about its axis until the clamp's end points away from the FC Shield.
- 11. Holding on to the clamp's end, pull the multiplier out of the clip.
- 12. Unpack the new multiplier. Notice that the multiplier has a mounting clamp already in place so there is no need to keep the old one. Avoid contamination.
- 13. Install the new multiplier in place reversing the removal steps. Clip it in place, rotate it about its axis so the clamp fits into the slot in the HV rod, and fasten the clamp with a small screw.
- 14. Visually inspect the assembly and do any adjustments that might be necessary. The CDEM should be standing straight up, centered between its two neighboring rods, and with its cone facing straight into the FC shield's hole. The CDEM anode should be 1-2 mm away from the multiplier's exit, and it can easily be pushed up or down with pliers for correct placement. There should be no electrical contact between the cone (HV) and any part of the probe. If necessary loosen the clamp's screws to do adjustments.
- 15. Once satisfied with the alignment, mount the probe back on the vacuum system and pump it down. Check the correct rotational orientation of the feedtrhu flange (i.e. same as it was before disassembly) before tightening the flange bolts.
- 16. Perform the **CDEM Preconditioning** steps described in this chapter before using the multiplier for actual measurements.
- 17. Calibrate the gain of the new electron multiplier before using the device for quantitative measurements. See instructions in Tuning chapter.

## **Quadrupole filter cleaning**

The quadrupole mass filter is the heart of the mass spectrometer. The sensitivity and resolution of the instrument are ultimately limited by the quality of the quadrupole field between its rods. Deposits on the rods accumulate electrostatic charge and distort the field, resulting in degraded performance. The deposits typically form at the entrance to the mass filter, when the analyzer is operated at high pressures or over long periods of time. They are often seen as a discoloration of the metal but in some cases (i.e. depending on the residual gas composition) they are invisible.

If contamination of the rods is suspected, the first step to take is a one- or two-day long "**Probe Bakeout**". If a bakeout does not eliminate the problem, the quadrupole filter will need to be cleaned. Note that in order to clean the rods, the entire quadrupole head assembly must be taken apart, and unless the exact nature of the deposits is known, the only sure way to remove them will be with a very fine abrasive.

#### Warnings

- The exact alignment of the rods in the quadrupole is essential to the optimum performance of the mass spectrometer.
- Do not scratch the surface of the rods.
- Do not remove excessive amounts of surface material with the abrasives.
- Use clean tools and procedures.

#### Equipment

- Clean, dust-free work area.
- Latex gloves/powder free.
- One standard OFHC copper gasket for 2.75" CF Flange. New and Clean!
- Replacement bowed e-clips (SRS Part# 0-00579-000, 16 count)
- Phillips head screwdriver: P1 head (clean.)
- Precision flat-head screwdriver: 2.5mm head (clean.)
- Precision hex-head screwdriver: 0.050" head.
- Needle nose pliers.
- Micro-Mesh abrasive sheets: 3200 to 12000 grit.
- Ultrasonic cleaner.
- Ultrasonic cleaning solution (high quality, no-rinsing residue).
- 1000mL beaker.
- Oil-free, dry nitrogen.
- Distilled or de-ionized water.
- Petri dish.

• Clean oven (180°C setting).

#### Procedure

- 1. **Read all warnings** at the beginning of this chapter before attempting to service the probe.
- 2. Set up in advance a clean dust-free working area where to carry out this procedure.
- 3. Turn off the CIS Analyzer and disconnect the ECU from the probe.
- 4. Wait for the probe to cool down for at least 30 minutes after the emission is turned off. Severe burns can result if the probe is handled too soon.
- 5. Without disconnecting the CIS Cover Tee from the vacuum port, remove the six bolts from the feedthru flange at the end of the probe and slide the entire quadrupole assembly out of the vacuum tube. The ionizer, the filter, and the detector are now fully exposed and easily accessible. The alumina seal (SRS# 7-00787) remains attached to the CIS Port held in place by a c-clip.

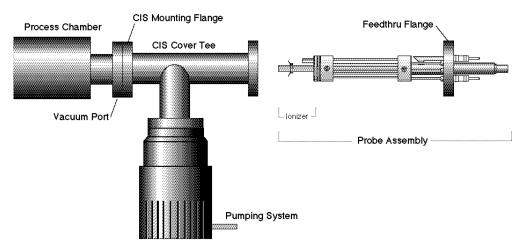


Figure 5 Probe Removal for Quadrupole Filter Cleaning

- 6. Carry the probe to a clean, dust free area immediately. **Avoid contamination** using handling procedures compatible with high vacuum requirements.
- 7. Hold the probe in a secure upright position and do a thorough visual inspection of the unit. Check for loose, damaged, misaligned and severely contaminated components. If excessive contamination is evident take the necessary steps to identify and eliminate the contamination sources in the vacuum system. Carefully inspect the ionizer and, if any problems are detected, replace the filament and/or the ionizer as part of this maintenance procedure. Inspect the CDEM for signs of arcing and contamination. If necessary replace the CDEM as part of this maintenance procedure.
- 8. <u>Head disassembly:</u> Only a rough outline of the complete disassembly of the probe is presented here. Use the **Assembly Drawings** in the Probe Assembly chapter of this manual as a visual aid during the process.

#### Note the placement of all parts and their orientation before disassembly!

Begin by removing the filament following the necessary steps from the "Filament Replacement" procedure described in this chapter. Next, remove the CDEM following steps from the "CDEM Replacement" procedure. Store the multiplier in a dry box or desiccator. Loosen the seven set screws that fasten the 1/8" diameter rods to the feedthru connectors, and remove the bowed e-clip from the exit plate align rod that is located right above the CDEM. You can now separate the entire quadrupole assembly from the detector/feedthru assembly. There are no serviceable parts in the flange. Set it apart in a safe, clean area. Next, separate the ionizer from the quadrupole filter by removing the e-clips that hold the 1/8" diameter rods in place. Store the ionizer electrodes in a safe clean area. Disassemble the quadrupole and set apart the precision ground rods for cleaning.

This is a good time to carefully inspect all the components of the probe for damage and contamination. Replace or clean any parts that are suspect.

**Note**: We do not recommend cleaning the alumina spacers, but if signs of severe contamination are evident, ultrasound clean the two rings for 15 minutes with diluted cleaning solution, and bake them out for 2 hours at 180° C after a thorough rinsing.

9. <u>Rods cleaning</u>: Use the Micro-Mesh abrasives to clean the rods. Begin by using the finest abrasive which will remove the residue, normally 3200 grit. Then consecutively use the next finer grit down to 12000 until the metal surface has a fine polished appearance. **Warning:** Do not remove excessive material from the surface of the precision-ground rods.

After all metal surfaces have been polished, they must be cleaned to remove all the abrasive compound from their surface. Begin by placing the rods in a beaker with the ultrasonic cleaner solution (Note: Any good quality cleaning compound that leaves no residue after rinsing is good for this job.) Place the beaker in the ultrasound cleaner and agitate for 30 minutes. Decant off the cleaning solution, and repeat the above agitation with 4 to 5 consecutive washes with distilled water to remove the cleaning solvent. Following the rinses, blow dry the rods with filtered dry nitrogen and then bake them out in an oven at 180°C for fifteen minutes. **Warning:** After the parts have been cleaned and baked out, they should only be handled wearing powderfree latex gloves.

10. **Head Reassembly:** Only a rough outline of the complete reassembly of the probe is presented here. Use the **Assembly Drawing** in the Probe Assembly chapter and your own notes as a visual aid during this process.

Begin by assembling the quadrupole mass filter. Loosely attach the cleaned rods to the alumina spacers with the spring-loaded, perforated screws. Tighten the screws following the **systematic procedure** described next. Work on one spacer at a time, using a single, small turn of the screwdriver on each screw. After a single round of eight screws, hold a spacer in each hand, and slightly torque the assembly back and forth about its axis to make sure the rods are comfortably seated on their alignment grooves. Repeat this tightening procedure, one spacer at a time, constantly checking the alignment, until the spring washers are completely compressed. Once the

quadrupole assembly is completed, mount the CIS ionizer on it, but do not install the filament at this time. Use new e-clips to hold the ionizer plates against the top alumina spacer, but do not install any e-clips on the bottom grooves of the connection rods at this time. Next, mount the quadrupole assembly on the exit plate of the feedthru flange and secure it in place tightening the connector set screws and inserting the necessary e-clips. Insert the rest of the rods in their correct locations and, while checking for the correct alignment of their grooves and slots, tighten the rest of the feedthru connector set screws. Once the rods are in place, attach the two RF ribbon connectors to complete the quadrupole filter assembly. If the CIS probe has a CDEM, mount the multiplier at this time. Adjust the HV connect rod as needed, and tighten all the necessary screws (The threaded hole on the side slot of the rod lines up with the CDEM Clamp hole, and the top groove should be correctly lined up with the edge of the alumina spacer's alignment hole.) Install new bowed e-clips in all the grooves located above the alignment holes of the bottom alumina spacer. The clips hold the filter tightly against the exit plate. Finally, install the filament and the repeller plate following the necessary steps from the Filament Replacement section in this chapter.

- 11. Visually inspect the probe to make sure all the parts are in place and correctly aligned. Use an ohmmeter to make sure the electrodes are electrically isolated from each other and from the body of the flange (ground).
- 12. Once satisfied, install the probe back in the vacuum system and perform a complete "Probe Bakeout" before using the instrument for measurements.
- 13. Perform a peak tuning procedure on the unit.

# **SRS Probe Refurbishing Service**

The procedures described in this chapter are designed to guide the user through the various steps needed to maintain and/or repair the different components of the CIS probe. These procedures should only be carried out by qualified personnel who fully understand the critical alignment aspects of the instrument. Users who do not feel comfortable or simply do not have the time to go through the different maintenance steps can choose to send the unit back to the factory for a complete Probe Refurbishing Service. Please contact SRS to obtain information on this very fast and convenient service.

**Important:** All users returning a CIS Probe back to the factory for repair and/or service must submit a correctly completed "Declaration of Contamination of Vacuum Equipment" form along with the instrument. The SRS personnel carrying out the repair and service of vacuum equipment must be informed of the condition of the components before any work is done on the parts. A copy of the form is attached at the end of this manual.

# Chapter 8 Troubleshooting

This chapter describes basic troubleshooting procedures for the CIS Analyzer.

### In This Chapter

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Filament's Background Protection mode	8-12

# Warnings

- The service information in this chapter is for the use of Qualified Service Personnel. To avoid shock, do not perform any procedures in this chapter unless you are qualified to do so.
- Read and follow all "Safety and Precaution" warnings before servicing the product.
- Dangerous voltages, capable of causing injury or death, are present in this instrument. Use extreme caution whenever servicing any of its parts.
- Do not substitute parts or modify the instrument. Because of the danger of introducing additional hazards, do not install substitute parts or perform any unauthorized modification to the product. Do not use the product if it has unauthorized modifications. Return the product to SRS for service and repair to ensure that safety features are maintained.
- Use only SRS supplied replacement parts.
- Disconnect the ECU box from the probe before servicing any of the probe components.
- Turn off the emission and wait for at least 30 minutes before removing a probe from a vacuum chamber. Serious burns can occur if the probe components are handled too soon.
- Use proper vacuum procedures when handling the probe.
- Avoid contaminating the probe:
  - \* Work in a clean, dust free area. A clean room compatible environment is best.
  - \* Do not talk or breath on any of the parts.
  - \* Wear gloves!
  - \* Use clean tools during service procedures!
  - \* Wear face masks, hair covers and no facial make-up.
- Protect the integrity of the Vacuum seals:
  - \* Avoid scratching the metal seals.
  - \* Do not use nonmetal seals.
- Verify that the vacuum port is electrically grounded before attempting installation of the CIS Head to the vacuum system.
- The ECU box does not have any serviceable parts and does not require any routine maintenance.
- Contact the factory for instructions on how to return the instrument for authorized service and adjustment.

### Internal Error Detection in the CIS Analyzer

Several firmware-driven checks automatically test the CIS Analyzer when the instrument is turned on, and continuously monitor the internal workings of the unit. A "Background Filament Protection Mode" is activated, when the filament is turned on to protect the delicate filament (and CDEM) from serious accidental overpressures. Several commands can be used to trigger hardware tests on the ECU. Any one of the internal checks just described can detect and report errors, but it is the responsibility of the user to monitor the CIS Head for error reports.

**Note**: Refer to the last section of this chapter for a listing of the built-in hardware checks available in the CIS Head.

**Important:** Even though the hardware tests built into the CIS Head can check the instrument for a large variety of problems, they cannot detect all possible error conditions.

There are two ways to detect the presence of errors in the CIS Analyzer:

- Visually: Inspecting the red LED's on the rear panel of the ECU. The Error LED is turned on whenever a hardware problem is detected, and it flashes twice if a communications error is generated during programming. The Burnt and Leak LED's indicate specific filament problems and are turned on, in addition to the Error LED, whenever the ionizer's emission is internally shut down or not established as requested.
- 2. Error Queries: Querying the Error Bytes with the Error Reporting commands. The "Error Byte Definitions" section of the Programming chapter describes the different error bytes used to store the results of the internal checks. The "Error Reporting Commands" query the error bytes and can be used to quickly diagnose problems.

RGA Windows supports all the Error Reporting commands and constantly monitors the value of the Error Bytes. "Error Report" windows immediately announce all detected errors and identify the different problems based on unique Error Codes. Refer to the next section of this chapter for Troubleshooting information specific to each Error Code.

Users developing their own control software should integrate the Error Reporting commands into their software, and use them periodically. Consult "Error Checking the CIS Analyzer", in the Programming chapter of this manual, for details. Use the Error Byte Definitions listing to obtain the Error Codes for the detected errors.

#### **Error Checking Tips:**

- Keep an eye on all three red (i.e. error reporting) LED's while operating the instrument.
- Diagnose and troubleshoot problems as soon as they are detected.

• <u>RGA Windows users</u>: Use the <u>R</u>etry button of the Error Report Window to trigger one or two fresh error checks on the hardware, before declaring a problem. <u>Programmers</u>: Repeat the query command, if a problem was detected after the last query.

Note that the values of the Error Bytes often change after an Error Byte query command is executed. Some query commands update the byte value after performing a fresh test on the hardware, while others clear error bits, after they are read, to provide a clean error-reporting slate. Please see the Error Reporting Commands list for details.

• Communications errors are different from hardware errors in that the Error LED only flashes two or three times when the problem is detected.

RGA Windows reports communications errors very explicitly as soon as they are detected. Programmers will need to use the EC? command to diagnose the problems.

- Programmers should consider having their software automatically report the Error Code for each problem detected.
- Use the Get Head Info... command in the Head Menu of RGA Windows to get a complete listing of all the Error Bytes at any time.

## **Basic Troubleshooting**

A unique Error Code has been assigned to each one of the fault conditions that can be internally detected by the CIS Head. The Error Codes are used only by the RGA Windows program to report all internally detected errors.

This section lists causes and troubleshooting procedures for all the possible Error Code values available in the CIS Analyzer.

Error Code:	DET1
Type of Error:	Electrometer
Error Message:	Electrometer Error: OP-AMP Input Offset Voltage out of range.
Error Cause:	A large offset input voltage is present in one or both of the I-V converter op- amps. This problem results in electrometer calibration errors, and will cause erroneous ion current readings.
Troubleshooting:	Contact SRS.
Error Code:	DET3
Type of Error:	Electrometer
Error Message:	Electrometer Error: COMPENSATE fails to read -5nA input current.
Error Cause:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a -5 nA input current.
Troubleshooting:	Contact SRS.
Error Code:	DET4
Type of Error:	Electrometer
	Electromator Error COMDENSATE fails to read 15nA input current
Error Message:	Electrometer Error: COMPENSATE fails to read +5nA input current.
Error Message: Error Cause:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a $+5$ nA input current.
0	The logarithmic output of the temperature-compensating picoammeter is not
Error Cause:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a $+5$ nA input current.
Error Cause:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a $+5$ nA input current.
Error Cause: Troubleshooting:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a +5 nA input current. Contact SRS.
Error Cause: Troubleshooting: Error Code:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a +5 nA input current. Contact SRS.
Error Cause: Troubleshooting: Error Code: Type of Error:	The logarithmic output of the temperature-compensating picoammeter is not within the levels expected for a +5 nA input current. Contact SRS. DET5 Electrometer

### 8-6 Basic Troubleshooting

Error Code:	DET6
Type of Error:	Electrometer
Error Message:	Electrometer Error: DETECT fails to read +5nA input current.
Error Cause:	The logarithmic output of the picoammeter is not within the levels expected for a +5 nA input current.
Troubleshooting:	Contact SRS.
Error Code:	DET7
<b>Type of Error:</b>	Electrometer
Error Message:	Electrometer Error: ADC16 Test Failure.
Error Cause:	The 16 bit analog-to-digital converter used to digitize the logarithmic output of the picoammeter reports an output greater than 15 mV when its input is grounded.
Troubleshooting:	Contact SRS.
Error Code:	EM7
<b>Type of Error:</b>	Electron Multiplier
Error Message:	Electron Multiplier Error: No Electron Multiplier Option available in this head.
Error Cause:	A function involving the electron multiplier was invoked in a unit that does not include the CDEM option (Option 01.)
Troubleshooting:	Do not use any of the CDEM related commands in this unit. Upgrade to an Electron Multiplier option (Option 01).
Error Code:	FL6
<b>Type of Error:</b>	Filament "Background Protection Mode".
Error Message:	Filament Error: Unable to set the requested emission current.
Error Cause:	The filament heater was unable to establish the requested emission current.
	The most common reason for this error is overpressure caused by a serious leak in the vacuum system or high a pressure $(>10^{-2} \text{ Torr})$ in the process chamber. In the event of an overpressure detection, the filament emission and the electron multiplier are immediately turned off, and the Error and Leak LED's are turned on to indicate the problem.
	The Leak LED will also be turned on if the filament is excessively worn down or damaged.
Troubleshooting:	The following troubleshooting steps are recommended: Important: Try turning the filament on after each step below. The CIS Head must be connected to a process chamber and operated at pressures under $10^{-2}$ Torr for the instrument to operate correctly.

	Check the pressure in the vacuum system. Check for leaks, open valves or large sources of contamination that might be causing severe overpressures.
	If the pressure is OK ( i.e. $<10^{-2}$ Torr in the ionizer, $<10^{-4}$ Torr in the quadrupole volume), check for shorts in the ionizer assembly. Using an ohmmeter check the conductance between the ionizer's connectors and the vacuum system. (The ionizer feedthrus can be easily identified using the drawings in the Probe Assembly Chapter).
	If a short is detected, remove the probe from the vacuum system, inspect the ionizer and fix any shorts. Note: Use the information in the Maintenance chapter to remove the repeller and/or service the ionizer.
	If no short is detected, the filament might be damaged or worn out. Inspect the filament (best with a magnifying glass) and look for damage or contamination on the wire. If a problem is suspected, replace the filament using the procedure in the Maintenance chapter.
	If the filament still does not turn on, contact SRS.
Error Code:	FL7
Type of Error:	Filament "Background Protection Mode".
Error Message:	Filament Error: No filament detected.
Error Cause:	No filament was detected after an attempt was made to establish emission current in the ionizer. The Error and Burnt LED's are turned on by this error.
Troubleshooting:	A probe must be connected to the ECU. The probe must have a filament. The filament must be intact. Use an ohmmeter to check continuity across the filament (The two filament feedthrus can be easily identified using the drawings in the Probe Assembly Chapter). The probe must be correctly connected to the ECU. Tighten the locking screws of the box
Error Code:	PS6
Type of Error:	24VDC P/S.
Error Message:	External 24V P/S error: voltage <22V.
Error Cause:	Voltage output of 24VDC Power Supply is under the acceptable 22-26V DC range. The Power LED is turned off and the Error LED is turned on instead.
Troubleshooting:	Check the voltage output of the external power supply with a voltmeter. Adjust the voltage to 24 V in adjustable external supplies or replace the power supply altogether if necessary. Contact SRS if your unit has a built-in power supply (Option 02).

### 8-8 Basic Troubleshooting

Error Code:	PS7
Type of Error:	24VDC P/S.
Error Message:	External 24V P/S error: Voltage >26V.
Error Cause:	Voltage output of 24VDC Power Supply exceeds the acceptable 22-26V DC range. The Power LED is turned off and the Error LED is turned on instead.
Troubleshooting:	Check the voltage output of the external power supply with a voltmeter. Adjust the voltage to 24 V in adjustable external supplies or replace the power supply altogether if necessary.
	Contact SRS for units with a built-in power supply (Option 02).
Error Code:	RF4
Type of Error:	Quadrupole Mass Filter RF P/S.
Error Message:	RF P/S Error: Power Supply in current limited mode.
Error Cause:	The circuit that drives the primary of the RF Transformer is in current limited mode.
Troubleshooting:	Check for a short in the quadrupole connections of the probe.
	Consult SRS if no obvious shorts are detected.
Error Code:	RF6
Type of Error:	Quadrupole Mass Filter RF P/S.
Error Message:	RF P/S Error: Primary Current exceeds 2.0 Amps.
Error Cause:	The circuit that drives the primary of the RF Transformer is delivering an unusually large current.
Troubleshooting:	Check for a short in the quadrupole connections of the probe.
	Consult SRS if no obvious shorts are detected.
Error Code:	RF7
Type of Error:	Quadrupole Mass Filter RF P/S.
Error Message:	RF P/S Error. RF_CT exceeds (V_EXT-2V) at M_MAX. (M_MAX=100 for the CIS100, 200 for CIS200 and 300 for CIS300)
Error Cause:	The RF P/S goes out of regulation when the quadrupole mass filter is set to M_MAX amu.
	Note: When RGA Windows detects an RF7 error, it automatically tests the CIS

can then choose to proceed (i.e. ignore the problem at own risk) or can get information on how to Troubleshoot the problem by pressing the Help button.

This problem should not be ignored since it can be associated to erroneous readings at high masses.

**Troubleshooting:** Is the quadrupole probe connected to the ECU box? • The ECU's RF P/S cannot operate in the absence of a probe. Is the CIS Cover Tee in place? • The CIS Analyzer will not operate without the CIS Cover Tee in place. Are the electronics warmed up? • The RF P/S is optimized at the factory in a *completely* warmed up ECU box. Cold operation reduces the efficiency of the RF P/S and can, in some cases, lead to an RF P/S error. To *completely* warm up the CIS electronics (and match the factory conditions) turn on the filament to the desired emission current and let the electronics warm up for at least 15 minutes. Once the warm-up time has elapsed, try to operate the analyzer to see if the problem has disappeared (for example, trigger a scan over a large mass range.) If the problem is still present, warm up the unit further by setting the quadrupole mass filter to  $\geq$ 50 amu for 15 minutes (see Programming the Quadrupole Mass Filter in the Programming chapter of the manual). It is good practice to keep the filament on as long as the instrument is turned on ans safe operating pressures are present. Operating the filament keeps

the electronics and the probe *completely* warmed up and assures reproducible readings at all times. The filament is burn-out resistant and will operate continuously for a long time.

Note: During the warm-up period, RGA Windows users should see that the mass range over which the instrument can be operated reliably increases with time until it goes beyond the user's requested scan range. No more warnings are posted beyond that point. If no improvement in the mass range is seen as the unit warms up then go on with the troubleshooting procedure.

- Is the probe correctly connected to the ECU box?
- Try disconnecting and reconnecting the ECU box to the probe. Securely tighten the thumb screws to make sure good contact is made between the RF P/S and the quadrupole rod pairs.
- I none of the above steps solves the problem, consult SRS, or your local representative, for additional help.

## **Built-in Hardware Checks**

Several firmware driven hardware checks are built into the CIS Head. Some checks are automatically performed as soon as the unit is powered up (i.e. Power-on checks), and others are activated when the emission from the ionizer is turned on (i.e. filament's Background Protection Mode). Most of the checks can be triggered by query commands (i.e. Error Reporting Commands) at any time.

This section describes the internal checks, based on the hardware components they test. The information presented includes:

- The type of check performed.
- The Status bit affected (See Error Byte Definitions).
- The specific Error Byte affected (See Error Byte Definitions).
- The query command that can be used to trigger the check (see Error Reporting Commands.)
- The prefix of the Error Codes associated to the check.
- Is this a Power-on check?

#### 24V DC Power Supply

STATUS Bit affected:	6
Error Byte affected:	PS_ERR
Error Reporting Command:	EP?
Error Codes prefix:	PS
Power-on check?	Yes

The output of the external 24 V DC Power Supply must be between 22 and 26 Volts at all times while the instrument is in operation. Its voltage level is automatically tested when the instrument is turned on and can be checked at any time with the query command: EP?. The same test is performed in units with built-in power modules (Option 02) to check the voltage output of the internal switching power supply. If a voltage out of the allowable range is found, the POWER LED is turned off, the red Error LED is turned on, Bit 6 of STATUS is set, and PS\_ERR is updated to indicate the specific problem.

#### Electrometer

STATUS Bit affected:	5
Error Byte affected:	DET_ERR
Error Reporting Command:	ED?
Error Codes prefix:	DET

Power-on check?

Yes

Ion currents from the Faraday Cup or the electron multiplier are measured with a very sensitive logarithmic electrometer. The voltage levels are digitized with a 16 bit analog-to-digital converter (ADC16) and turned into current values using a digital logarithmic interpolation algorithm that calculate the currents from an internal calibration curve. The Electrometer/ADC16 system is automatically tested by the firmware on power-on and can be checked at any time with the query command: ED? Several tests are performed during the check:

- 1. The ADC16 input is grounded and its digital output is measured to make sure it corresponds to less than 15 mV.
- 2. A current of +5nA is injected into the electrometer and the output is read and compared to expected values.
- 3. The same test is repeated with -5nA of input current.

If an error is detected: Bit 5 of STATUS is set, DET\_ERR is updated, and the Error LED is turned on. The detector is reconfigured to its pre-check configuration once the tests are over.

#### **Quadrupole Mass Filter RF P/S**

STATUS Bit affected:	4
Error Byte affected:	QMF_ERR
Error Reporting Command:	EQ?
Error Codes prefix:	RF
Power-on check?:	Yes

The RF power supply that biases the rods of the Quadrupole Mass Filter is thoroughly checked upon a power-on reset and can be tested at any time using the query command: EQ?

The RF amplitude is set at the level corresponding to the maximum mass value for the head (100 amu for CIS100, 200 amu for CIS200 and 300 amu for CIS300) and the following tests are done:

- The voltage on the primary of the RF transformer is checked to make sure it is at least 2V lower than the DC voltage used to power the instrument.
- The amount of current flowing through the primary of the transformer is checked to make sure it is less than 2.0 Amps.
- RF\_PRI is also checked (against RF\_SET) to make sure the driver is not in current limiting mode.

If a problem is detected in any of these checks Bit 4 of STATUS is set, QMF\_ERR is updated, and the Error LED is turned on.

#### Filament's Background Protection mode

STATUS Bit affected:	1
Error Byte affected:	FIL_ERR
Error Reporting Command:	EF?
Error Codes prefix:	FL
Power-on check?	No

The filament is by far the most carefully protected component of the CIS Analyzer.

When electron emission current is requested, the CIS Head biases the ionizer and activates the filament's heater until the desired electron current is achieved. The Filament LED is turned on to indicate the presence of emission and the filament's "Background Protection Mode" is automatically enabled to continuously (675Hz) monitor the operation of the filament.

The conditions detected during Background Filament Protection are:

- Filament not present (burnt or not installed)
- Unable to set the requested emission current.

If any of these two error conditions is detected while the filament is on, the heater is immediately shut down and the specific problem is reported as follows:

- 1. Bit 1 of STATUS is set.
- 2. FIL\_ERR is updated
- 3. Filament LED is turned off.
- 4. Error LED is turned on.
- 5. The Leak and Burnt LED's are updated according to the specific problem encountered.
- 6. The Electron Multiplier is also turned off.

Once detected, a filament error can only be cleared by successfully turning on the filament to a finite electron emission current. It is always good practice trying again, if the first attempt to get emission failed. Please refer to the FL command in the "CIS Command Set" to get more details on the operation of the filament.

# Chapter 9 Circuit Description

This chapter describes the electronics circuits located inside the Electronics Control Unit of the CIS Analyzer.

The same basic electronics are used in the RGA and CIS line of quadrupole mass spectrometers manufactured by Stanford Research Systems.

There are no electronics components inside the CIS Probe.

This information is provided for the exclusive use by qualified technical personnel during service and repairs.

#### Warnings

- The ECU is to be serviced by qualified technical personnel only. There are no user serviceable parts inside.
- Dangerous voltages, capable of causing injury or death, are present in this instrument. Use extreme caution whenever the ECU covers are removed. Do not remove the covers while the unit is plugged in.
- Always disconnect the power before removing the covers of the ECU.

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## **Overview of the RGA**

The SRS RGA is a mass spectrometer consisting of a quadrupole probe, and an Electronics Control Unit (ECU) which mounts directly on the probe's flange.

The quadrupole probe is a mass spectrometer sensor consisting of an ion source, a quadrupole mass filter, a Faraday cup and an optional electron multiplier (Option 01). Ions are created from the residual gas of a vacuum system by electron impact ionization. Ions of a particular mass-to-charge ratio have stable trajectories through the mass filter, depending on the DC and RF voltages applied to the quadrupole rods. The selected ions are collected in a Faraday cup or multiplied in an electron multiplier.

The ECU box (3" x 4" x9") contains all the electronics necessary to operate the RGA. It connects directly to the probe's feedthru flange, and also to a host computer. Its regulated power supplies set the ionizer voltages, the filament's electron emission current, the high voltage across the electron multiplier and the RF/DC levels on the quadrupole filter. A logarithmic transconductance amplifier converts ion currents into voltages that are read by an A/D converter. A microprocessor and control firmware direct the operations of the spectrometer, the data acquisition, and the communications through the RS232 serial link.

The electronics operate from a single  $24 \pm 2 \text{ V}$  DC power supply. The peak current requirement is about 2A. A universal input (90-264VAC, 47-63Hz) switching power supply is provided as an option (Option 02) so that the system may be operated directly from a line cord.

# **Circuit Description**

#### **General Description**

The specifications and features of the many circuits that drive the RGA are determined by characteristics of the quadrupole mass spectrometer such as:

- the ionizer settings available to the user,
- the characteristics of the quadrupole mass filter,
- the magnitude of the ion current levels detected during measurements,
- an optional electron multiplier.

The ionizer consists of a dual thoriated iridium filament heated by a current of about 3A at 1.7V. The filament and the associated repeller shield are held at a negative potential between -13 and -97V, determined by the electron and ion energy settings (Vrepeller = ion energy - electron energy). Electrons from the filament are accelerated toward the anode grid cage, which is held at a positive potential of +8 (low) or +12V (high). The 25-105 eV electrons ionize residual gas molecules in the grid cage. The ions are then injected into the mass filter, aided by the focus plate, which is held at a negative potential of about -90VDC (adjustable from 0 to -150V) to optimize the throughput of the mass filter and to deflect electrons.

The mass filter requires two potentials be applied to two pairs of rods. The potentials are given by:  $Vx/y(t) = +/- [U + V_0 \cos(\omega t)]$ , where U is a DC potential amplitude and  $V_0$  is an RF potential amplitude. The physics of the quadrupole mass filter show that the resolution of the instrument improves with the frequency  $\omega=2\pi f$  ( the ions interact with the RF field for more cycles ), however the required RF amplitude increases with the square of the frequency. An operating frequency f=2.7648 MHz requires an RF amplitude,  $V_0$ , of 1284 V (2568 Vpp) to measure ions with mass of 300 amu (8.56 Vpp/amu.). The ratio of DC to RF amplitude, U/V<sub>0</sub>, controls the fractional mass resolution, M/ $\Delta$ M. For f= 2.7648 MHz, the DC potential, U, will +215VDC on one rod pair, and -215 VDC on the other, for masses of 300 amu.

The rod structures are primarily capacitive, so to reduce the circuit drive requirements, the rod reactance is resonated with an inductance. The advantages of this resonant drive circuit is the primary reason for operation at a fixed frequency.

Ions with the selected mass are collected in a Faraday cup. The ion currents can be very low, a few femtoamps, and have a very large dynamic range, up to about 100 nA. A log I/V converter capable of simultaneously measuring ion signals over several orders of magnitude is a natural choice. Low Pass filtering of the signal is essential in the presence of the large rod potentials.

To improve the detection limit, by overcoming the noise floor imposed by the I/V converter, an electron multiplier may be used. The electron multiplier needs to be biased with as much as -2500VDC to provide gains as large as  $10^7$ .

#### **Circuit Boards**

There are two main PCBs inside the ECU package.

The top PCB has the CPU, RS232, digital ports, the analog electronics for A/Ds and D/As, and the RF amplitude detection circuit. A small vertical PCB which holds the log I/V converter connects to the top PCB. A second small vertical PCB holds the electron multiplier's HV supply. A third small vertical PCB is used to pass signals and power between the two main PCBs.

The bottom PCB holds all of the power electronics for the RF, filament heater, ionizer bias, and system power supplies.

Four PCB's between the two main boards are used:

- 1. To connect to the probe.
- 2. For the I/V converter.
- 3. To pass signals between the main boards.
- 4. To supply High Voltage to the CDEM.

### **Description of Schematics**

#### Schematic name: QMSE\_T1

#### Microprocessor

An MC68HC11E9 microcontroller is used to control the system and to communicate with the host computer. This central processing unit (CPU) also has RAM, ROM, EEPROM, UART, octal 8-bit A/D converter, counter timers, and a multiplexed address/data bus to accommodate an external 32Kx8 RAM. The ROM is used for program storage, the RAM for data storage, the EEPROM contains calibration values for the particular unit.

The power-up reset and power-fail interrupts are provided to the CPU by U100, a MAX705.

#### **Digital I/O Ports**

The microprocessor's I/O ports are assigned as follows:

PA0 PA1 PA2 PA3 PA4 PA5 PA6 PA7	-RTS -BUSY CAL_EN LE_DAC18 -CS_DAC8 LD_LEDS LD_MPX LD_MISC	Low indicates that the host computer is ready for RS232 data. Low indicates that the 16-bit signal A/D converter is busy. High enables modification of EEPROM calibration data. Set high to latch SPI data into 18-bit (RF control) D/A converter. Strobe low to assert CS to octal 8-bit DAC. Strobe high to load LED data into output register. Strobe high to load multiplexer data into output register. Strobe high to load multiplexer data into output register.
PB 0-7	A8-15	High address bits for external RAM
PC 0-7	A0-7 & D0-7	Multiplexed address and data bits for external RAM
PD0	RXD	RS232 data received from host computer.
PD1	TXD	RS232 data transmitted to host computer.
PD2	SPI_IN	Serial peripheral interface data from 16-bit A/D converter.
PD3	SPI_OUT	Serial peripheral interface data to 8 and 18-bit D/A converters.
PD4	SPI_CLK	Serial peripheral interface data clock.
PD5	-CTS	Low to allow host computer to send RS232 data.
PE0	+24/6	A/D input: +24VDC supply divided by 6.
PE1	RF_CT/5	A/D input: RF primary voltage divided by 5.
PE2	RF_PRI	A/D input: RF primary current: 1V/400mA
PE3	GND	A/D input: 0 VDC
PE4	GND	A/D input: 0 VDC
PE5	FOCUS_I	A/D input: focus plate ion current: 1V/5µA
PE6	FIL_DUTY	A/D input: filament heater duty cycle: 1V/20%
PE7	FIL_PRI	A/D input: filament heater primary current: 3V/A

In addition to the I/O port on the microcontroller, there are three 8-bit digital shift registers which are loaded via the SPI, then strobed by a LD bit to transfer data to the parts' output registers. The three digital output ports are assigned as follows:

#### The LED port

- Q0 Set high to light an LED to indicate that the +24V power supply is "okay".
- Q1 Set high to light an LED to indicate that the filament is "on".
- Q2 Set high to light an LED to indicate that the unit is degassing.
- Q3 Set high to light an LED to indicate that the CDEM high voltage is "on".
- Q4 Strobe high to set I/V converter to LOG mode
- Q5 Set high to light an LED to indicate that there has been an error.
- Q6 Set high to light an LED to indicate that the pressure is too high.
- Q7 Set high to light an LED to indicate that the filament is burnt-out.

#### The MPX port

Q0	CAL_0	LSB of current detector calibration attenuator multiplexer.
Q1	CAL_1	Middle bit of current detector calibration attenuator multiplexer.
Q2	CAL_2	MSB of current detector calibration attenuator multiplexer.
Q3	MPX_0	LSB of 16-bit A/D converter's input multiplexer.
Q4	MPX_1	Middle bit of 16-bit A/D converter's input multiplexer.
Q5	MPX_2	MSB of 16-bit A/D converter's input multiplexer.
Q6	EMIT_CTL	Filament heater duty cycle control: 0=direct, 1=regulate
Q7	GRID_SEL	Low for low grid potential, high for high grid potential.

#### The MISC port

Q0	RF_ON	Low to turn-off RF, high to turn on.
Q1	IONS_OFF	Pulse high for 50 ms to reset relay and ground FC
Q2	IONS_ON	Pulse high to set latching relay so FC signal enters I/V.
Q3	MULT_OFF	Pulse high to ground e-multiplier anode.
Q4	MULT_ON	Pulse high to connect e-multiplier to I/V.
Q5	LINEAR	Pulse high for linear I/V converter (offset calibration).
Q6	R/-C	Set high to read 16-bit A/D, low to initiate conversion.
Q6	CS_VETO	Set high to prevent (or release) -CS to 16-bit A/D converter.

#### Clocks

The master clock of the system is an 11.0592 MHz crystal oscillator. This frequency was selected for the following reasons:

- 1. It is close to the maximum operating frequency for the 68HC11 (12 MHz).
- 2. When divided by four it provides a usable RF frequency (2.7648 MHz).
- 3. When divided by 128 it provides a clock for switching power supplies (86.4 kHz).
- 4. When divided by 256 it provides a clock for the high voltage DC-DC converter.
- 5. When divided by 384 it provides an exact PC baud rate (28.8 kbaud).
- 6. When divided by  $2^{14}$  it provides a A/D convert rate (675 Hz).

Since the A/D convert rate is a sub-multiple of all the other system clocks, crosstalk from the clocks will be synchronous, and will generate a fixed offset to the signal (which may be measured and subtracted) instead of noise.

The clock division for the 28.8 kBaud rate is done by the microcontroller; the rest of the clock division is done by U108, a 74HC4020 14-stage ripple divider. The 172.8 kHz square wave is formed into a 100 ns wide pulse to be used as a switching power supply sync pulse, which is divided by two by the power supply controller. U109, a 74HC74 dual D-type flip-flop, provides the complimentary drive required for the RF and high-voltage inverter circuits. Either of these drives may be turned off (both Q and -Q outputs set high) by setting the control bits RF\_ON or HV\_ON low.

#### **RS232 Interface**

The microcontroller communicates with a host computer via the RS232 interface. The RS232 interface is configured as a DCE (data communications equipment) at a fixed baud rate of 28.8k, with hardware handshaking via CTS (clear-to-send) and RTS (request-to-send), and uses a PC compatible female DB9 connector. So, the quadrupole will transmit data on pin 2, receive data on pin 3, assert CTS on pin 8, and look for RTS on pin 7. Pin 5 is the signal and chassis ground.

#### Schematic name: QMSE\_T2

#### I/V calibration

The current signal from the Faraday cup or electron multiplier is converted to a voltage by the logarithmic I/V converter (see schematic QMSE\_V1). This converter requires careful calibration and bias in order to assure accurate readings over its 8 decade range. A programmable voltage source (U209 and associated components) can provide voltages from microvolts to volts, which are applied to the I/V converter via a 1G $\Omega$  resistor to generate calibration currents from femtoamps to nanoamps. The calibration source is also used to bias the I/V converter during scans to establish the bandwidth/noise floor tradeoff.

A second I/V converter channel is biased by the calibration source to allow for temperature compensation of the detection channel. Both I/V converters are biased by equal currents from the calibration source, so that their output voltages are nearly the same when there is no ion current being detected. The outputs of each I/V converter may be read via the 16-bit A/D for calibration and offset nulling. The outputs of the I/V converters are subtracted in a differential amplifier (U211A) with a gain of 5.23 and a bandwidth of 1 kHz.

The differential amplifier is followed by a Butterworth filter (U211B) with a gain of 1.59, a -3 dB point of 300 Hz, and a roll-off of -12 dB/octave. The filter is used to reduce the noise on the signal which is above the Nyquist bandwidth for a 675 Hz sampling rate. (In low bias current cases, the bandwidth of the signal is much lower, per the diode time constant.) R233 insures that the output of the filter cannot exceed the +/-5V range of the 1:8 multiplexer at the input to the 16-bit A/D converter.

#### A/D Conversion

Several different signals and a ground reference may be multiplexed to a 16-bit A/D converter by U201, a 74HC4051. The control bits MPX\_0-2 are used to select one of the

8 inputs. The selected input is amplified by U202A by 2x to scale the input for the +/-10V range of the A/D converter, U203.

The CPU can measure the offset of the input multiplexer, op-amp, and A/D converter by selecting input X7 (the circuit ground). The measured offset is subtracted from readings taken for the other inputs.

A conversion is initiated by -CS\_ADC16 going low while R/-C is low. -CS\_ADC16 is asserted when the 675 Hz convert clock from the 74HC4020 (U108) goes low provided that CS\_VETO (MISC port bit 7) is low. The 675 Hz convert clock going low also initiates an -IRQ to the CPU. In response to the -IRQ, the CPU sets CS\_VETO high (which sets -CS\_ADC16 high) and sets R/-C high to allow the data to be read. The -BUSY output will remain low for up to 20µs during a conversion. When -BUSY goes high, the CPU returns CS\_VETO low, which again asserts the -CS\_ADC16 (this time with R/-C high), and reads the data from the ADC via the SPI. Since the ADC shifts its data on the rising edge of the data clock (i.e. data should be read on the falling edge of the data clock) the inverted SPI clock is used as the data clock to the ADC. This will also permit simultaneous transmission of data to the 18-bit RF amplitude control D/A. The CPU sets R/-C low after the data is read to prepare the A/D for the next conversion cycle.

#### **Power-up Conditioning**

The A/D reference output (+2.50VDC) is amplified, buffered, and inverted by U302A&B to provide system references of +/-5.00VDC. These references are set to zero volts when the system is reset so that other analog signals (such as the HV and filament control signals) will stay at zero until set by the microcontroller.

The triple 2:1 analog switch U301, a 74HC4053, is used to make certain that various circuits remain off when the system is reset, until the CPU can establish the system environment. Upon reset the dual flip-flop U300 is reset. The output of the second flip-flop, which controls all three channels of the analog switch, will return high with the second port strobe from the CPU to the MISC bits port. Immediately following a reset, EMIT\_CTL will be low (to multiplex EMIT\_SET to control the duty cycle of the filament heater circuit directly), the  $\pm 5.00$ REFs will be set to zero (so that all the analog control signals will be zero), and the output current from the 18-bit DAC which controls the RF amplitude will be shunted to ground.

#### Schematic name: QMSE\_T3)

#### **DC Control Voltages**

U307, a MAX528 octal 8-bit DAC, provides DC voltages to control various parameters in the system. Each output has a range from 0 to 4.98V with a step size of about 19.5 mV. The eight outputs and their functions are listed here:

0	RES_CTL	This output (-2.5V and times 1) increases the rods' DC.
1	EMIT_SET	This output (times 1 or 10mA/V) sets the emission current.
4	RPL_SET	This output (times -20) sets the repeller and filament bias.
3	FOC_SET	This output (times -30) sets the focus plate bias.
7	HV_SET	This output (times -500) sets the electron multiplier bias.
5	+OFFSET	This output (-2.5 & times .001) fixes the detector offset.

5	-OFFSET	This output (-2.5 & times .001) fixes the reference offset.
6	CAL	This output $(-2.5V)$ then times $-2$ is the calibration source.

This output (-2.5V then times -2) is the calibration source. CAL

#### **RF Amplitude Detection**

The amplitude of the RF is detected by a full-wave charge pump detector. In order to provide a symmetrical load to the generator, the amplitude on both rods is detected and summed. The charge pump works as follows: as the potential on the rod reaches a peak, the 0.5 pF capacitor (C750 on the PCB which holds the flange socket) is charged to the maximum voltage, Vp + Vdc - Vdiode with current flowing to ground via D303, a Schottky diode. During the next half cycle, C750 is charged to -Vp + VDC + Vdiodewith current flowing through D302 from the virtual ground at the inverting input of U305. The total charge transfer to the op-amp is  $C \ge dV = C \ge (Vpp-2Vdiode)$ . This charge is pumped every cycle from two charge pumps, so the current is I = 2fC(Vpp-2Vdiode). For C=0.5pF and f=2.7648 MHz, and ignoring Vdiode, the current is 2.76 $\mu$ A/Vpp. This current flows through the 698 $\Omega$  feedback resistor, R312 in parallel with R322, which is selected to calibrate the detector's sensitivity.

The output of the charge pump's I/V converter (U305) is filtered and attenuated by the 3rd order elliptic filter (C303-305 and L300) which has a characteristic impedance of  $200\Omega$  and a notch at 5.6MHz, which corresponds to the second harmonic of the detected RF signal. The potentiometer at the output of the filter (P300) is adjusted to provide a voltage of Vpp/1000. This signal is used as the feedback signal to control the RF amplitude.

The adjustment of P300, the RF amplitude detection gain potentiometer, is very important in order to maintain constant resolution and sensitivity throughout a scan. P300 should be adjusted so that He and Kr have the same resolution with RES\_CTL at a fixed nominal level of +2.50VDC. From here, empirical adjustments to RES CTL vs. amu may be made to optimize the instrument's performance, but to first order, RES\_CTL will be a constant. (Factory cal only with DS=0.)

#### **RF Amplitude Control**

The detected RF amplitude signal, VPP/1000, is compared to the RF level control (RF SET) from the 18-bit DAC U303. If the detected level is below the set level then the output of U306 slews upward, increasing the primary drive, increasing the RF level, until the RF level reaches the set level. U306 is configured as a differential amplifier with a gain of 5, which uses the ground reference from the bottom PCB, so as to reduce the effects of noise and ground offsets between the PCBs. The CPU may scan the mass filter by ramping the 18-bit DAC output between 8.56mV (for 1 amu) and 2.56V (for 300 amu).

#### Foldback Current Limiting

The RF output is approximately linear in the RF primary drive current. The scale factors for the detected RF\_SET and the measured RF primary drive current (RF\_PRI = 1.5V/A) were chosen so that RF\_SET should always be larger than RF\_PRI if the system is operating properly. In the case of a failure which causes excessive primary drive current, the output of the difference amplifier, U304B, goes positive as RF\_PRI goes above RF\_SET. The output of U304 will limit the primary drive current as the current through D304 increases. In this case, which is detected by the CPU as RF\_PRI>RF\_SET, the RF level will be less than the level set by the 18-bit DAC.

The 18-bit DAC output is also used to set the DC potentials applied to the mass filter. RF\_SET is multiplied by 4 by the differential amplifier U304A, which uses the bottom PCB for its ground reference. The output of U304A is passed to the bottom PCB via JP301 to control the DC bias sources.

### Schematic name: QMSE\_B1.

#### Mass filter RF Supply

The Toroid: The design approach was dominated by the characteristics of the RF transformer. This iron-powder toroid provides a step-up of 39:1 for the RF. The secondaries have a self-inductance of about 16  $\mu$ H so as to resonate at 2.7648 MHz with the capacitive load presented by the rods in the mass filter together with the parasitic capacitance of the secondaries.

The primary of the transformer is a single turn driven by a square wave voltage at 2.7648MHz through a series inductor (T401) of  $0.5\mu$ H. The step-up transformer is driven slightly off resonance, so that its input has a capacitive reactance, which resonates with the 0.5 $\mu$ H inductor at the drive frequency. This approach is used to reduce power losses in the drive circuit (the FETs are switched when the current is nearly zero), and to reduce the distortion in the RF output (as the drive current is very nearly sinusoidal).

Due to variations in the core material used in the step-up transformer, each unit must be tuned by adding capacitors across the primary. This is done by programming the unit to operate at 100 amu, and by finding the combination of capacitors (connected between J401 and J403) which minimize the drive voltage as measured at J400 (which is RF\_CT/5). Polypropylene capacitors with a voltage rating of 50V are used to tune the primary reactance for minimum power loss.

The 39 turn secondaries are wound in a very special fashion: the two windings are placed side-by-side so that the magnetic flux seen by the two windings is very nearly identical. Measurements on these transformers show a difference in self-inductance between the two secondaries of less than 0.1%. This is important to assure that the magnitude of the RF for the two secondaries will be very nearly equal. Silver plated, Teflon coated wire is used for the secondaries to reduce skin effect losses and to minimize interwinding capacitance, and to reduce the loss tangent in the dielectric.

Primary Drive: The square wave voltage drive is provided by a pair of IRF510 MOSFETS, Q402 and Q403. Theses FETs have delay and transition times on the order of 25 ns, and are operated as switches. The gates of the FET's are driven by complimentary square waves at 2.7648MHz with 50% duty cycles from U401, an SN75372. Each FET is on for about 180 ns.

The amplitude of the square wave drive is controlled by the DC supplied by Q401, a D44VH10 high speed npn power transistor. The op-amp U400 integrates the error signal from U306 on the top PCB to maintain the detected RF equal to the signal "RF\_SET" from the 18-bit D/A.

The primary current is passed through the FET's  $0.25\Omega$  source resistor. The voltage across this resistor is amplified by U402B and may be read by the CPU. Approximately 1.5A is required to generate 2568Vpp required at 300 amu.

At low drive levels, the charge injected via the gate-source and gate-drain capacitances of Q402/403 are a significant source of error. To compensate, the network of R413, R414 and C406 couple a signal with the correct amplitude and phase into the primary drive inductor, T401, to cancel the charge injected via the FET gates.

#### **DC Potentials**

In addition to the RF, DC potentials of about  $\pm 1/12$ th the RF peak-to-peak value is required for the two rod pairs. The op-amp U403A is the error amplifier which maintains the negative potential equal to a fraction of the set RF level. When DC\_SET goes up, the output of the op-amp goes up, increasing the current in Q406, increasing the current in Q407, bringing down the collector of Q407, which is the output of the negative potential voltage regulator.

The op-amp U403B is the error amplifier for the positive potential. The positive potential is compared to the negative potential by R431 and R432. When the negative potential increases, the non-inverting input to U403B will go down, lowering the op-amp output, increasing the current in Q405, increasing the current in Q404, thereby increasing the positive potential until it matches the magnitude of the negative potential.

#### Schematic name: QMSE\_B2.

#### Filament Heater Supply

The sensitivity of the quadrupole depends on the filament emission current, which depends on the filament temperature and bias with respect to the grid. As the grid to filament bias is fixed to optimize the electron impact ionization cross section, the filament emission current is controlled by controlling the filament temperature via the filament heater current's duty cycle.

A switching power supply, which is synchronized to a sub-multiple of the master clock at 172.8 kHz, is used to provide heater power to the filament. (Note that two clocks are required for each cycle, so the primary drive current is at 86.4 kHz.) The switcher's transformer has a 6:1 turns ratio, reducing the +24V input to a 4V output, which leaves about 3.5V after rectification in the dual Schottky diode, D501.

The primary of the transformer is operated from the +24VDC supply, and is driven differentially by a pair of IRF530 MOSFET's. An LT3525 switching controller, U501, is used to drive the FET gates. The duty cycle of the gate drive depends on the voltage at the COMP pin (pin 9). A voltage of about 0.9V or below will set the duty cycle to zero. The duty cycle increases to a maximum near 50% (for each gate) as the voltage on this input is increased to about 3.3V.

The analog switch, U500, controls the source of the signal to the COMP pin. If EMIT\_CTL is low (as it is after a reset) then the DAC signal EMIT\_SET is connected directly to the COMP pin allowing for direct control of the filament heater's duty cycle by the CPU. This mode is used when the filament is first turned "on" to verify that the pressure is low enough to allow the filament to remain "on".

When EMIT\_CTL is set high, the COMP pin is connected to the compensation capacitor C500, which will be charged and discharged by the transconductance error amplifier in U501. The error amplifier will compare the EMIT\_SET voltage (or the attenuated EMIT\_SET voltage, depending on the state of the DE\_GAS bit) to the voltage seen on

the 100 $\Omega$  emission current shunt resistor, R522. (The current sensed by this resistor is actually composed of three components: the filament emission current, the repeller voltage/100k $\Omega$ , and Vref (+5V)/30k $\Omega$ . All of these components sum. With a repeller voltage of -60V, the two non-emission sources sum to 0.60 + 0.166 = 0.766 mA. So, to set an emission current of 2 mA, EMIT\_SET, which provides 1 mA/V, is set to 2.766VDC.) The emission current may be set to values as high as 50 mA during the degas procedure.

The primary side current drive may be sensed in the  $0.5\Omega$  FET source resistor. The voltage across this resistor is filtered and amplified by 6X, and may be measured by the CPU. The duty cycle of the FET switches may also be measured by the CPU via the voltage labeled "FIL\_DUTY". A drop of about 40% in the primary side current for a particular duty cycle value indicates that one of the two parallel filaments has burnt out. A drop of 90% indicates that both filaments are missing.

If the source sense resistor voltage reaches 1V (when the primary side current reaches an instantaneous value of 2A) the LT3525 will terminate the gate drive to FET for the remainder of the cycle. This feature protects the circuit in the case of a short circuit or other severe fault.

#### **Bias Regulators**

The regulated repeller and focus plate biases are controlled by the CPU. The set voltages, RPL\_SET and FOC\_SET are multiplied by -20 and -30, respectively, by the bias regulators.

If the control signal RPL\_SET is increased, the non-inverting input (and so the output) of the op-amp U502B will go up, increasing the current in the opto-isolator's LED, increasing Q502's collector current, increasing the magnitude of the (negative) repeller bias.

If the control signal FOC\_SET is increased, current is injected via the  $61.9k\Omega$  resistors R538 and R539 directly into the emitter of Q506 and away from the emitter of Q505 which are biased at ground. These currents are mirrored by Q503/504 and Q507/508 to the output node, which has a 1M $\Omega$  resistance to ground via R531, providing an overall gain of -30.

Imbalances in currents due to focus plate currents or current mirror mismatches are made up by the op-amp U504A. If the output is smaller (less negative) than it should be, the non-inverting input to U504A will be positive, causing its output to ramp up. This increases the current into the emitter of Q506 and decreases the current into Q505. These currents are mirrored to the output, increasing the current in Q508 and decreasing the current in Q504, pulling the output more negative. The output of U504A will go up by 1V for each 1 $\mu$ A of ion current collected by the focus plate. The output may be digitized by the CPU to monitor the ion current in order to watch for excessive operating pressure.

Since the focus plate may collect ions when set to any potential below the grid potential, and since it may collect electrons when set to any potential above the repeller voltage, the focus plate regulator is designed to sink or source current.

The grid voltage is supplied by the op-amp U502A. This op-amp is operated with a gain of six to amplify the voltage selected by the analog switch U500. The op-amp is protected from possible discharges, which would connect the grid to the repeller potential, by R517 and D500.

For a high power de-gas, a DPDT relay is used to by-pass the bias regulators, connecting the grid and repeller directly to the un-regulated +250Vdc and -150Vdc supplies. During de-gas, the filament emission current is set to 20 mA, which will provide about 8W of power to heat the grid, in addition to 15W of filament heater power.

#### Schematic name: QMSE\_B3

#### **Power Supplies**

The unit is operated from +24VDC, and requires up to 2.0A. (The current draw will be the highest when the unit is scanning at the high end of the mass range, or during de-gas.) The unit is protected against power supply reversal by D610. A 5A circuit board mounted fuse limits damage in the case of a severe circuit failure. The DC input is filtered by C615, L601 and C616, which also reduce conducted emissions from the unit.

A DC-DC inverter is operated from the +24V supply to provide -140V for the repeller,  $\pm 250V$  for the mass filter bias sources, and -20V for the negative linear voltage regulators. The primaries to the inverter are driven by FET's. A switching power supply regulator, U601, provides 12V square wave drive voltages to the FET's which are sync'd to a sub-multiple of the master clock. Overcurrent protection is provided by U601 on a cycle by cycle basis. U601 does not regulate the outputs of the inverter, rather, its COMP pin is tied high to provide the maximum duty cycle.

The first of three secondaries is full wave rectified by D611-D614. This output is used only to bias the repeller, which allows the filament emission current to be monitored and controlled with high precision. The next secondary has a pair of half-wave doublers (C635, C636, and D615-D618) to provide  $\pm 250$ V. The third secondary has a full wave rectifier (D619-D622) to provide -20VDC, which is filtered by C622,C623 and L603.

All three secondaries are balanced so that no DC current is drawn in order to avoid saturation in the un-gapped transformer core of T601.

The positive power supplies (+15 for the op-amps, +12 for the RS232 and MOSFET drivers, +5\_CLEAN for the signal multiplexers and A/D converter, and +5 for the logic circuits) are derived via linear three terminal regulators (U602-605) from the +24V supply.

The negative supplies (-15 for the op-amps, -12 for the RS232, and -5 for the A/D and analog multiplexers) are provided by the linear regulators U606-608 from the -20V generated by the main inverter.

#### Schematic name: QMSE\_V1

#### Signal Conditioning

The output from the mass filter is a very small ion current which is proportional to the pressure of the selected mass. The lowest pressure which may be measured by the instrument is proportional to the lowest current which may be detected. Extreme care is required to achieve low drift and low noise current measurements at these very low

current levels. In addition, the instrument needs to measure a wide range of pressures, which requires current measurements over a wide range.

The sensitivity of the ionizer and mass filter is about  $100\mu$ A/Torr, and is nearly constant from 0 to  $5x10^{-5}$  Torr, so we expect ion currents from 0 to 5 nA. To accommodate this large dynamic range, a log I/V converter is used, whose output is amplified, filtered, and digitized by a 16-bit A/D converter. (The high resolution A/D is required to assure that the quantization noise of the A/D is smaller than the shot noise of the I/V converter's bias source, and to provide sufficient resolution.)

At the core of the detection system is the log I/V converter. A standard arrangement is used, where the current is applied to the inverting input of a very low bias current opamp (U700) and a diode is used between the output and the inverting input of the opamp. Small signal JFETs transistors (Q700/701) are used as diodes, as they have well characterized (and very low) reverse gate leakage currents.

A programmable current source is also applied to the inverting input of the op-amp. This source serves two purposes: it is used to calibrate the gain of the I/V converter (which changes with temperature), and it provides a bias current to the diodes during data collection. As a calibrator, the current source can output currents from 0 to 5 nA. As a bias source, it is programmed to provide 500 fA to 64 pA depending on the speed and sensitivity required for a scan. (High speed scans require high bias currents which reduce the time-constant though the diode, and high sensitivity scans require low bias currents to reduce the shot noise of the bias current source.)

At 500 fA bias current, the Johnson noise current of the 1G $\Omega$  current source resistor (about 4 fA/ $\sqrt{Hz}$ ) establishes the noise floor for ion current measurements. At higher bias currents, the shot noise of the bias current establishes the noise floor. For example, shot noise associated with a 500 pA bias current is about 12.7 fA/ $\sqrt{Hz}$ . In addition, the bandwidth of the detector increases with bias current, increasing the noise.

Back-to-back diodes are used to allow either ion currents (Faraday cup case) or electron currents (electron multiplier case) to be measured. During calibration procedures, the ion current is shunted to ground by a latching relay, U701. A latching relay is used so that the relay coil will not be active during either calibrations or measurements.

The input offset voltage of the I/V converter's op-amp is nulled prior to calibrating the converter's gain. This is done by strobing the MISC port bit (LINEAR) high, which activates the relay U702, configuring the op-amp to a linear amplifier with a gain of 1001 for it's own input offset voltage. The CPU then adjusts +OFFSET to null the output of the op-amp, nulling it's input offset voltage. The I/V converter is returned to the log mode by strobing the LOG bit high.

A second and identical I/V converter resides next to the I/V converter described above. The second I/V converter is operated and calibrated in the exact same manner, however, collected ion currents are not applied to its input. The primary purpose of the second channel is to correct the output of the first I/V converter against temperature effects.

#### Schematic name: QMSE\_V2

#### Electron Multiplier High Voltage Power Supply

The electron multiplier option extends the operation of the RGA to much lower pressures. By multiplying the ion current before detection in the I/V converter, the signal to noise ratio is not affected by the bias current noise of the I/V converter. Since the gain of the electron multiplier varies rapidly with applied bias, well regulated negative high voltage supply is required.

A switching power supply, synchronized to a sub-multiple of the master clock at 43.2 kHz, followed by a voltage multiplier, is used to generate a negative high voltage. The multiplier output is compared to the set level (HV\_SET) by an error amplifier (U800A) which controls the voltage on the center tap of the primary via Q800. The primary current in the HV inverter is sensed by R814, a 2.2 $\Omega$  resistor. If the primary drive exceeds 230 mA, the output of the difference amplifier U800B will go positive, reducing down the primary drive level.

The HV power supply piggy-backs off the vertical interconnect PCB in between the top and bottom PCBs. The HV power supply will only be installed in units which are ordered with the channel electron multiplier option.

# Chapter 10 Probe Assembly

This chapter contains several CIS Quadrupole Probe assembly schematics.

### In This Chapter

CIS Quadrupole Probe Assembly Schematic	.10-3
CIS Ionizer Exploded Schematic	.10-4
Feedthru Flange Connectors Schematic	.10-5

•	8	0-00500-000	WASHER, P2, MINIATURE
~	8	7-00702-721	A LONENT RD
-	8	0-00573-024	SCIEV. 0-60 X 1/8 PM SS 18-60
~	×	7-00583-720	R.F. CDAECTOR
-	2	7-00505-721	FOLK COMECT ROD
•	8	7-00567-721	LL TRA PRECISION SWET
~	21		ALUMIN SPICER
-	8		SOUT OF CONECT ROD
	9	0-00278-000	BELEVILLE SPEING WASER
•	8	0-00571-021	REPORTED SOLEY. 4-40 X 5/16 PM PAULUTS
9	17	0-00279-000	SOMED E-CLIP
-	9	1-00509-721	FOLLATE ALIGN BO
-	5		NUT HEX. 0-60
•	-	0-00577-02M	SCREW, 2-55 X 1/8 PAN SS 18-60
-	<b>•</b>	0-00574-024	SCREW. 0-60 X 3/15 PAN SS 18-60
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-	2	1-00801-721	LOG OF COMECTOR ROD
•	9	0-00570-021	PERCENTED SCIEN, 4-40 X 1/8 PHA PALLUYS
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-	-	7-00682-721	COEM MODE
-	З	7-00683-721	FARMONY CUP
-	2	7-00696-720	COBI CLAIP
-	-	7-00603-721	BEA COPPLAT FLANCE
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SRS CIS 100/200/300 EXPLODED VIEW NOTES:

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LAST MODIFIED: 7/23/97 BY JG SHEET I

CIS GUADRUPOLE PROBE ASSEMBLY THIS DRAWING DOES NOT SHOW THE IDNIZER COMPORENTS. SEE SHEET 2 FOR THE IDNIZER EXPLODED VIEW AND PARTS LIST.

17 7-00531-721 16 7-00713-720		-
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	FILAMENT_WITH_CLIPS	_
15 7-00777-720	FILAMENT_SUPPLY_MOUNT	-
4 7-00778-720	FILAMENT_RETURN_MOUNT	-
13 7-00780-720	FILAMENT_CONNECT_ROD 2	2
12 7-00779-720	FILAMENT_ALIGNMENT_ROD 3	3
1 7-00785-720	EXTRACTION_PLATE I	-
10 0-00778-000	CRESCENT_WASHER	_
9 7-00788-720	CF_TEE	_
8 0-00794-000	C-CLIP	_
7 0-00579-000	BOWED_E -CLIP 3	ñ
6 7-00782-720	ANODE	-
5 7-00787-720	ALUMINA_SEAL	-
4 7-00786-721	ALUMINA_RING 2	2
3 0-00796-000	2_ROUND_SLOTTED_188	2
2 0-00795-000	0-80_WASHER	-
0-00574-024	0-80_ROUND_SLOTTED_188 6	9
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## Appendix A Vacuum Diagnosis with SRS RGA's

## Introduction

Residual Gas Analyzer (RGA) is the term for a class of mass spectrometers. They are all quadrupole mass spectrometers and typically cover mass ranges from 1 to 100 or 200 amu (atomic mass units). The RGA's resolution is sufficient to clearly distinguish peaks that are 1 amu apart. They are designed for the analysis of the gases present in high and ultra high vacuum systems. The specifications of the RGA are a perfect match for vacuum diagnosis. Not many materials with a mass greater than 200 amu will be volatile, and so a small mass range is suitable. The high resolution of a research grade mass spectrometer is not necessary for the analysis of low molecular weight species. Overall, RGA's are affordable instruments that can be permanently attached to a vacuum system.

Experiments and processes are performed under vacuum for two main purposes: to lower the total pressure to a suitable level and to provide an extremely clean environment. An ion gauge addresses the first purpose, but only an RGA can address the second. The high sensitivity of some processes to specific impurities makes the goal of cleanness more difficult to achieve than the required total pressure. A typical method of making a system cleaner is to lower the total pressure. The assumption being that the partial pressure of the impurities is directly related to the total pressure. This assumption fails in two ways. First, it is simply not true; a total pressure measurement cannot tell the user tell the user the level of impurities. As an example, a system operating at a total pressure of  $1.1 \times 10^{-8}$  mbar might consist of  $1.0 \times 10^{-8}$  mbar of water due to outgassing and  $0.1 \times 10^{-8}$  $10^{-8}$  mbar of air due to a leak. An overnight bake out of the vacuum system reduces the total pressure to  $0.2 \times 10^{-8}$  mbar;  $0.1 \times 10^{-8}$  mbar is water and  $0.1 \times 10^{-8}$  mbar is air. Is the vacuum system cleaner than before the bakeout? If the process or experiment is sensitive to oxygen, the system is no cleaner than before the bakeout. Second, the assumption fails because total pressure measurement is not very precise; measurements more precise than 10% are difficult and expensive. If a system is operating at a standard pressure of  $1.0 \times 10^{-7}$  mbar and the next day the pressure is  $1.1 \times 10^{-7}$  mbar would there be cause for concern? The vacuum system could be filled with 10% oil vapor or the pressure gauge sensitivity could have changed 10%. The RGA is designed to address exactly these types of questions, by immediately showing the user what is in their vacuum system.

The primary application of the RGA is to analyze the composition of a vacuum system. The composition can be used to detect impurities, monitor gas fills, or analyze ongoing chemistry. Another application of the RGA is as a leak detector. It serves this use very well and in many ways is superior to a portable helium leak tester. In the remainder of

## 2 Appendix A

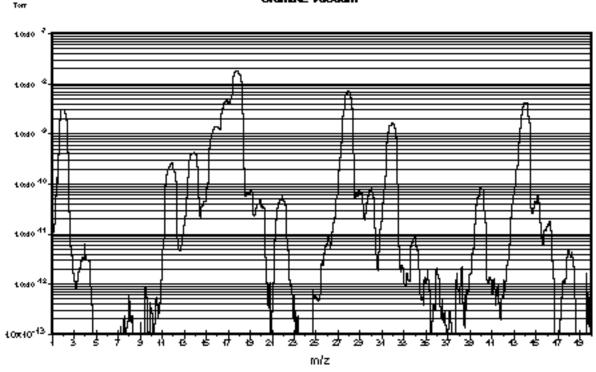
this application note we will illustrate the usefulness of an RGA. The data shown are real results from experiments designed to illustrate certain points.

## **Composition Analysis**

The SRS RGA software allows the composition of the vacuum system to be analyzed by two methods. The most common is to measure the mass spectrum of the vacuum. This provides a "fingerprint" of the residual gases in the vacuum system. A second method is to track specific species or peaks of the mass spectrum. The first method, analog scan mode, is most useful when the user does not know what is present in the chamber. Once the identities of the species have been determined, individual peaks can be tracked using either pressure vs. time, table or annunciator mode.

#### **The Mass Spectrum**

The fundamental operation of the RGA is as a mass spectrometer. Figure 1 shows a graph of partial pressure versus mass, which was measured with an RGA with an electron multiplier detector.



#### Ultimate Vacuum

Figure 1: Partial Pressure vs. Mass

The scan was taken of a vacuum system near its ultimate vacuum. The pressure axis is plotted on a logarithmic scale so that a large range can be seen. The log scale makes the peaks appear wider than when plotted on a linear scale. This scan from 1 to 75 amu shows some gases commonly present in vacuum chambers. There are many peaks, but they are caused mainly by 7 species: Hydrogen is at 2 and helium at 4. Water gives primary peaks at 16,17, and 18 due to the species  $O^+$ ,  $HO^+$ , and  $H_2O^+$ . The smaller peaks at 19 and 20 are due to <sup>18</sup>O which is naturally present at 0.2%. Nitrogen is at 28 and also causes the peaks at 14 by atomic  $N^+$  and the doubly ionized  $N_2^+$ . Molecular oxygen shows a peak at 32 and an isotope peak at 34. Argon shows a peak at 40.

Carbon dioxide shows a peak at 44 and a peaks for  $CO_2^{++}$  and  $C^+$  at 22 and 12. The other peaks are caused by fragments of these species and contaminants.

The presence of air components in the spectra might lead us to believe that the system is leaking, but this is untrue. The hybrid turbomolecular pump has simply reached its compression limit. The foreline of the pump was operating at a total pressure of 0.5 Torr; thereby the compression ratio is in the  $10^8$  range (as the pump specifications indicate). Nitrogen, oxygen and argon are all present in the same ratios as standard atmosphere. The presence of helium is interesting, because it is present in the atmosphere at about 7 ppm. Its peak might be expected 6 decades smaller than the nitrogen peak. The low compression ratio of the turbo pump for helium ( $10^5$ ) explains why the peak is only three decades smaller.

The ability to detect these common species and many others is the essence of the RGA. The fragmentation of molecules in the ionizer of the RGA gives each molecule a distinct fingerprint. The fragmentation patterns for many molecules is available from the library in the SRS RGA program. Keeping a historical record of the typical spectrum of a vacuum system allows the appearance of peaks to be instantly detected. For instance, the peak at 48 in Figure 1 is SO from SO<sub>2</sub> (a matching peak at 64, not shown on the figure confirmed this). On occasion this peak is seen in our chambers. If SO<sub>2</sub> appeared in your vacuum system would you want to know? In addition to these simple gases, we are interested in molecules with higher weights. The next two examples show how oil and solvents can be detected.

#### **Oil Contamination**

Figure 2 contains a mass spectrum of a common contaminant of vacuum systems, oil. The top pane is the measured spectrum and the bottom pane is the library data.

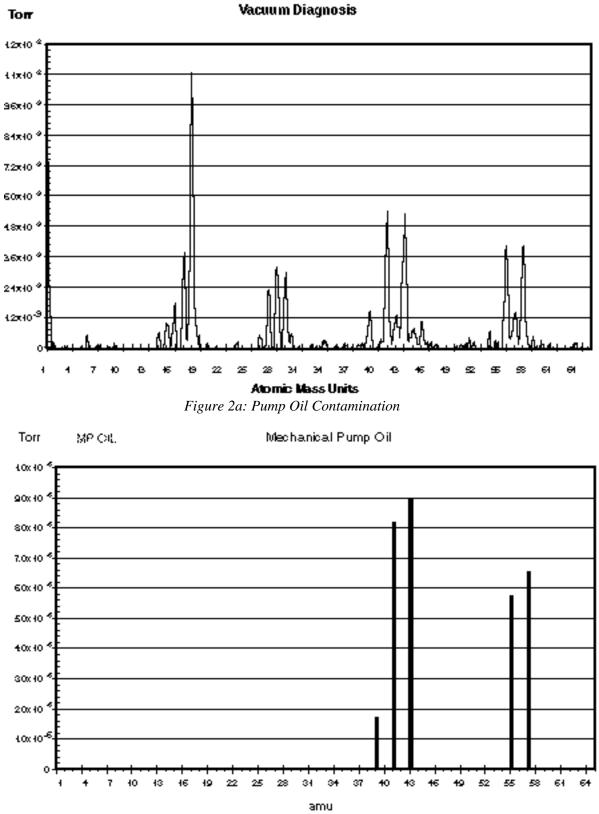
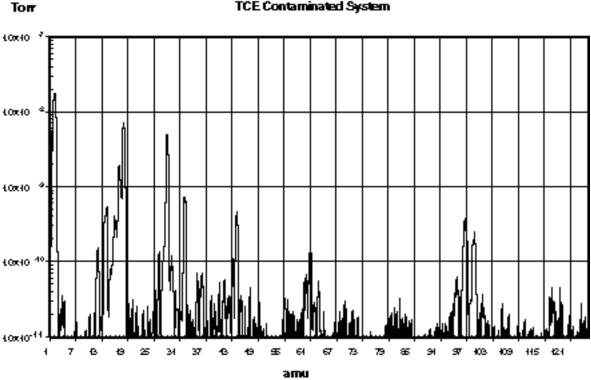


Figure 2b: Library Pump Oil Data

The presence of mechanical pump oil is immediately obvious. The peaks at masses 39, 41, 43, 55, and 57 are caused by mechanical pump oil backstreaming into the vacuum chamber during a load lock sequence. The total pressure in the chamber was dominated by water and was less than  $2 \times 10^{-8}$  Torr. In this case, the total pressure might satisfy operating conditions but the spectra reveals that the system is heavily contaminated with oil. This could have been caused by improper valve sequencing or a saturated oil trap. Without an RGA, only operating procedures for valves and a maintenance schedule for traps can ensure that the cleanliness of a vacuum system is maintained. With the RGA, the cleanliness can be guaranteed before a process or experiment begins.

#### **Solvent Contamination**

Oil contamination is common in vacuum systems. Cleaning parts with solvents is a common approach to removing this contamination. We have observed that organic solvents such as acetone and 1,1,1-trichloroethane (TCE) are more tenacious contaminants than the oil they are designed to remove. Figure 3 shows the spectrum of a vacuum system contaminated with TCE as evidenced by the major peaks at 97 and 99, and the minor peaks at 61, 63, 117 and 119. The paired peaks are caused by the natural isotopic occurrence of <sup>35</sup>Cl and <sup>37</sup>Cl (75% and 25%). This spectrum was measured one week after the initial contact with the solvent. The TCE permeated into the o-rings in the system during a cleaning step. The TCE continued to outgas from the o-rings for two weeks and showed no signs of stopping. At that time they were removed and baked in an oven, which eventually removed the TCE.



#### TCE Contaminated System

Figure 3: TCE Contaminated System

Data like this is invaluable to the development of cleaning procedures. While the TCE successfully removed the oil, the vacuum chamber was left more contaminated than it

would have been without the cleaning. The mass spectrum provides a more accurate evaluation of cleaning procedures than pump down time and base pressure. Just because a system pumps down quickly does not guarantee that undesirable contaminants have been eliminated.

The large dynamic range of the RGA also allows evaluations to be made more quickly. After a vacuum system has been brought up to atmospheric pressure, it will require an extended period to pump back down to its ultimate vacuum. If the vacuum system does not quickly reach its ultimate vacuum, the next step is commonly a high temperature bakeout. If the vacuum system has still not reached its ultimate vacuum, problems are suspected and typically a leak tester would be attached to the system. With the RGA, the user does not have to wait several hours for the water to pump down or perform a bakeout before determining if a system is contaminated. The RGA can make measurements as soon as the total pressure reaches  $10^{-5}$  mbar. The large dynamic range will allow impurities to be seen at  $10^{-10}$  mbar or leak testing to be performed in the presence of a this high total pressure. Thereby, the RGA allows a great reduction in turn-around time after a vacuum system has been brought up to atmosphere.

## **Single Mass Measurement**

For vacuum systems that only need to be clean, the mass spectrum is the most useful measurement. During experiments and processes the partial pressure of certain species is of more interest. The RGA software provides three modes that are used to measure selected peaks. The selection of which mass is associated with which species is usually straightforward, *i.e.* the mass of the molecule is chosen. When two species have overlapping peak patterns, the user chooses the strongest peak that does not interfere. For example  $N_2$  and CO both have a mass of 28. In a system with a large CO interference,  $N_2$  could be measured at mass 14. Because the peak at 14 is smaller than the major peak, a scaling factor is required. The set of peaks of interest are entered into the RGA software by the user. During measurements, the RGA measures only each peak. Because the whole spectrum is not measured, data is acquired much faster. The two examples that follow show an interesting pressure vs. time experiment and a method of increasing the dynamic range of measurements.

### Pressure vs. time

Figure 4 shows the use of the P vs. t Mode to monitor an air lock sequence. The process is to open an air lock, place the sample in it, and move the sample into the main vacuum chamber. The air lock is pumped from atmospheric to rough vacuum using the same mechanical pump as the main vacuum chamber. This requires isolating the foreline of the turbo pump during the time the sample is in the air lock. To keep oxygen out of the main vacuum chamber, the air lock is flushed with dry nitrogen then pumped to rough vacuum. The flush is repeated two additional times before the sample is finally transferred. If this procedure is successful, the main vacuum chamber should not be disturbed.

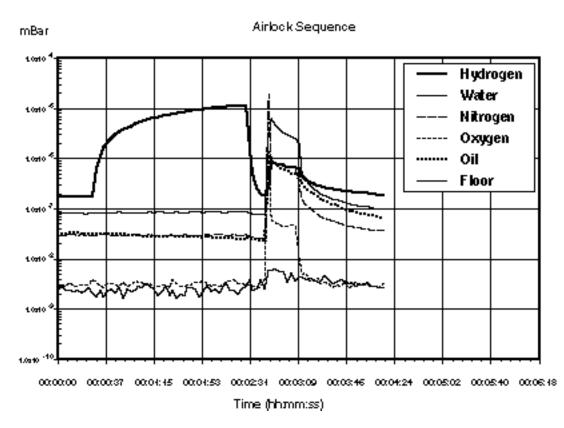


Figure 4: Airlock Sequence

To make these measurements, the electron multiplier detector has been used with a gain of 100, which allows all six channels to be recorded every three seconds. The standard Faraday cup detector is able to detect these partial pressures, but not at this rate. The "floor" channel is set to mass 21. There is rarely anything present at this mass, which allows it to be used as an indicator of the minimum detectable partial pressure.

The sequence starts with the main vacuum chamber at its base pressure of  $2 \times 10^8$  mbar. The chamber is pumped by a turbomolecular pump that is backed by a rotary vane pump. A small load lock is attached to the chamber, which can be rough pumped by the same mechanical pump and purged with nitrogen.

At 0:30, the isolation valve between the turbo pump exit and mechanical pump is shut so that the mechanical pump can be used to rough pump the load lock. During this time, the load lock is repeatedly filled with nitrogen and pumped down. Of interest in the data is the rise in the hydrogen partial pressure during this step of the sequence. The partial pressure of hydrogen increases by a factor of 100 while the partial pressure of the other gasses barely increase. This difference is caused by the low compression ratio that turbo pumps have for light gasses. The heavier gasses are being compressed into the dead volume between the turbo pump exit and isolation valve. But, the turbo pump has insufficient compression ratio to store hydrogen in this manner, causing the partial pressure of hydrogen to rise.

At 2:30, the load lock has been roughed and the turbo pump foreline isolation valve is opened. The pressures of  $H_2$  immediately drops back to the base pressure values.

At 2:40, the load lock is opened to the main chamber causing a jump in pressure. The rise in oxygen and oil pressure indicates that the procedure is operating poorly. Even though the load lock was purged three times with 99.999% nitrogen, oxygen was still introduced into the chamber. This was either caused by a small air leak into the load lock, or permeation of oxygen out of the elastomer seals on the load lock. The rise in oil partial pressure indicates that the trap on the mechanical pump is exhausted and has allowed oil to backstream into the load lock.

At 3:09, the valve between the load lock and main chamber is closed, and the pressures begins to return towards their base values. Oxygen is pumped out of the chamber. Hydrogen, water, and nitrogen recover their original values, but slowly. The oil is alarming because it persists at a higher concentration. If this sequence occurred several more times, the oil would continue to increase. The RGA allows it to be detected before reaching undesirable levels.

### **Table Mode**

The noise floor of the Faraday cup detector is about  $10^{-10}$  mbar. Since the maximum operating pressure is  $10^{-4}$  mbar, the dynamic range of the RGA is 6 decades or 1 ppm. The noise floor of the channel electron multiplier (CEM) is lower, but its maximum operating pressure also decreases with the noise floor. The two pressure limits change such that the dynamic range of the CEM is still 6 decades. By switching between the two detectors, measurements covering more than 6 decades can be made. The table mode of the SRS RGA software allows such a measurement to be made. The CEM status can be set independently for each mass being monitored. Figure 5 shows a configuration where the prevalent gases are detected with the FC and the low pressure gases are detected with the CEM. A comparison of the value for nitrogen and "floor" show that the apparent dynamic range is 8 decades or 10 ppb. The program will automatically sort the channels so that all the measurements requiring the CEM are made as a group, which minimizes the switching on and off of the detector. Without the ability to choose between FC and CEM detector for each channel, the CEM would have to be used for all channels so that the low pressure gases could be detected. Operation like this would cause the CEM detector to saturate at the high pressure peaks. Saturation of the detector makes the value useless, and also increases the physical wear on the CEM.

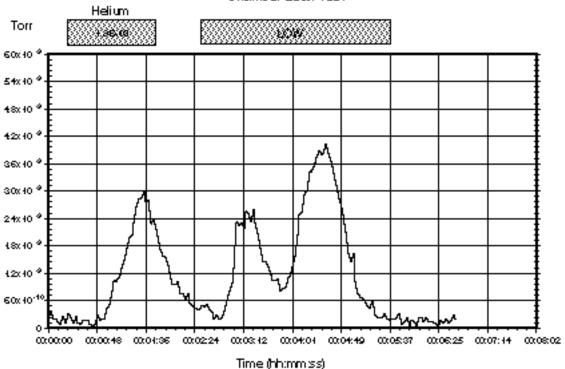
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3	Nitogen	28		(#C91	1	1.00	OFF
4	Orgen	32		NE21982AL	3	1.00	OH
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Figure 5: SRS RGA Table Display

### Leak Testing

In addition to the diagnosis of vacuum systems, the RGA is invaluable as an intrinsic leak detector. It is always available and does not require perturbing the system. The user does not have to roll up a large leak detector and attach it to the vacuum system. The system does not have to be brought up to atmospheric pressure. The RGA can operate in leak detection mode using any gas, so it does not require helium. For moderate leaks, argon or tetrafluoroethane (a typical gas in cans of "dust off") can be used. Helium is necessary for only the smallest leaks. Having a built-in leak detector makes working with vacuum systems much easier and faster, and the SRS RGA is far less expensive than a standard helium leak detector.

The process of leak detection with an RGA is the same as with a traditional helium leak detector. Place the software in leak detection mode, indicate the mass of the test gas and watch the partial pressure as various joints in the vacuum system are sprayed with the test gas. When the leak is sprayed with the test gas, the partial pressure will rise. The response is immediate if the leak is in a direct path from the outside to the inside of the system. Figure 6 contains the result of a leak test with helium on a vacuum chamber. The tester moves the helium probe towards and then past the leak, causing the first peak. Once the location of the leak is bounded, the tester tries to precisely locate the leak. For most situations leak testing is straightforward and no different than traditional methods. In the following sections, we discuss a few situations where traditional methods fail and how the RGA provides a better method.



Ghamber Leak Test

Figure 6: Helium Leak Test Data

The partial pressure of the test gas is directly related to the leak rate into the chamber. Assuming that the vacuum pump is not operating near its compression limit, the throughput of the test gas is equal to the product of the partial pressure and the effective speed of the pump at the RGA ionizer (Q = S P). In Figure 5, the partial pressure was measured in Torr. The effective speed of the turbo pump for helium was approximately 50 liter s<sup>-1</sup>. The largest peak in this data at  $4 \times 10^{-9}$  Torr represents a leak of  $1.5 \times 10^{-7}$  scc sec<sup>-1</sup>. (0.76 scc sec<sup>-1</sup> = 1 Torr liter s<sup>-1</sup>). From this figure we can estimate a minimum detectable leak of  $1 \times 10^{-8}$  scc sec<sup>-1</sup>, which is measured with a FC detector. To measure smaller leaks, a CEM detector can be used, or the turbo pump can be throttled to decrease the pumping speed.

#### **Supply Gas Valve Seats**

Leaks across valves that supply gasses to a vacuum system cannot be detected with conventional helium leak testers (unless the valve supplies helium). To test a suspect valve would require removing it and attaching it to the leak tester. Because the RGA can monitor any gas, this is unnecessary; the valves on gas supply lines can be tested *in situ*. The procedure is simple: monitor the composition of the vacuum system with a high pressure and then a low pressure behind the valve seat in question. If the partial pressure of the gas in question changes, the valve seat is leaking.

#### **Supply Gas Manifolds**

Leak testing supply gas lines can be a very trying experience. The difficulty is mostly because compression type fittings do not have a leak test port. Another difficulty is that supply manifolds commonly have a large number of connections in close proximity. Because the leak in a compression fitting is inside the fitting, transporting the test gas to

the leak requires a large flowrate and waiting for an extended time for the gas to diffuse into the fitting. Because of the flowrate and time, it is possible that the test gas can travel to adjacent tube fittings and cause a misleading indication of a leak. Often times "fugitive" leaks appear and disappear at a specific fitting. What is happening is that the test gas is inadvertently flowing to another fitting, which has a real leak. Whether this inadvertent gas flow occurs depends on exactly how the test gas is applied to the fitting with the "fugitive" leak. Small air currents caused by equipment fans or ventilation systems can move the test gas in unpredictable ways. Confining the test gas to the fitting under question can help, but the RGA provides a easier solution: use a gas other than helium. Helium will spread in air quickly and diffuse into many fittings. A heavy gas like argon or tetraflouroethane is far easier to confine to a specific fitting. Once the moderate leaks have been located and eliminated, a follow up with helium to check for tiny leaks is warranted.

#### **Bellow Valves**

Bellow valves can be difficult to leak test due to the large volume of gas contained between the bellow and the valve body. To perform a quick leak test it is required to change the composition of the gas in this trapped volume quickly. Unfortunately, for some valves, this volume of gas is not highly accessible. This greatly reduces the response time of a leak test. For a leak causing a base pressure of  $10^{-7}$  mbar in a vacuum system with a 70 l s<sup>-1</sup> pump, the volumetric flowrate of gas entering from the atmospheric side of the leak is 7 nanoliters per second. For a bellow with a trapped volume of 1 ml trapped, the response time constant would be over 40 hours. This emphasizes the importance of leak test ports on vacuum hardware.

In such situation it is common to place a bag over the body of the valve and fill the bag with helium. The RGA allows us to consider using gases other than helium. The permeability of helium through elastomeric seals can give a false leak reading. And as in manifolds, unless the helium can be strictly confined to the valve body, it may spread to adjacent connections. Given the amount of work and lost time required to remove and repair large valves, false leak readings are expensive. A second test with another gas, such as argon, can confirm that a suspect valve is leaking before starting out on the repair.

## Conclusion

An RGA is a real eye opener for users of vacuum systems. With an RGA the process of working with vacuum systems is elevated from an empirical trial and error approach to a systematic approach. The status of the vacuum system can be constantly assessed. When an experiment or process is having problems, the possibility of contamination or leaks in the vacuum system can be immediately ascertained. The RGA provides not only troubleshooting but also historical data. Slow trends in a vacuum system can be noticed and acted on before they become serious problems. Hard to correlate cause-effect relations are much easier to establish with the wealth of statistical data that can be obtained with a daily spectrum analysis. Good procedure and strict adherence to them allows a user to infer the integrity of a vacuum system. An RGA allows users to prove the integrity of a vacuum system with hard data.

## **Further Reading**

Dawson, P.H., *Quadrupole Mass Spectrometry and its Applications*, American Institute of Physics, 1995.

Drinkwine, M.J., and Lichtman, D., *Partial Pressure Analyzers and Analysis*, American Vacuum Society, New York.

Wilson, N.G., Beavis, L.C., *Handbook of Vacuum Leak Detection*, American Vacuum Society, New York, 1979.

## Appendix B Using SRS RGA's to Sample High Pressure Gasses

## Introduction

The types of analysis performed by an RGA are useful in many applications other than vacuum systems. But, the RGA is intrinsically a vacuum instrument that operates best under  $10^{-5}$  mbar. The instruments response becomes non-linear above  $10^{-5}$  mbar. To sample gases at higher pressures, a pressure reduction system is needed. These systems are basically a restriction and a vacuum pump package. Common restrictions are pinholes and capillaries, which can provide pressure reductions of more than 6 decades of pressure. The vacuum pump package consists of a turbomolecular pump and a backing pump. In addition to achieving the desired pressure reduction, the design of a system should provide for a fast response and high signal to background ratio.

At pressures common to vacuum processes, a simple aperture based pressure reduction system is suitable. At atmospheric and higher pressures, a two stage reduction based on a capillary and aperture is used. These two systems will be used to illustrate the design of pressure reduction systems for RGA's.

## Vacuum Process Sampling (10 to 10-5 mbar)

Figure 1 shows a schematic of a basic pressure reduction system. The system has two paths to the RGA: a high conductance path and an aperture path. The high conductance path (through Valve Hi-C) is provided so that the RGA can monitor the ultimate vacuum of systems before a process begins. The Hi-C path is also used when leak testing the vacuum system with the RGA Software's leak test mode. The aperture path provides the pressure reduction for when the vacuum process is operating at pressures up to 10 mbar.

The design of the aperture path is straightforward. First, the RGA operating pressure is chosen, for example  $10^{-6}$  mbar. The pressure, P, and the pumping speed, S, determine the throughput of the aperture, Q, by the equation Q = P S. To keep the system cost low, a small turbo pump is chosen. For a 70 liter s<sup>-1</sup> pump, the required throughput is  $7 \times 10^{-5}$  mbar liter s<sup>-1</sup>. The throughput of the aperture is related to its conductivity, C, and the pressure drop,  $\Delta P$ , by the equation Q = C  $\Delta P$ . The process pressure determines the pressure drop thereby determining the required conductivity. Formulas for conductivity of various geometries, *e.g.* capillaries, and pinholes, are available in many references (some are listed at the end of this note).

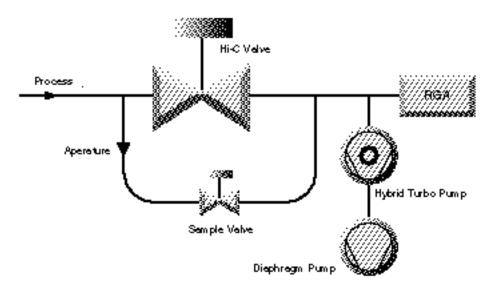
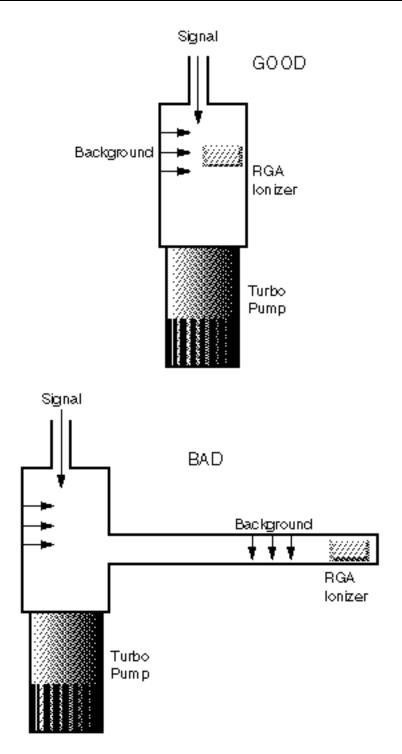


Figure 1: Schematic of a mid-vacuum pressure reduction system

Apertures can be readily designed for process pressures in the range from  $10^{-3}$  mbar to 10 mbar. If the process always operates within a small range, the aperture can be optimized to deliver gas to the RGA at about  $10^{-6}$  to  $10^{-5}$  mbar. By operating the RGA at its optimum pressure the data acquisition time is kept to a minimum and the full dynamic range in partial pressure is available. For many applications, the process is operated at one pressure and the aperture can be optimized. If the process pressure varies over a range of 2 decades or more, the aperture size must be compromised to tolerate the pressure range. For example, consider a process pressure that varies from  $10^{-1}$  to 10 mbar. The aperture would be designed to drop the pressure from 10 mbar to  $10^{-5}$  mbar. When the process pressure was at  $10^{-1}$  mbar, the pressure at the RGA would be  $10^{-7}$  mbar. The minimum detectable partial pressure (MDPP) of the RGA does not depend on the operating pressure; for a Faraday cup detector it is about  $10^{-10}$  mbar. Therefore the dynamic range of the measurement varies from 5 decades at high process pressure to only 3 decades at the low pressures. For applications where the full dynamic range is not needed, operating the RGA at low pressure may be acceptable. If the full dynamic range is required over a variety of process pressures, a variable reduction is required. Suitable variable leak valves are available, but are significantly more expensive than a fixed aperture. Another method of increasing the dynamic range and data acquisition rate is to use an RGA with an electron multiplier. The electron multiplier provides gains up to  $10^6$  and lowers the MDPP to under  $10^{-13}$  mbar. This lower MDPP allows the RGA to provide large dynamic range even at low operating pressures.

A high operating pressure (or throughput of the aperture) at the RGA also improves the signal to background ratio. In this context, signal is the gas that is drawn through the aperture and background is outgassing from the system plus backstreaming through the turbo pump. The ultimate vacuum of many turbo pump packages is about  $10^{-9}$  mbar.

The outgassing background will be mostly hydrogen, water, and nitrogen. The backstreaming background will be air. If measurements are being made near these background peaks, the operating pressure should be kept as high as possible. The background can be minimized by designing the tubing such that the effective pumping speed at the RGA ionizer is as high as possible. Figure 2 shows two layouts that both have the same "signal" level. The layout with the RGA at the end of a small tube has a small effective pumping speed and will show a larger background level.





The system shown in Figure 1 can be assembled as a simple package. Using a small (70 liter  $s^{-1}$  or less) hybrid turbo pump and a diaphragm backing pump will eliminate any concern of oil. The use of this pump pair also eliminates foreline traps and isolation valves. The operation of the system should be simple: open the Hi-C valve at low pressures, or open the sample valve at high pressures.

## High Pressure Sampling (>100mbar)

At high pressure the aperture assembly is insufficient to reduce the pressure, while maintaining response time. Consider an aperture that reduces the pressure from 10 mbar to  $10^{-6}$  mbar when used with a 70 liter s<sup>-1</sup> turbo pump. The volumetric flowrate on the high pressure side of the aperture would be 7 microliter s<sup>-1</sup>. Any dead volume on the high pressure side of the aperture (Figure 3) would cause a large response time constant (t<sub>c</sub> = volume / flowrate). If the aperture had a small dead volume of 1/2 inch of 0.250 OD tube (0.028 wall), the time constant would be 35 seconds. This is not an acceptable response time.

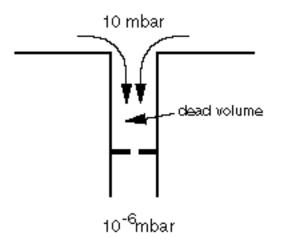


Figure 3: Small Dead Volume Slows Process Response time.

To achieve a fast response time, a capillary inlet is used with bypass pumping as shown in Figure 4. The system reduces the pressure in two stages. Most of the sampled gas is drawn through the capillary and directly to the diaphragm pump, *i.e.* bypasses the RGA. The pressure at the exit of the capillary is about 1 mbar. A small amount of the sampled gas is diverted to the RGA through an aperture. This configuration improves the response time by two methods. First, the pressure on the high side of the aperture is held to about 1 mbar. But even this pressure would give a time constant of 3.5 seconds in the 1/2 inch dead volume example mentioned above. The second method to decrease the time constant is to ensure that any dead volume is well mixed. After the capillary, the gas is traveling at significant velocity (several meters per second). Proper layout of the inlet tubing will use the kinetic energy of the sampled gas to mix the dead volume (in a sense keeping the volume alive). Figure 5 shows the response to bursts of gas at the inlet of an atmospheric sampler designed with the above considerations. The sub-second response and cleanup are almost as fast as the RGA can acquire data.

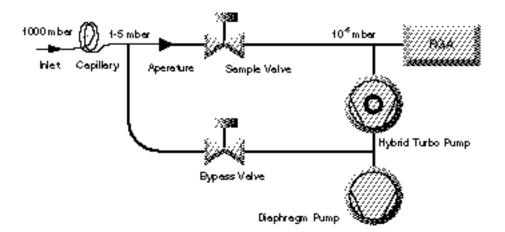


Figure 4: High Pressure Sampling Using Bypass Pumping

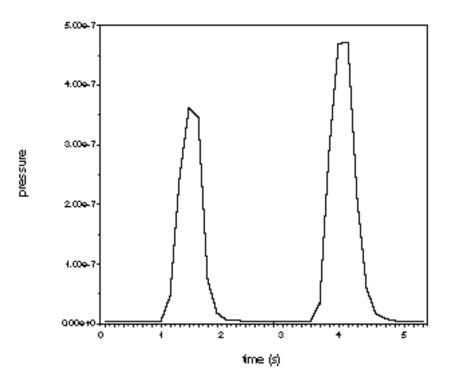


Figure 5: Response of Bypass Pumped System to Gas Bursts.

Glass capillaries are available with small enough bores to reduce pressure from  $1000 \text{ mbar to } 10^{-6} \text{ mbar without bypass pumping}$ . While it is possible to build a atmospheric sampling system based on a 1/4 meter 50  $\mu$ m glass capillary, there are considerable reasons to use a bypass pump configuration. Bypass pumping improves the operation of a system by increasing the flowrate of gas through the capillary about 3-4 orders of magnitude. The higher flowrates and smaller pressure drop allow a wider selection of capillaries to be practical. Stainless steel and PEEK capillaries are more affordable and flexible than glass capillaries. A large flowrate means that the volumetric

flowrate at the inlet of the capillary is more reasonable. For a system with 70 liter s<sup>-1</sup> pumping speed, operating at  $10^{-6}$  mbar, the volumetric flowrate at the inlet would be 70 nanoliter s<sup>-1</sup>. Any dead volume at the inlet of the capillary would result in an unreasonable response time. With such small flowrates, inlet devices such as filters, valves, or connecting hardware cannot be used. Overall, the bypass pumped capillary system is more flexible and only requires a minor addition of hardware (one valve and some tube).

The configuration seen in Figure 4 is made possible by the recent advances in hybrid turbomolecular/drag pumps and diaphragm pumps. Traditional designs would have relied on a **two** rotary vane pumps and standard turbomolecular pump. The high compression ratios of the hybrid turbo pumps allow the two streams (bypass and sample) to be combined. The low ultimate vacuum of contemporary diaphragm pumps makes them suitable as a foreline pump. The combination of these modern technologies means that an atmospheric sampling system can be constructed into a very small packages (less than 8 inch high in a 19 inch rack mount chassis), which is portable and easy to operate.

## Conclusion

Although the RGA is intrinsically a vacuum instrument, inlet systems are easily designed that allow it to sample gasses at any pressure. A more descriptive name for such systems would be "online quadrupole mass spectrometer". Mass spectrometry is a well proven analytical technique, but traditionally used an expensive large machine. Reduction in cost of quadrupoles and vacuum pumps, along with the development of easy to use software interfaces makes process analysis with mass spectrometry an attractive technique.

## **Further Reading**

Lewin, O., *An Elementary Introduction to Vacuum Technique*, American Vacuum Society, New York, 1987.

Moore, J.H., Davis, C.C., and Coplan, M.A., *Building Scientific Apparatus*, 2nd ed., Addison-Wesley, New York, 1989.

# Appendix C SRS RGA Output Control Options O100TR and O100TS

## WARNINGS

- Please note that the TTL output signals are always active high and may not be configured otherwise. This means that any time an output channel's alarm is triggered, its corresponding TTL signal is set to a high TTL level. The untriggered state is a Low TTL level.
- All output signals should be considered INVALID unless the "Outputs Valid" signal is set high (when the RGA is scanning and acquired data has been validated).
- When the I/O board is reset all the output signals are reset to Low TTL levels. This happens anytime the computer is turned on or the I/O board is initialized.

## Introduction

With the Output Control option the RGA Windows program controls TTL output signals that may be used in a variety of process control applications. The 24 TTL signals (2 reserved for future use) are located on a PC expansion bus that is installed in the same computer as the RGA program. The user can access the TTL signals using a 37-pin cable and a universal screw terminal board. An electromechanical relay board (compatible with the TTL output connector and cable) may be attached instead of the screw terminal to drive solenoids and other system components.

## **Signal Description**

## **Table Channel Outputs (11 output channels)**

10 TTL signals are mapped to the 10 channels associated with the Table, P vs. T, and Annunciator modes. The user assigns a trigger and hysteresis level for each channel independently. When enabled, the channel is monitored after each scan and when the pressure goes above (or below) the trigger value + hysteresis %, the corresponding TTL bit is set high. The user may temporarily override any channel by setting its output Low or High. The Total Pressure value is the eleventh channel and is monitored in the same fashion as the channels above.

#### Valid Outputs (1 output channel)

1 TTL signal is used to indicate that the table channel outputs are valid. This signal is set High while the RGA is scanning (after completion of the first scan) and set low as soon as the scan is stopped (the output card must be enabled).

### Manual Outputs (10 output channels)

10 TTL signals are mapped so that the user can directly set them Low or High manually from a dialog box in the RGA program. The use of these outputs is left to the discretion of the user.

## Hardware requirements

## TTL I/O card (Required)

The TTL I/O card is a 24-bit Input /Output card that is mapped directly into the PC I/O space (no IRQ is required). An installation diskette is shipped with each card to help the user install and test the card. The installation diskette is essential since it installs files that the RGA program requires. The I/O card is installed in the same PC where the RGA program is running so an empty 8-bit or 16-bit expansion slot is required in that computer. A 37 pin D-Connector cable is shipped with the card to allow access to the signals. All signals are active high (initially low).

#### **Electromechanical Relay board (optional)**

This 24 relay board is available as an option and is 100% compatible with the I/O board mentioned above. The cable shipped with the I/O board connects directly to the relay board. The relay board is shipped with a power cable that connects the any internal PC power plug. Each relay has a Connect, Normally Open, and Normally Closed screw terminal connection. The board has 8 mounting holes and comes with standoffs for testing on a lab bench. See attached data sheets for more details.

#### I/O Screw Terminal Board, 12-22 AWG terminals (optional)

This is a simple 4"x 4" screw terminal board that can connect directly to the TTL I/O card cable to give easy access to the TTL signals.

## **Installation Procedure**

Every I/O board is shipped with an installation diskette from the manufacturer (Computer Boards, INC.). Please read all the documentation and manuals that are shipped with the I/O board before installing it. You must use the I/O board installation diskette (Instacal program) before attempting to use the RGA program with the board.

#### **Installation Instructions**

- 1. Make sure the RGA program and RGA Head are operating properly before installing the I/O board.
- 2. Configure the I/O board hardware switches following the manufacturer's directions (see the Computer Boards, INC. manual for your board).

- 3. Install the board in the computer following the manufacturer's directions (see the Computer Boards, INC. manual for your board).
- 4. Turn on the computer and run the "Install.exe" program on the diskette that shipped with the board. Keep in mind the following points: a) Select **No** when asked if you will be using a PCM series card, b) Select **No** when asked if you want to install Universal Library or LabView software, c)Select **YES** when asked if you want the AUTOEXEC.BAT file modified.
- 5. Reboot the computer
- 6. Run the "Instacal.exe" program from the I/O board directory that you specified in the "Install.exe" program above.
- 7. Configure the "Instacal" program for your board name and settings (see the Computer Boards, INC. manual for your board). Make sure you run the internal test routines to verify that the board is functioning correctly.
- 8. Start the RGA program and select the "Setup Output Card" command from the "Output Control" menu.
- 9. Press the "Scan for I/O Boards" button. If you get a warning message select Yes to proceed with the scan. At this time the board name and number should appear in the dialog box. If the board is not found by RGA, run the Instacal program and test the board to make sure it is operating correctly.

## **RGA Software Operation**

Please refer to the On-Line Help documentation of the RGA program for a full description of the software.

## Appendix D PPM-Level Gas Analysis: The Closed Ion Source (CIS) Advantage

## Introduction

Quadrupole Mass Spectrometers are used in a large variety of analytical applications, from basic research to on-line process monitoring and control. Ease of use, a compact design, excellent dynamic range and very stable operation are some of the main features that make the instruments so popular.

The most common use of quadrupole mass spectrometers in vacuum applications is as residual gas analyzers (RGA). Most commercially available RGA's are very small and can be attached to virtually any vacuum chamber. The prototypical RGA has an **open ion source (OIS)** and the entire analyzer operates at the pressure of the vacuum system. The maximum operating pressure is  $10^{-4}$  Torr.

More recently, quadrupole mass spectrometers have been increasingly used as process monitors. By far, their most common application is for the detection of low-level contaminants in process gases. Modern day contamination requirements often fall in the PPM and sub-PPM levels, and will continue to decrease as processes become more sophisticated and the components being manufactured require additional refinement.

Although traditional quadrupole mass spectrometers designed for residual gas analysis have adequate dynamic range to detect PPM-level contaminants in principle, interferences from process gases, and background interferences from the sensor itself can make the detection of PPM levels of certain contaminants difficult in practice. The partial pressure detection limit of a mass spectrometer depends both upon the sensitivity of the sensor and upon the background signals that are present in its mass spectra. In applications requiring the measurement of pressures between  $10^{-4}$  and  $10^{-2}$  Torr, the problem of background contributions to the spectra can be significantly reduced by replacing the RGA with a **closed ion source (CIS)** sampling system.

The following sections of this document describe the performance specifications of open and closed ion source quadrupole mass spectrometers. The main objective of this information is to introduce the basic concepts required to choose the best gas analyzer for any practical application, and to discuss some of the basic operating principles that must be kept in mind to assure the optimum performance of the instrument selected.

## Residual Gas Analyzers (P<10<sup>-4</sup> Torr)

The prototypical RGA has an open ion source (OIS) and is attached directly to the vacuum chamber so that the entire analyzer is at the same pressure as the rest of the vacuum system. The maximum operating pressure is  $10^{-4}$  Torr.

In **high vacuum applications** such as research chambers, surface science setups, accelerators, aerospace chambers, scanning microscopes, outgassing chambers, etc., RGA's are effectively used to monitor the quality of the vacuum and can easily detect even the most minute impurities in the low pressure gas environment. Trace impurities can be measured down to  $10^{-14}$  Torr levels, and sub-PPM detectability is possible in the absence of background interferences. During system troubleshooting, RGA's are also used as sensitive, in-situ, helium leak detectors.

In the **semiconductor industry**, RGA's are best used in evaporators, sputterers, etchers or any other high vacuum systems that are routinely pumped down to better than  $10^{-6}$ Torr. The main use is to check the integrity of the vacuum system and the quality of the vacuum before the wafers are committed to the process. Air leaks, virtual leaks and many other contaminants at very low levels can ruin wafers and must be detected before the process is started. As the semiconductor processes become more sophisticated, they also become less tolerant to contaminants. Residual gas analysis in a process chamber helps eliminate uncontrollable variables and thus increase up-time and production yield and reduce cost of ownership.

RGA's are not limited to the analysis of gases at pressures below 10<sup>-4</sup> Torr. Higher gas pressures can be sampled with the help of a differentially pumped **pressure reducing gas inlet system (PRGIS),** consisting of a restriction and a vacuum pump package. Common restrictions are pinholes and capillaries, which can provide pressure reductions of more than 6 decades of pressure. Vacuum pump packages typically consist of a turbomolecular pump backed by a foreline pump.

## The Open Ion Source (OIS) ( $P < 10^{-4}$ Torr)

The standard ion source used in most commercially available RGA's is the **open ion source (OIS)**. This source is considered the "*do it all*" source for RGA's. It has been around, in its cylindrical, axially symmetrical version since the early 1950's. A schematic of the OIS design used in the SRS RGA Probe is shown in the following figure.

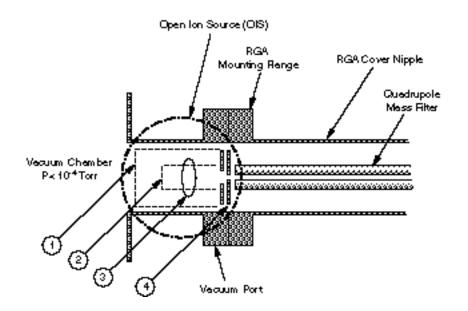


Figure 1 SRS Open Ion Source (OIS) schematic.

1. Repeller, 2. Anode Grid, 3. Filament, 4. Focus plate

The OIS penetrates into the process chamber. The filament wire and the anode wire cage are "open" to the surrounding vacuum chamber. All molecules that are moving around in the vacuum chamber can easily move through the ion source. The pressure in the ionizer is the same as in the rest of the surrounding vacuum and also the same as in the quadrupole mass analyzer and ion detector. The OIS is "*open*" to all the gaseous molecules in the vacuum chamber and can be used to monitor and detect changing gas levels as long as the overall pressure remains under 10<sup>-4</sup> Torr. Higher pressures result in a decrease in sensitivity due to space charge repulsion between ions.

## **Performance limitations of the OIS**

Overall, most commercially available OIS RGA's do a very good job at measuring residual gas levels without affecting the gas composition of their vacuum environment. However, some potential limitations must be kept in mind, particularly when the sensor is used routinely to monitor minute trace impurities (i.e. PPM and sub-PPM levels) or UHV environments ( $10^{-9}$  Torr or less).

The following is a list of the different ways in which an OIS RGA can contribute to its background signals, decreasing the detection capabilities of the sensor:

## Outgassing

The OIS is a **hot-cathode** ion source. The filament wire (i.e. the cathode) must be heated to high temperatures (i.e.> $1300^{\circ}$  C) in order to establish an electron emission current. In the high vacuum, most of the energy required to heat the filament is dissipated to the surroundings through radiative processes. As a result, the entire ionizer and the adjacent walls "*run hot*". The elevated temperatures

result in increased **outgassing** from the OIS itself, and from the adjacent chamber walls. In the most benign cases outgassing simply modifies the composition of the gas mixture being measured. However, under some circumstances outgassing can be a serious problem and even ruin experiments and samples.

The gases emitted by the outgassing process obscure **the minimum detectable partial pressures (MDPP 's)** of many important gases including: H<sub>2</sub>, H<sub>2</sub>O, N<sub>2</sub>, CO and CO<sub>2</sub>. **Degassing** the ionizer can help minimize some of the background signals; however, this is usually only a temporary solution (During a degas the ion source is ramped up to a temperature significantly above its normal operating value, and bombarded with high energy electrons, in order to accelerate the outgassing process.)

Some RGA manufacturers offer UHV versions of their OIS with anodes (and sometimes entire ionizer assemblies) made out of **platinum clad molybdenum** wire. This highly inert material exhibits decreased adsorption for many gases and provides reduced outgassing and ESD.

Water outgassing is a frequent interference, and especially important because it is a serious source of contamination in many high vacuum processes. Overnight **bakeouts** at >200°C are the best option to minimize water outgassing from an OIS RGA.

 $H_2$  outgassing from the OIS electrodes is only a concern for users operating in the UHV regime.  $H_2$  is dissolved in most varieties of SS300 and readily outgasses from the hot OIS electrodes contributing to the background signals for the gas.

#### **Electron Stimulated Desorption (ESD)**

Even after an RGA has been thoroughly baked out, peaks are frequently observed at 12, 16, 19 and 35 amu, which are formed by ESD from surfaces within the OIS rather than by electron-impact ionization of gaseous species. ESD affects the RGA performance in a way similar to regular outgassing.

Several steps can be taken to minimize the effect:

• Degassing with high electron energies.

• Gold plating the ionizer decreases the adsorption of many gases and hence reduces the ESD effect. Using platinum clad molybdenum ionizers is another alternative.

- Reducing the extent of the electron beam.
- Reducing the surface area of the OIS (For example, use wire mesh instead of solid perforated metal.)
- Avoid contaminating the ionizer.

#### **Background interferences**

The quadrupole mass filter assembly has a large surface area in comparison to the ionizer and even though it does not get as hot as the ionizer during operation it still outgasses. The fact that the OIS is exposed to the same vacuum environment as the rest of the sensor makes the ionizer sensitive to the impurities outgassed by the rest of the quadrupole assembly. A serious problem for a lot of RGA users (particularly in the UHV range) is H<sub>2</sub>O outgassing from unbaked RGA's.

However, lots of other species are also present that can affect the background readings. For example, high Ar backgrounds can be expected if the sensor was recently exposed to large levels of the gas because it tends to get adsorbed on all surfaces and desorbs slowly.

The ionizer is also sensitive to impurities generated at the hot filament. Gas molecules can suffer thermal cracking and chemical reactions at the filament surface and the products of the reaction can easily find their way into the ionization region. The impurities generated in this fashion are usually an important source of contamination of the ionizer's surfaces and have a serious effect on the RGA's long term stability. For example, CO and CO<sub>2</sub> are emitted by most hot filaments (Carbon impurities in the filament wire react with Oxygen) and easily find their way into the ionizer and vacuum system.

# Partial Pressure Reduction (PPR) systems (10<sup>-4</sup> Torr < P < 10Torr)

RGA's are not limited to the analysis of gases at pressures below 10<sup>-4</sup> Torr. Higher gas pressures can be sampled with the help of a differentially-pumped **pressure reducing gas inlet system (PRGIS),** consisting of a restriction and a **vacuum pump package**. Common restrictions are pinholes and capillaries, which can provide pressure reductions of more than 6 decades of pressure. Vacuum pump packages typically consist of a turbomolecular pump backed by a foreline pump. The combined RGA, gas inlet system and pumping station constitute what is usually referred to as a **Partial Pressure Reduction (PPR) System**. These systems are commonplace in gas phase processes and are available from most RGA manufacturers.

The PPR system, manufactured by Stanford Research Systems, and depicted in figure 2, is a perfect example of the typical pressure reduction setup used to step process pressures down to levels acceptable to the OIS RGA. The PRGIS actually contains two inlet paths to the RGA: a **high conductivity path** for monitoring base vacuum and a **low conductivity** (i.e. pressure reducing ) path for monitoring gases at operating pressure.

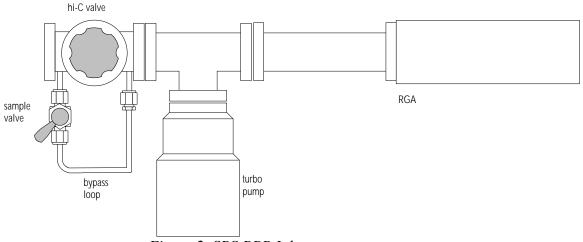


Figure 2 SRS PPR Inlet system components.

The **high conductivity** (**Hi-C**) path is used when the vacuum system is at pressures below  $10^{-4}$  Torr. At high vacuum, typical applications are leak testing and monitoring the ultimate vacuum of the chamber. For example, in a sputtering chamber, the first stage of the process is always a pump-down to  $<10^{-6}$  Torr. At this point the RGA may be used to check the quality of the background for leaks and contaminants. Once the quality of the vacuum is satisfactory the sputtering chamber is backfilled with argon at a few mTorr and sputtering is started.

The **low conductivity** (Lo-C) path is used when the process chamber is at pressures above  $10^{-4}$  Torr. This path contains a micro-hole orifice which reduces the pressure several decades to a level suitable for the RGA (typically around  $10^{-5}$  Torr). Apertures are available for operating pressures as large as 10 Torr. For example, during a sputtering process the RGA may be used to monitor water vapor and hydrocarbon levels to assure they do not exceed certain critical levels that degrade the quality of the sputtered films.

A pair of pumps draws the gas through the aperture to the RGA and establish the pressure drop. The pumps are compact, completely oil-free and require minimal maintenance.

For pressures higher than 10 Torr the gas flow rates into the sample inlet side become extremely small and the time response is too slow for any practical measurements. In those cases, a **bypass pumped sampling system**, with a much larger gass flow rate and faster response, is a much better choice than a single-stage PRGIS. A commercially available example of a bypass pumped system is the QMS Gas Analyzer manufactured by Stanford Research Systems.

# MDPP limitations of a PPR system with an OIS RGA

An OIS RGA connected to a pressure reducing gas inlet system does an excellent job sampling gases at pressures  $>10^{-4}$  Torr. However, even though most of the commercially available RGA's have adequate sensitivity and dynamic range to detect PPM level contaminants in principle, interferences from process gases and background interferences from the sensor itself can make the detection of PPM levels of impurities difficult in practice.

The two basic sources of interference that are observed in PPR systems are:

## **Background interference**

The background gases present in the analyzer chamber can obscure the MDPP's of many important gases (particularly:  $H_2$ ,  $H_2O$ ,  $N_2$ , CO, and  $CO_2$ ). Background gases are due to outgassing, electron stimulated desorption, and the finite compression ratio of the pumping system.

In order to best illustrate the limitation in MDPP levels, lets consider, as an example, the analysis of water in a  $10^{-2}$  Torr Ar sputtering process. During process monitoring, the mass spectrometer typically runs at about  $10^{-5}$  Torr,

corresponding to a three decade reduction factor across the low conductance path of the PRGIS. The pressure drop, brings a 1 PPM of water in the process chamber to a partial pressure in the mass spectrometer of about  $10^{-11}$  Torr (well within the detection limit of a typical RGA). However, with the mass spectrometer isolated form the process gases, the residual pressure in the PPR chamber is, at best, in the order of  $10^{-9}$  Torr with most of that being water. This water level is one hundred times larger than the  $10^{-11}$  Torr corresponding to 1 PPM of water in the process chamber, meaning that the water vapor concentration cannot be reliably measured to better than 100 PPM under these "*very common*" operating conditions. The MDPP limit could be improved to 20 PPM increasing the operating pressure in the RGA chamber to  $5 \cdot 10^{-5}$  Torr during analysis. However, even a 20 PPM MDPP limit for water is not low enough in many cases.

The same limitations must be kept in mind for all other potential interfering gases. In order for any species to be detectable at the PPM level (i.e.  $10^{-8}$  Torr in a 10mTorr process) the residual mass spectrum for the PPR must show pressure readings of less than  $10^{-11}$  Torr at the mass values corresponding to the peaks of that species. Such levels are not easily achieved repeatedly in any vacuum system unless very special precautions are taken to eliminate all sources of contamination. The problem is particularly serious for masses under 50 amu where there is always a variety of background peaks in the residual mass spectrum. Even though the RGA is intrinsically capable of performing sub-PPM measurements, it is usually not easy to find places in the residual mass spectrum of the RGA where the background is readily in the PPM levels.

The MDPP limit for air is usually limited by the compression ratio of the pumping station. In the SRS PPR the N<sub>2</sub> levels usually go down to  $<10^{-9}$  Torr, with oxygen levels approximately 5 times lower. This corresponds to MDPP levels of better than 20 PPM for 28 amu and 4 PPM for 32 amu. However, the MDPP levels at 28 amu are also affected by the CO contribution from the OIS filament.

Other sources of background interference are contamination from pump oil backstreaming into the RGA chamber in systems that use conventional, oil-based, roughing pumps.

Hydrogen is usually impossible to detect at PPM levels because it outgasses readily from the analyzer. A gold coated or platinum clad OIS can help in this case.

#### **Process Gas Interference**

The other limitation to PPM detection levels in a typical RGA based PPR system is caused by interference from the same process gases that are being analyzed.

The best way to illustrate this point is to go back to the example of water analysis in the 10 mTorr Ar sputtering process. We saw that detecting water at better than 20 PPM levels is very difficult unless the PPR chamber is very carefully baked out and protected from water contamination. However, as we will see, this is only part of the problem, there is also a serious interference at m/e 18 from the same Ar used in the sputtering system. The isotope <sup>36</sup>Ar is present at 0.34%. In the electron ionization process, doubly charged argon is formed leading to peaks at m/e 20 (<sup>40</sup>Ar<sup>++</sup>) and m/e 18 (<sup>36</sup>Ar<sup>++</sup>). For 70 eV electron impact energy, a typical level of <sup>36</sup>Ar<sup>++</sup> is 350 PPM. So if you want to detect PPM levels of water in an Ar based sputtering system , you must solve two problems:

- 1. Background contribution of water outgassing from the sensor
- 2. Interference at m/e 18 from  ${}^{36}\text{Ar}^{++}$ .

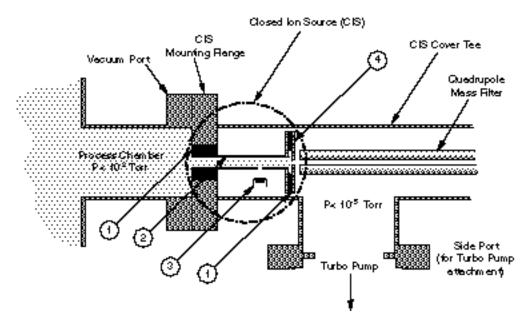
A thorough bakeout can reduces the background water contribution to the low tens-of-PPM levels, but eliminating the  ${}^{36}Ar^{++}$  interference requires the use of several tricks that are used by most RGA users. As usual, some of those tricks work better than others.

Some users simply choose to monitor the m/e 17 peak due to the  $[OH]^+$  water fragment. For 70 eV ionizing electrons, this peak is four times smaller than the main one at 18 amu. This results in a significant reduction in sensitivity for water detection and also adds the problem of abundance sensitivity while trying to measure the mass 17 intensity next to a large  ${}^{36}Ar^{++}$  peak at 18 amu.

A better option (and the one recommended for RGA's with programmable ionizer voltages) is to operate the ionizer with the electron impact energy reduced to <40eV. This ionization energy is below the appearance potential (43.5eV) of  $Ar^{++}$ . For example, the peaks at masses 18, 19 and 20 due to  $Ar^{++}$  disappear while operating an RGA with 35 eV electrons, and this is achieved with minimal reduction in the sensitivity of detection of  $Ar^{+}$  at 36, 38 and 40 amu. Reduction of the electron energy usually imposes an extra load of work on the filament and will reduce its lifetime. However, the reduced interference effects offset the extra costs of filament replacement. As we will see later, the more compact design of closed ion sources makes it very natural for them to operate with 35 eV electrons, without sacrificing the filament's lifetime.

## The Closed Ion Source (CIS) (10<sup>-4</sup>Torr < P < 10<sup>-2</sup>Torr)

In applications requiring the measurement of pressures between  $10^{-4}$  and  $10^{-2}$  Torr, the problem of background and process gas interferences to the mass spectra can be significantly reduced by replacing the traditional OIS PPR configuration described above with a **closed ion source (CIS) sampling system**. A cross section of a generic CIS setup is shown in the following figure.



*Figure 3 Cross section of a generic closed ion source (CIS)* 1. Insulator and seal, 2. Ionization Tube(Anode), 3. Filament, 4. Exit Plate

The CIS Ionizer sits on top of the quadrupole mass filter replacing the more traditional OIS used in conventional RGA's. It consists of a short, gas-tight tube with two very small openings for the entrance of electrons and the exit of ions. Electrons enter the ionizing region through an entrance slit of small dimensions. The ions are formed close to, and attracted by, a single extraction plate and exit the ionizer through a circular aperture of small diameter. Alumina rings seal the tube from the rest of the quadrupole mass assembly and provide electrical insulation for the biased electrodes. Ions are produced by electron impact directly at the process pressure. A pumping system, similar to the one used in PPR systems, keeps the filament and the rest of the quadrupole assembly at pressures below 10<sup>-5</sup> Torr through differential pumping. The design is very simple and was successfully used for many years in gas chromatography/mass spectrometry instruments before it was applied in general to quadrupole gas analyzers.

## Advantages of the CIS

An understanding of the performance differences between the CIS setup and the more traditional RGA based PPR system is indispensable when trying to choose the sensor setup that is best suited for a particular process application. A CIS system usually offers several advantages over the OIS PPR. Process engineers should carefully weigh those differences before choosing an analyzer configuration for their application.

#### **Direct sampling**

The CIS Anode can be viewed as a high conductance tube connected directly to the process chamber. The pressure in the ionization area is virtually the same as that in the process chamber. The CIS Ionizer produces ions by electron impact directly at process pressure, while the rest of the mass analyzer and the filament are kept under high vacuum. The electrons emitted by the filament in the high vacuum region enter the high pressure ion formation region through the emission aperture on the side of the anode tube. Once formed, the ions are extracted for mass analysis through the ion extraction orifice centered on the exit plate. Direct sampling provides good sensitivity due to the large ion densities available and also fast response times. The "*memory effects*", typically associated to pressure reduction and conductance orifices, are significantly reduced.

#### Improved Signal-to-background ratios

Because the sampling pressure in the CIS is typically two decades higher than that of the rest of the sensor's vacuum system, the signal-to-background ratio is significantly increased relative to the OIS PPR systems. *This is particularly important when measuring common residual gases, such as water*. In order to best illustrate this point we go back to the example of a 10<sup>-2</sup> Torr Ar sputtering process. The sampling orifice in the OIS PPR inlet system reduced the Ar partial pressure from 10<sup>-2</sup> down to 10<sup>-5</sup> Torr at the OIS. Water vapor levels were measured in a background of at least 10<sup>-9</sup> Torr of residual water. This corresponded to a 100 PPM MDPP limit for water. Things are much better in the CIS system. In this case, the Ar gas is ionized directly at 10<sup>-2</sup> Torr ( i.e. three orders of magnitude higher than in the OIS PPR!) but in the same background (10<sup>-9</sup> Torr) of residual water. This residual level now corresponds to a 100 PPB MDPP level for water in the CIS system. *This is quite an improvement over the OIS PPR performance*!

The combination of direct sampling and differential pumping provides the potential for PPM and sub-PPM detection limits for even the most pervasive residual gases.

For other common interferences, such as organic contaminants or reaction byproducts of the filament, the gas tight design of the source reduces the visibility of the ionization region to those gases providing a very clean residual gas spectrum, free of many of the spectral overlaps that are common in OIS PPR setups.

Interference from contaminants generated by ESD is also reduced in the CIS because a much smaller electron beam penetrates the ionizing volume. In addition, the inside walls of most commercially available CIS's are made out of highly inert materials such as gold, platinum clad and pure molybdenum which adsorb less impurities than stainless steel.

#### **Reduced Ionizer Contamination**

In an OIS PPR system, sample molecules that have suffered thermal cracking or chemical reaction at the filament, are free to drift into the ionization region. This is a very significant source of surface contaminants for electron impact ionizers. In contrast, the gas tight design of the CIS reduces the visibility of the source to those contaminant gases, providing reduced contamination and better long term stability. Most CIS manufacturers utilize exclusively Tungsten filaments in their systems. W resists many corrosive gases (such as  $WF_6$ ) and reactive gases (such as Silane) minimizing reactions at the filament that contribute to the background, also resulting in extended filament lifetime.

#### Versatility

CIS based systems are extremely versatile. The low detection limit together with the wide dynamic range provided by the quadrupole mass filter fitted with an electron multiplier, makes the CIS a versatile system for: on-line process monitoring and control, verification of process gas purity at the point of use, high vacuum residual gas analysis, and process equipment leak checking.

Different modes of operation can easily be achieved by simply changing some of the sensor's operating parameters. A CIS Gas Analyzer, even though not as sensitive as an RGA, can tackle most residual gas analysis and leak checking tests. The sensitivity of the CIS is reduced over the OIS because of the very small holes for electron entrance and ion exit. However, in most cases this reduction in sensitivity is easily made up by running the electron multiplier at higher gain levels than the RGA. The electron emission current is usually raised while using the sensor for residual gas analysis to increase sensitivity, and reduced during process monitoring to avoid space charge saturation effects in the ionizing volume. The tight design of the CIS also makes it possible to operate the ionizer at lower electron ionization energies than are possible with OIS's. As a result, most of the commercially available CIS systems offer two modes of operation with electron ionization energies of 70 and 35 eV. The 70eV setting is mostly used for leak testing and routine residual gas analysis. The spectra collected are virtually identical to those obtained with standard RGA's. The 35eV setting is used during process monitoring to eliminate process gas interference peaks. A good example is the elimination of the doubly ionized  ${}^{36}Ar^{++}$  peak that interferes with water detection at 18 amu in sputtering processes. Different electron ionization energies are often used to selectively ionize species in a gas mixture.

# High Pressure Sampling with a CIS Analyzer (10<sup>-2</sup>Torr < P < 10Torr)

CIS Analyzers can usually sample gases *directly* up to about  $10^{-2}$  Torr pressure levels. The upper pressure limit is set by the reduction in mean free path for ion-neutral collisions, which takes place at higher pressures, and results in significant scattering of ions and reduced sensitivity. *However, operation is not limited to the analysis of gases at pressures below*  $10^{-2}$  *Torr*. Higher gas pressures can be sampled with the help of a differentially pumped **pressure reducing gas inlet system (PRGIS)** just as it is done with conventional RGA's. A pressure reducing gas inlet system matched to the conductance of the CIS analyzer will allow the sensor to sample gas pressures as large as 10 Torr. As in the case of the PPR systems, the penalty paid is reduced sampling speed at the sample inlet and possible memory effects at the ionizer. However, these problems can

be usually eliminated by the addition of bypass pumping in the ionizing region as the process pressures start to get large.

## Conclusions

Strict modern-day contamination control requirements for gas phase processes are constantly pushing the limits of performance of quadrupole gas analyzers. The technology is rapidly evolving and adapting to lower contamination level specifications. A good understanding of the different factors affecting the detection limits of the different gas analysis systems currently available, is an essential tool while selecting a sensor for a specific application. As it is usually the case, most choices involve compromises, and a good understanding of the basic tradeoffs will minimize mistakes and maximize productivity. A well-matched gas analyzer rapidly becomes an integral part of the processing system, and its information eliminates most of the guesswork that is part of all troubleshooting procedures.

The main purpose of this application note was to introduce the basic concepts required to choose the best gas analyzer for any practical application, and also to present some of the basic operating principles that must be kept in mind to assure the optimum performance of the instrument selected.

All gas phase processing setups can benefit from the addition of a quadrupole gas analyzer. Miniaturization, smart software interface, and improved detection limits are some of the features to look for in future instruments. As sensors become more affordable, they will rapidly become commonplace in all industries requiring the strict control of contamination levels in process gasses.

## Declaration of Contamination of Vacuum Equipment

The repair and/or service of vacuum equipment or components can only be carried out if a completed declaration has been submitted. SRS reserves the right to refuse acceptance of vacuum equipment submitted for repair or maintenance work where the declaration has been omitted or has not been fully or correctly completed. SRS also reserves the right to refuse servicing any vacuum equipment which could potentially be harmful to the personnel carrying out the repair and service of the equipment. Contact SRS to request additional copies of this form or if you have any questions regarding the contents of this declaration.

#### **Description of equipment**

Equ	uipment type/model:				
Ser	rial No:				
Re	eason for return (circle one):	• Repair	•	Maintenance	
Ple	ease describe:				
Eq	quipment condition				
1. 2.	Has the equipment been used ? (circle of Describe the operating environment the			No	
1.	Was any of the equipment exposed to p • No	otentially harm	ful substances	? (circle one)	
	• Yes. Please attach list of all known associated with the substance a Were any of the harmful subst	and first aid me	-	•	nbol, precautions
	♦ Radioactiv	ve?	• Yes	• No	
	◆ Toxic?		• Yes	• No	
	<ul> <li>Corrosive</li> </ul>		• Yes	• No	
	<ul> <li>Explosive</li> </ul>		• Yes	• No	
	Was equipment decontaminate	ed/cleaned befo	re being shipp	ed to SRS?	
	• Yes	• No	• Not Ap	plicable	
* *	SRS will not service any equipment that SRS will not accept any equipment white evidence that such equipment has been	ch has been rad	dioactively, or		

#### **Legally Binding Declaration**

I hereby declare that the information supplied on this form is complete and accurate. The dispatch of equipment will be in accordance with the appropriate regulations covering Packaging, Transportation and Labeling of Dangerous Substances. Name (print):

Name (print).		
Job Title:		
Organization:		
Address:		
Telephone:	Fax:	
Legally binding signature:		_ Date:

## **Glossary of Terms**

The following is a listing of some of the most important terms used throughout the SRS RGA Operations Manual. For a more complete listing of terms relevant to partial pressure analyzers in general, refer to:

"A Dictionary of Vacuum Terms used in Vacuum Science and Technology, Surface Science, Thin Film Technology and Vacuum Metalurgy", edited by M. S. Kaminsky and J. M. Lafferty, published by the American Vacuum Society, 1979.

A. Basford et. al., J. Vac. Sci. Technol. A 11(3) (1993) A22-40: "Recommended Practice for the Calibration of Mass Spectrometers for Partial Pressure Analysis. Update to AVS Standard 2.3".

Anode Grid (Ionizer Component): Positively biased grid cage of the ionizer within which the ionization of the gas molecules takes place. <u>Note</u>: In the SRS RGA, the voltage bias of the anode grid, in Volts, sets the ion energy (in eV) for the spectrometer.

Atomic mass unit (abbreviation: amu). A unit of mass equal to one twelfth the mass of a neutral carbon atom having six protons and six neutrons ( $^{12}$ C); equivalent to 1.660566 $^{10}$ <sup>27</sup> kg.

**Background signal,**  $H_0$ . Output signal, measured with respect to baseline, which is obtained before the introduction of any gas in the chamber.

**Base pressure (also Background pressure).** Total pressure before introduction of any gases into a vacuum system. Usually, the base pressure is the lowest pressure that is typically achieved in the vacuum chamber.

Baseline. The output signal from the RGA when no ions are arriving at the detector.

**CDEM.** Abbreviation for the type of electron multipliers known as: Continuous Dynode Electron Multiplier.

**CF.** Abbreviation for Conflat type flange.

**CF Nipple**. A short section of vacuum pipe terminated with a standard Conflat flange connector at each end.

**Charge**, **Q**. The electron charge of an ion. Ion charge occurs in multiples of the electron charge, e<sup>-</sup>.

Drift. A change in time in the avearge output signal at constant partial pressure.

ECU. Abbreviation for electronics control unit.

**Electron emission**. The release of electrons from the heated filament in the ionizer. The electrons are accelerated into the **anode grid** where the ionization of the gas molecules occurs.

**Electron emission current.** The electron current from the filament to the grid in mAmps.

Ion current signals scale linearly with the electron emission current. <u>Note</u>: The available emission current range in the SRS RGA is 0 to 3.5 mA.

**Electron Energy**. The kinetic energy of the electrons (in eV) used for electron bombardment in the ionizer. <u>Note:</u> In the SRS RGA the Electron Energy is equal to the voltage difference (in Volts) beween the filament and the anode grid.

**Electronics Control Unit (abbreviation: ECU)**. Electronics box that attaches directly to the probes feedtrhu flange and contains all the necessary components to operate the quadrupole mass spectrometer and communicate with a host computer.

**Faraday Cup**. A charged particle detector, consisting of a metal electrode, for the direct collection and detection of charged particles. <u>Note:</u> Typical designs are cup shaped to minimize secondary electron losses.

**Filament (Ionizer Component).** The source of the ionizing electrons. Thin, thoria coated, Iridium wire that operates at a negative potential relative to ground and is resistively heated to incandescence with an electrical current from the emission regulator. The thermionically emitted electrons are accelerated towards the anode grid which is positively charged with reference to the filament and ground.

**Focus Plate (Ionizer component).** Ionizer's electrode plate that serves the double purpose of drawing the ions away from the anode grid, and containing the ionizing electrons inside the source.

Fragment ion. An ion of mass smaller than that of the original parent molecule.

**Fragmentation factor**. The fragmentation factor of gas g at mass M is defined as the ratio of ion signal at mass M to the ion signal at the principal mass peak of gas g.

**Fragmentation of molecules**: The breaking of multi-atomic molecules into units of fewer atoms, some of which are usually electrically charged.

**Fragmentation (or cracking) pattern**. The fragment distribution of ionic species which results from dissociation and ionization of multi-atomic molecules of given species in the ionizer.

**Head (also RGA Head).** The combination of the Probe and the Electronics Control Unit.

**Inert Gas**. A gas that does not normally react chemically with other substances. Example: He, Ar, Kr and Xe. Typically used for calibration purposes in RGA's.

**Ion**. An atom or molecule which has gained or lost one or more electrons and therefore has a negative or positive charge. Note: Most RGA's use electron bombardment to ionize molecules and detect only positive ions.

Ion current. The rate of ion flow into the detector. Usually expressed in units of amps.

**Ion Energy.** The kinetic energy of the ions as they move down the quadrupole mass filter, and expressed in eV. <u>Note</u>: In the SRS RGA the Ion Energy is equal to the voltage biasing of the anode grid in Volts and has two possible settings (i.e. Low and High).

**Ionization.** The process that results in the formation of ions from neutral atoms or molecules. During ionization, electrons are added or removed from the molecules to form negative or positive ions respectively. <u>Note</u>: In the SRS RGA the ionization is caused by electron bombardment; outer electrons are removed by the impact of energetic electrons on the molecules.

**Ionization efficiency**. The ionization probability normalized to the probability of ionization of a reference gas.

**Ionization Potential**. The minimum energy per unit charge (often in eV) required to remove an electron from an atom (or molecule) to infinite distance. <u>Note</u>: In the SRS RGA the Electron energy must be set above the ionization potential of the molecules for ionization to occur.

**Ionization probability**. The number s of ion pairs (equal amounts of positive and negative charges appear as ions) produced by an electron traveling a unit ditance (typically 1 cm) through a gas at unit pressure (typically 1 mTorr). It depends on the ionization potential of the electrons used for bombardment.

**Ionizer (Probe Component).** The portion of the mass psectrometer probe that generates the ions and accelerates them as a beam. <u>Note:</u> The components of the SRS RGA ionizer are: anode grid, repeller, focus plate, and filament.

**Linearity**. The extent to which the change in output signal of the RGA is proportional to the corresponding change in partial pressure.

**Linear response range**. The partial pressure range over which linearity in the signal response is observed. See linearity.

**Mass-to-charge ratio:** The mass-to-charge ratio,  $\mathbf{M}/\mathbf{Q}$ , is defined as the ratio of the mass number M of the ion to its charge Q, measured in units of the electron charge e<sup>-</sup>. For example: doubly charged ions of argon isotope 36 ( $^{36}\text{Ar}^{2+}$ ) and singly charged ions of water,  $^{1}\text{H}_{2}^{16}\text{O}^{1+}$ , have  $\mathbf{M}/\mathbf{Q} = 18$ , and cannot be differentiated from each other with most mass spectrometers. **Note:** Mass spectrometers do not actually measure the molecular mass directly, but rather the mass-to-charge ratio of the ions. For singly charged ions, the mass to charge ratio is numerically equal to the mass of the ion in atomic mass units (amu).

**Mass analyzer (Probe component)**. The portion of the mass spectrometer probe that separates the ion beam into its various mass-to-charge ratio components. Also referred to as Quadrupole mass filter.

**Mass Number, M**. The mass number, M, is the sum of the number of protons and neutrons in an atom or molecule.

**Mass peak.** The ion current pattern in the vicinity of maximum amplitude by scanning through a small portion of the mass range containing ions of a single mass-to-charge ratio. Very often, the term "mass peak" refers to the signal developed from singly charged ions. For example, nitrogen is said to have a "mass 28 peak".

**Mass Range.** The range of mass numbers defined by the mass number of the lightest and the heaviest singly charged ions which can be detected by the RGA.

**Mass spectrometer**. An instrument which produces a beam of molecular ions from a sample, separates the resulting mixture of ions according to their mass-to-charge ratios, and provides output signals which are measures of the realtive abundances of the ionic species present.

Mass spectrum. A graph of ion abundance vs mass-to-charge ratio.

Minimum Detectable Partial Pressure change (MDPP). The partial pressure change corresponding to the smallest signal change which can be distinguished from noise. A common prescription is,  $\Delta p_{min} = \sigma/S_g$ , where  $\sigma$  is the noise and  $S_g$  is the partial pressure sensitivity for the gas being measured.

**Noise**. Random fluctuation in the output signal unrelated to a change in the partial pressure of the gas from which the signal is derived. The appropriate measure of noise is the standard deviation  $\sigma_N$  of N independent determinations of the average output signal obtained at constant partial pressure.

**Open Ion Source (Also Nude Ion Source).** An ionizer with electrodes of high transparency to gases.

**Outgassing**. The evolution of gas molecules from the internal wall surfaces of the vacuum system.

Parent ion. An ion of the same mass as that of the original parent molecule.

**Partial Pressure**. The pressure of a designated component of a gaseous mixture within a system as it is exerted on the chamber walls. The sum of the partial pressures of all the kinds of gases gives the total pressure.

**Partial Pressure Analyzer (abbreviation: PPA).** A compact mass spectrometer used to analyze the residual gas composition in a vacuum system.

**Peak height (also peak intensity).** The maximum ion signal developed in the RGA for a given mass-to-charge ratio peak.

**Peak width** ( $\Delta M_{v\%}$ ). The difference between the mass-to-charge ratio values on either side of a mass peak at which the signal has dropped to v% of the peak height, H. See also Resolution.

**Principal mass peak.** The most intense peak in the fragmentation pattern of any molecule.

**Probe** (also RGA Probe). Quadrupole mass spectrometer sensor consisting of an ionizer, a mass analyzer and a detector (Farday cup or optional electron multiplier).

**Repeller (Ionizer Component).** The repeller grid cage completely encloses the ionizer, is biased negative relative to the filament, and prevents the loss of electrons from the ion source.

**Residual Gas Analyzer (abbreviation: RGA).** A compact mass spectrometer used to analyze the residual gas composition in a vacuum system (same as Partial Pressure Analyzer).

**Resolution, or Absolute Resolution,**  $\Delta M_{10\%}$ . The width  $\Delta M$  of the pass band of the filter, defined as the full width at which the ion current falls down to 10% of the maximum value. In mass-to-charge ratio units. See also Peak width.

**Resolving Power:**  $\mathbf{R} = \mathbf{M}/\Delta \mathbf{M}_{10\%}$ : Ratio between a particular mass-to-charge ratio  $\mathbf{M}$  and the resolution,  $\Delta \mathbf{M}_{10\%}$ , at that mass. Dimensionless ratio.

RGA. Abbreviation for Residual Gas Analyzer.

RGA Cover Nipple. CF Nipple that covers the RGA Probe.

**Scan Speed** (mass spectrometer). The speed at which the RGA scans through a range of succesive mass numbers.

**Scanning.** The procedure of continuously changing the mass tuning of the quadrupole mass spectrometer to bring successive mass numbers into tune.

#### Sensitivity.

#### Partial Pressure:

The partial pressure sensitivity of the RGA to a gas g,  $S_g$ , is defined as the ratio of the change  $(H-H_0)$  in principal mass peak height to the corresponding change  $(P-P_0)$  in total pressure due to a change in partial pressure of the particular gas species.  $H_0$  and  $P_0$  are background values.

$$S_g = (H-H_0) / (P-P_0)$$

The units of  $S_g$  are of ion current per unit pressure (amp/Torr, for example).

#### **Total Pressure:**

The total pressure sensitivity of the RGA to a gas g, is defined similarly as the ratio of the change  $(H-H_0)$  in total ion current to the corresponding change  $(P-P_0)$  in total pressure due to a change in partial pressure of the particular gas species.  $H_0$  and  $P_0$  are background values.

**Sensitivity calibration**. The act of establishing a a correspondence between the change in ion current and the corresponding change in partial pressure of the gas from which the ion is produced. The correspondence might be represented graphically or as a table of values. See also Sensitivity.

**Space charge**. The electrical charge carried by a cloud of free electrons or ions. Space charge can causes serious changes in the potential distributions of the ionizer, and ultimately limits the pressure operating range of all RGA's.

**Total Pressure**. The average normal force per unit area exerted by all the gas molecules impacting on the internal surfaces of the vacuum chamber. Units are typically Torr, mbar or Pascal. Note: The SRS RGA is capable of collecting total ion currents that can be turned into total pressure measurements with the help of gas dependent total pressure sensitivity factors.

**UHV**. Abbreviation for Ultra High Vacuum. Pressure  $< 10^{-9}$  Torr.