

# Partial Pressure Analysis with RGAs

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AVS MID-ATLANTIC CHAPTER SPRING PROGRAM

April 21, 2005

Jefferson Laboratory



**Jefferson Lab** 

The logo for Jefferson Lab, consisting of the text "Jefferson Lab" in a bold, sans-serif font, followed by a blue graphic element that resembles a stylized swirl or a wave.

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# Education

- **B.S. Analytical Chemistry** - University of Buenos Aires, Argentina (1980-1985)
- **Ph.D. Physical Chemistry** - Colorado State University, Fort Collins, CO (1986-1990)
- **Post Doc Physical Chemistry** - University of Southern California, Los Angeles, CA (1991-1992)

# Work Experience

- Stanford Research Systems, Inc. (1992-present)
- [www.thinkSRS.com](http://www.thinkSRS.com)
- Design Engineer, Senior Chemist, Product Manager and Product Design Manager
- Vacuum & Analytical Instruments Division Manager

# **Job**

Design and commercialize scientific instrumentation

## **Design Philosophy**

Build affordable, high quality instrumentation without any compromise in performance.

“All the features and more for half the price”

## **Vision**

Make SRS a brand name in the vacuum and analytical chemistry markets

# Preliminary statements

- I am not the vacuum expert in the room. You are!
- What I learn from you today will help me direct SRS's future efforts in vacuum technology. Share your wishes.
- If I do not know the answer to your question I will let you know.
- The RGA is my baby, but do not let that discourage you.
- I tend to talk too fast. Stop me if it gets out of control.

- In case you did not notice, I do have an accent... Fell free to make me repeat myself.
- Ask questions any time you want, you can bring me back if I get lost.
- Some slides are pretty loaded. Do not worry, I will go through them with you.
- I believe in repetition, don't get annoyed if I repeat some things a few times....
- Most of what I will say today is in the SRS RGA Manual.

- I will use RGA Windows ver. 3.0 throughout the software demo. Ver 2.3 is officially obsolete.
- I will use the SRS RGA as my model to explain the physics of quadrupole mass spectrometers. However, all quad RGAs work pretty much the same way.
- This seminar is not a substitute for the RGA's Operation Manual!

# About SRS

- Private company
- Scientific and Test&Measurement Instrumentation
- First commercial product 1982 - Boxcar Integrator
- First vacuum product 1995 - SRS RGA
- Wide product range
- R&D engineers have wide responsibilities
- In-house manufacturing

[Home](#) > **Products**

*[New Catalog Available! ... click here](#)*

## SRS Product Categories

### Scientific Instruments

- Small Instrumentation Modules
- Lock-In Amplifiers
- Optical Chopper
- Preamplifiers
- Digital Delay Generators
- High Voltage Power Supplies
- Gated Integrators and Boxcar Averagers
- Photon Counter

### Time & Frequency Instruments

- Frequency Standards/Oscillators
- Time Interval and Frequency Counters

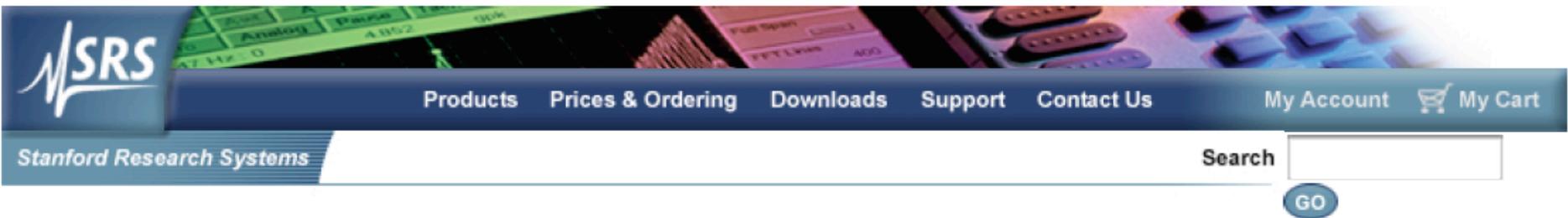
### Test & Measurement Instruments

- Synthesized Clock Generator
- Synthesized Function Generators
- FFT Spectrum Analyzers
- LCR Meters
- Thermocouple Monitor
- Dual Channel Programmable Filters

### Vacuum & Analytical Instruments

- Melting Point Apparatus
- Quartz Crystal Microbalances
- Nitrogen Laser
- Ion Gauge Controller
- Residual Gas Analyzers
- High Pressure Sampling Systems

# Vacuum and Analytical Instruments



[Home](#) > [Products](#) > **Vacuum & Analytical Instruments**

*[New Catalog Available! ... click here](#)*

## Vacuum and Analytical Instruments

### Melting Point Apparatus (OptiMelt)

- [MPA100 Automated Melting Point System](#)

### Quartz Crystal Microbalance

- [QCM100 Quartz Crystal Microbalance](#)
- [QCM200 Quartz Crystal Microbalance](#)

### Nitrogen Laser

- [NL100 Nitrogen Laser](#)

### Ion Gauge Controller

- [IGC100 Ion Gauge Controller](#)
- [PG105U Pirani Gauge](#)
- [Bayard-Alpert Gauges](#)

### Residual Gas Analyzers

- [RGA100 100 amu Residual Gas Analyzer](#)
- [RGA200 200 amu Residual Gas Analyzer](#)
- [RGA300 300 amu Residual Gas Analyzer](#)
- [PPM100 Partial Pressure Monitor for RGA](#)
- [CIS100 100 amu CIS Analyzer](#)
- [CIS200 200 amu CIS Analyzer](#)
- [CIS300 300 amu CIS Analyzer](#)

### High Pressure Sampling Systems

- [QMS100 100 amu Atmospheric Sampling System](#)
- [QMS200 200 amu Atmospheric Sampling System](#)
- [QMS300 300 amu Atmospheric Sampling System](#)
- [PPR100 100 amu Process Monitoring System](#)
- [PPR200 200 amu Process Monitoring System](#)
- [PPR300 300 amu Process Monitoring System](#)

# Vacuum Products

# RGAs and Gas Sampling Systems

# Residual Gas Analyzer (RGA)



...starting at  
\$3,750...

- *100, 200 and 300 amu systems*
- *Better than 1 amu resolution*
- *6 decades of dynamic range*
- *$5 \times 10^{-14}$  Torr detection limit*
- *RGA Windows & LabVIEW software*
- *Field-replaceable electron multiplier and filament*
- *RS-232 interface*

# Did the world need another RGA?

## Yes!

SRS RGA was designed with these goals in mind:

- Low Cost
- Serviceability
- Reliability
- Ease of Use

# Unique features

- Simplicity of Design (minimalist's approach)
- Self aligned probe assembly
- UHV compatible
- Small insertion volume
- Low surface area (rapid pump-down)
- Low Maintenance
- Field replaceable: filament, ionizer, multiplier
- Long life , state-of-the art electron multiplier
- Computer Interface w/high level command set
- Powerful; operating modes built into RGA head
- Multiplexing capability

# more unique features...

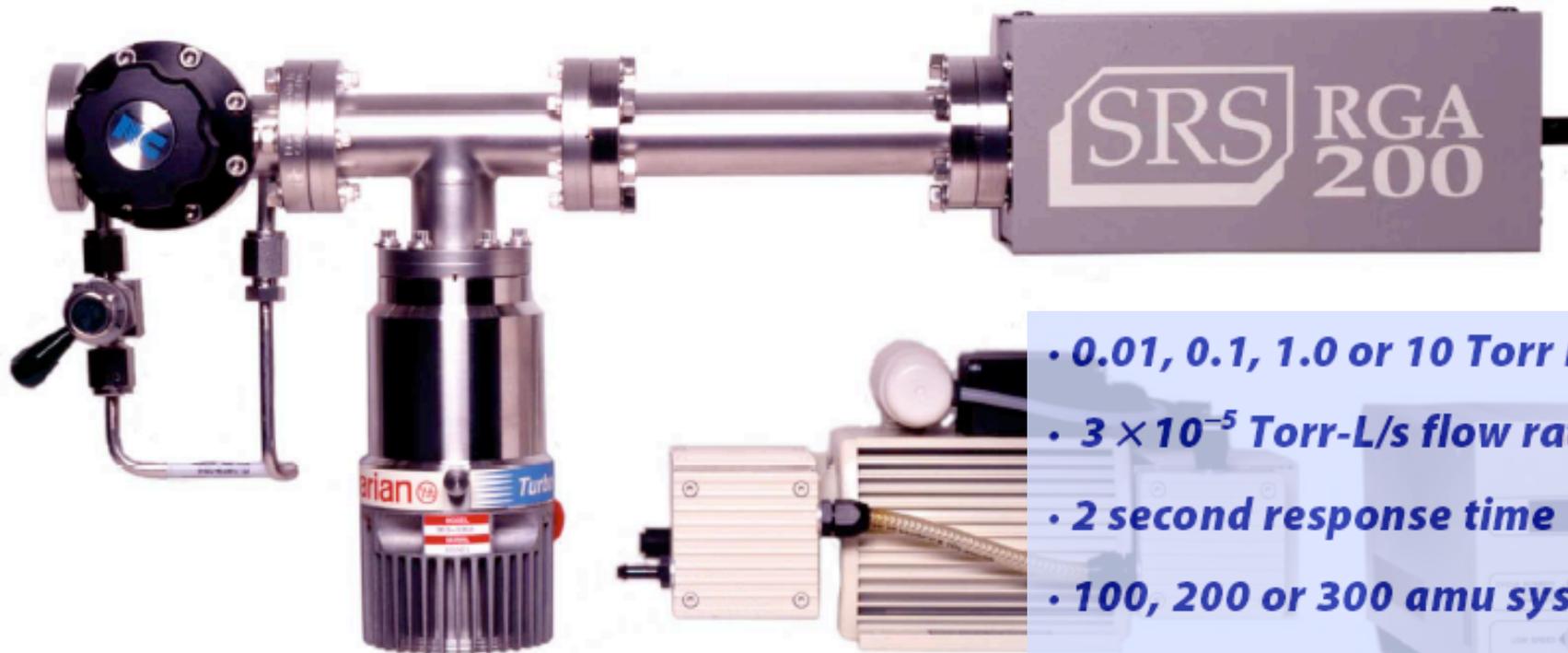
- Built-in filament and multiplier protection
- Removable source
- Upgrade options for detector and mass range
- Stable operation
- Built in power module
- Logarithmic pico-ammeter (auto-ranging electrometer)
- LED Status indicators
- Free software
- Multiplexing capabilities
- Ion Counting output (electron multiplier)
- Low cost
- Good Manual

# How many SRS RGA's does Jefferson Lab own?

**77**

**26 RGA200 & 51 RGA100 - starting Jan 1996** (as of 4/5/2005)

# Process Pressure Reduction System (PPR)



- 0.01, 0.1, 1.0 or 10 Torr inlet pressure
- $3 \times 10^{-5}$  Torr-L/s flow rate
- 2 second response time (0.1 Torr)
- 100, 200 or 300 amu systems
- Field-replaceable electron multiplier and filament
- 6 orders of magnitude dynamic range
- RGA Windows software

# Closed Ion Source System (CIS)



- *100, 200 and 300 amu systems*
- *1 ppm detection*
- *10 mTorr max. operating pressure*
- *$1 \times 10^{-12}$  Torr detection limit*
- *RGA Windows software*
- *Field-replaceable electron multiplier and filament*
- *RGA and CIS mode*

# Atmospheric Sampling System (QMS)



- *100, 200 or 300 amu systems*
- *Pressures from 10 mbar to 1 bar*
- *Better than 1 amu resolution*
- *<0.5 second response time*
- *Compact, transportable design*
- *6 decades of dynamic range*
- *RGA Windows software*
- *RS-232 computer interface*

# Vacuum System Controllers

# Ionization Gauge Controller (IGC100)



- 1000 Torr to UHV range
- Highly accurate, stable controller
- Pressure vs. time curves
- 4 analog input/output ports
- RS-232 interface
- 8-channel process control (opt.)
- GPIB and Web interfaces (opt.)

# LCD Display

Pressure SR 03:50 PM

IG1	8.00-07	fil 1 Manual Torr IG1
IG1	 8.00-07	fil 1 Manual Torr
IG1	 8.	

Menu Gauges Process

*Numeric readout, bar graph*

Chart 14:44

	IG1	PG2	CM1
current	6.34-06	0	0
14:36:32	*	6.49	0

1e+003 10Volts

Y Axis

X Axis

Setup

Help

Back

Pressure 1e-005 60sec / div span



*Pressure vs.*

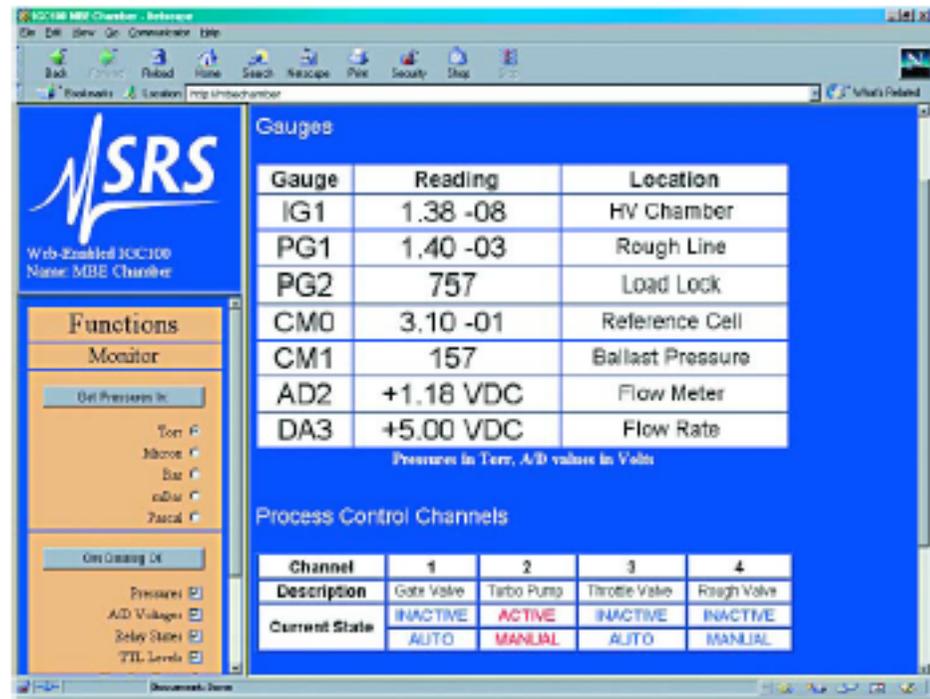
12:39

ROUGH VALVE 1	Auto	GATE VALVE 3	FORE STATUS 4
CLOSED	-----	CLOSED	OK
Inactive	Inactive	Inactive	Inactive
Manual	-----	Auto	Manual
VENT VALVE 5	Active	Channel 7 7	Channel 8 8
VENTING	-----	Inactive	Inactive
Active	-----	Manual	Manual
Manual	Edit rules		
	Edit Messages		

Pressure Help Pressure Help

*8-channel process control*

# Web Access.....



The screenshot displays a web browser window with the URL <http://mbschamber>. The page features the SRS logo and the text "Web-Enabled IGC100 Name: MBB Chamber".

**Functions**

**Monitor**

Get Pressures In

- Torr
- Micro
- Bar
- mBar
- Torr

Get Gauges On

- Pressure
- A/D Voltage
- Relay State
- TTL Levels

**Gauges**

Gauge	Reading	Location
IG1	1.38 -08	HV Chamber
PG1	1.40 -03	Rough Line
PG2	757	Load Lock
CM0	3.10 -01	Reference Cell
CM1	157	Ballast Pressure
AD2	+1.18 VDC	Flow Meter
DA3	+5.00 VDC	Flow Rate

Pressures in Torr, A/D values in Volts

**Process Control Channels**

Channel	1	2	3	4
Description	Gate Valve	Turbo Pump	Throttle Valve	Rough Valve
Current State	INACTIVE	ACTIVE	INACTIVE	INACTIVE
	AUTO	MANUAL	AUTO	MANUAL

*Sample of IGC100 web page*

Check and control your system from your desk...

# Loaded back panel.....



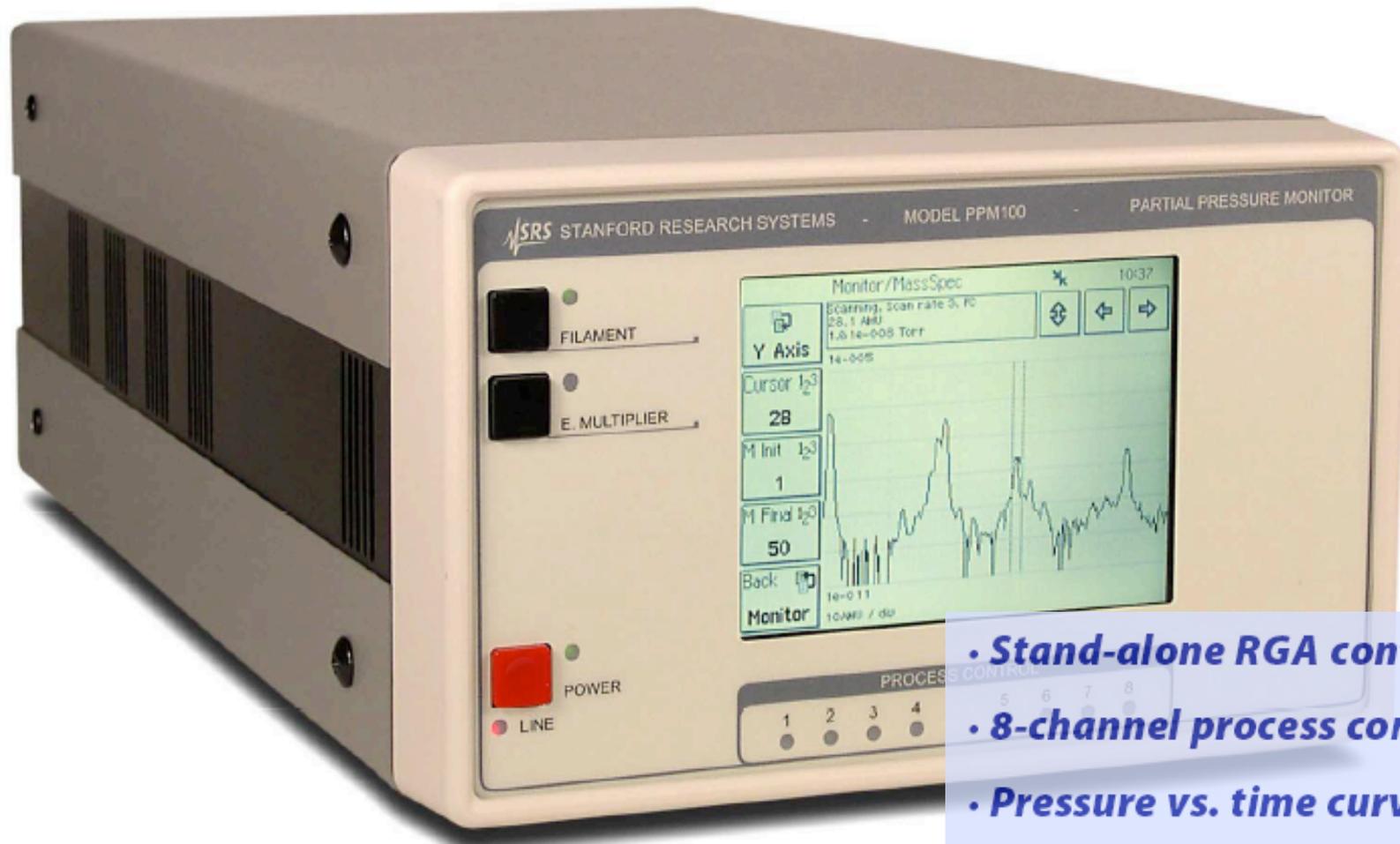
... powerful Vacuum System Controller

# Pirani Gauge (PG105-UHV)



- **1000 Torr to  $10^{-4}$  Torr range**
- **UHV compatible**
- **Bakeable to 250 °C**
- **Fast response time**
- **Compatible with IGC100 controller**

# Partial Pressure Monitor (PPM100)

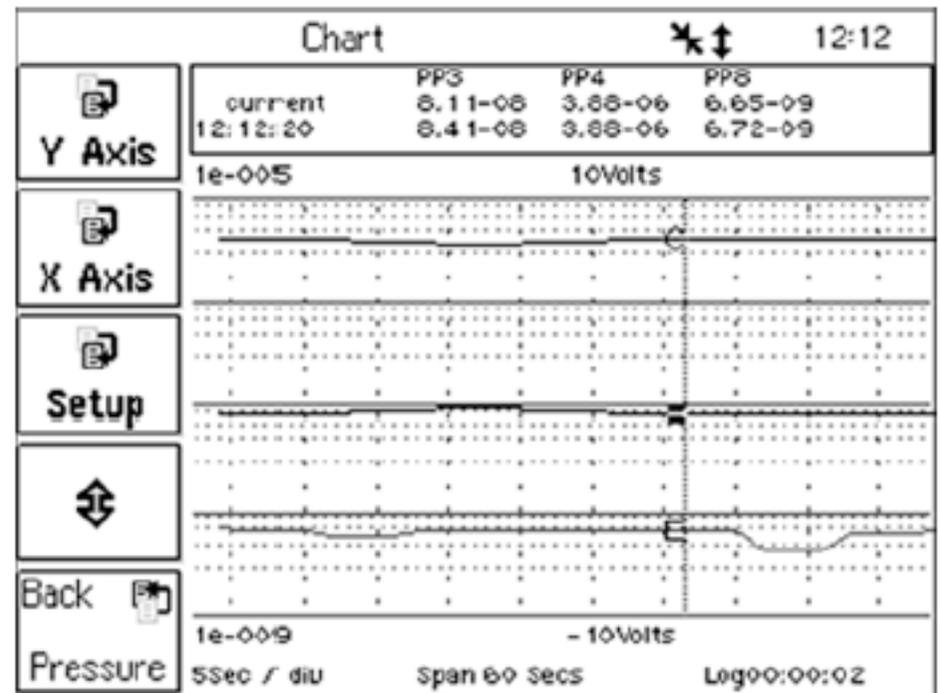


- **Stand-alone RGA controller**
- **8-channel process control**
- **Pressure vs. time curves**
- **4 analog input/output ports**
- **RS-232, GPIB and USB interfaces**
- **Ethernet interface**
- **PPM100 ... \$2295 (U.S. list)**

Monitor				⌘ ↕	11:54
PP1 2 <b>6.09-08</b> Hydrogen	PP2 4 <b>4.95-09</b> Helium	PP3 18 <b>1.03-07</b> <b>Alarm High</b> Water	PP4 28 <b>3.86-06</b> Nitrogen		
PP5 32 <b>7.70-07</b> <b>Alarm High</b> Oxygen	PP6 40 <b>5.86-08</b> <b>Alarm Low</b> Argon	PP7 43 <b>2.90-09</b> Pump oil	PP8 44 <b>7.02-09</b> CO2		
<b>Pressure</b>	<b>AnalogIO</b>	<b>MassSpec</b>	<b>Back</b>	<b>Help</b>	

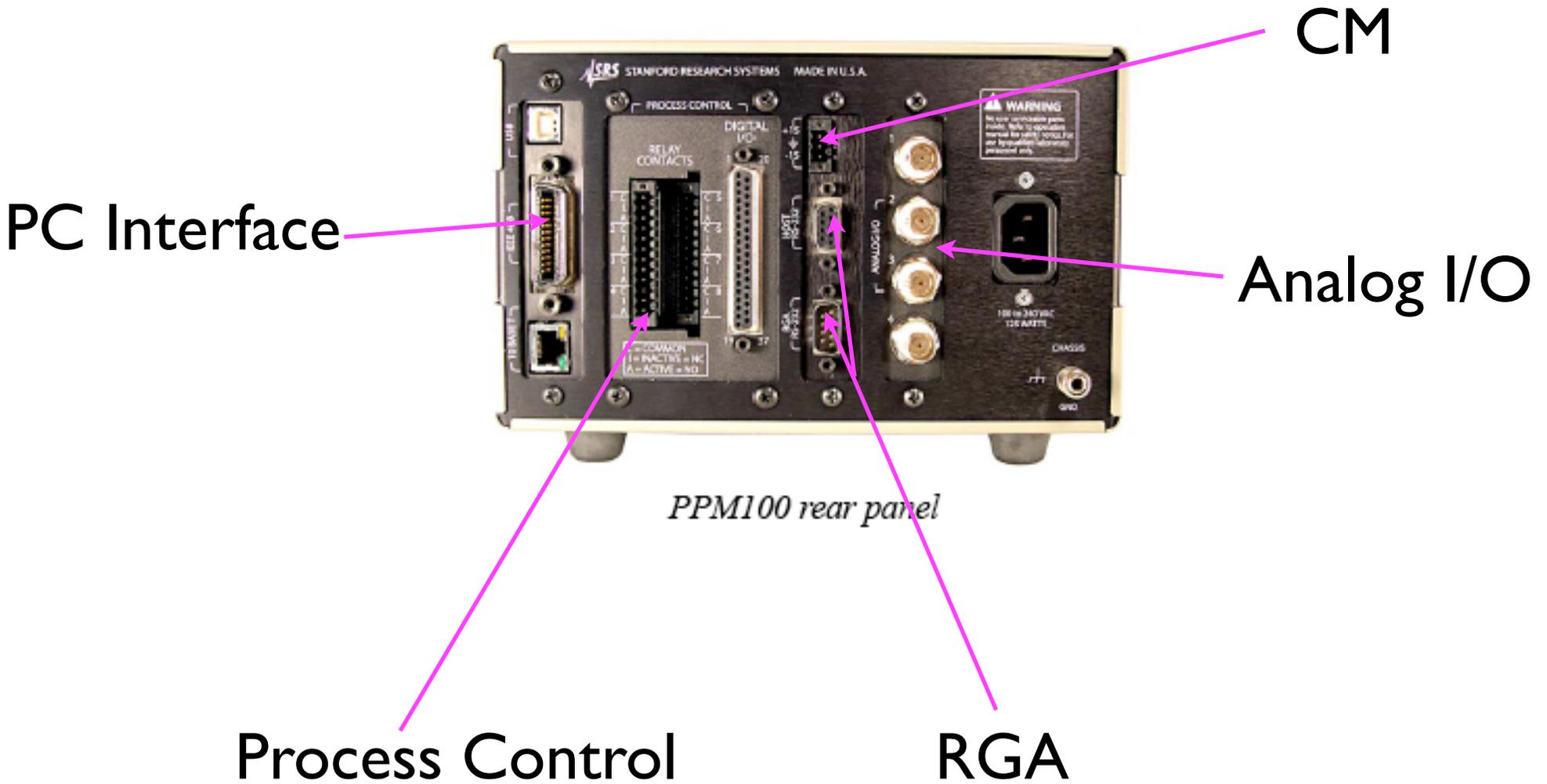
*Monitor mode*

## LCD Display



*Chart recording mode*

# Vacuum System Controller



# Quartz Crystal Microbalance (QCM200)



*QCM200 Quartz Crystal Microbalance*

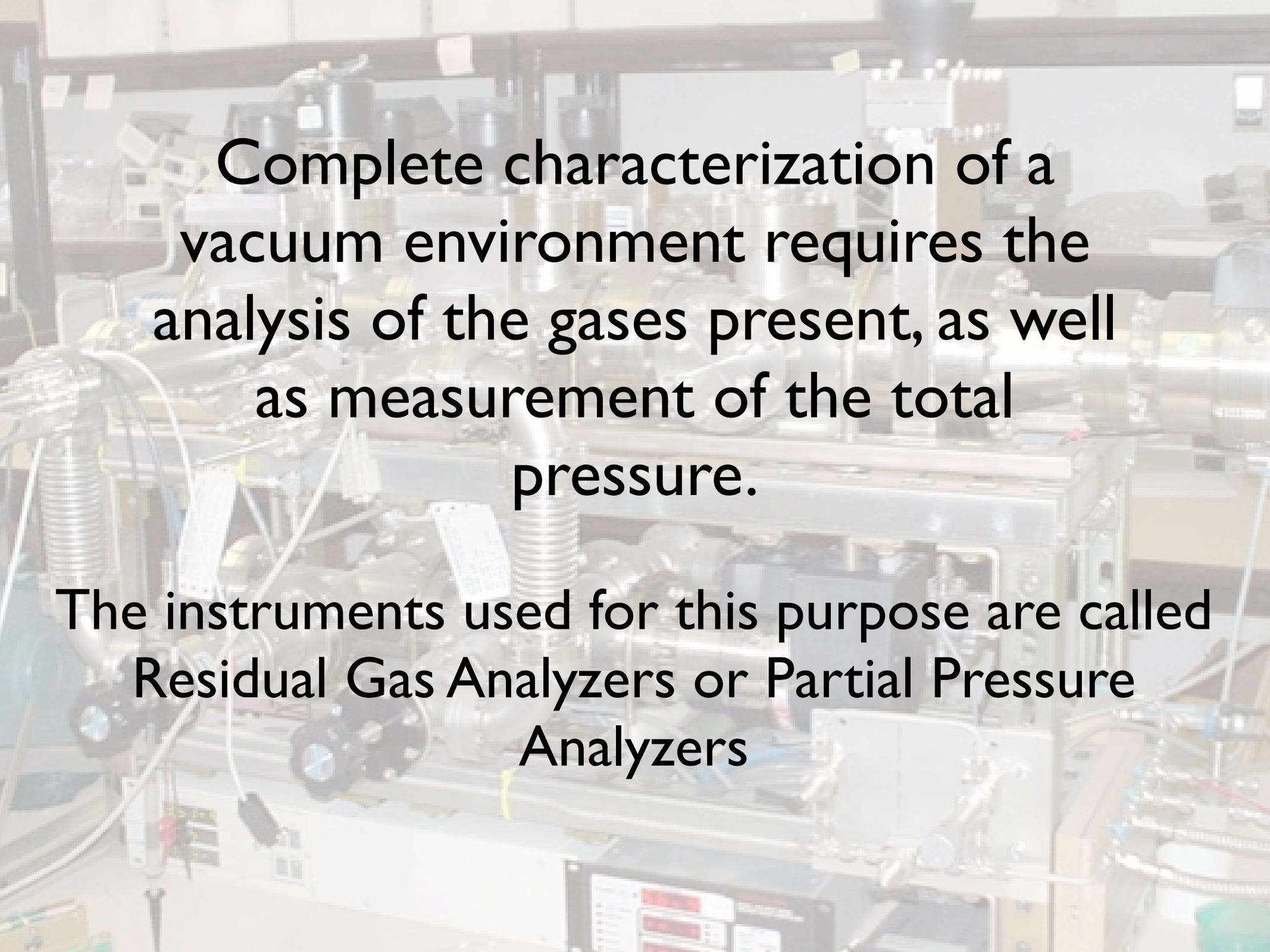
**Gas, Biological, Particulate and Electrochemical Sensor**

# Nitrogen Laser (NLI00)



- **337 nm wavelength**
- **170  $\mu$ J pulse energy**
- **Internal or external triggering to 20 Hz**
- **Replaceable laser cartridge**
- **No mirror alignment necessary**
- **TTL level sync output pulse (opt.)**
- **Fully compatible with VSL-337i OEM**

**Applications of SRS Quadrupole  
Mass Spectrometers Models  
RGA100, RGA200 and RGA300  
to Residual Gas Analysis and  
Leak Detection**



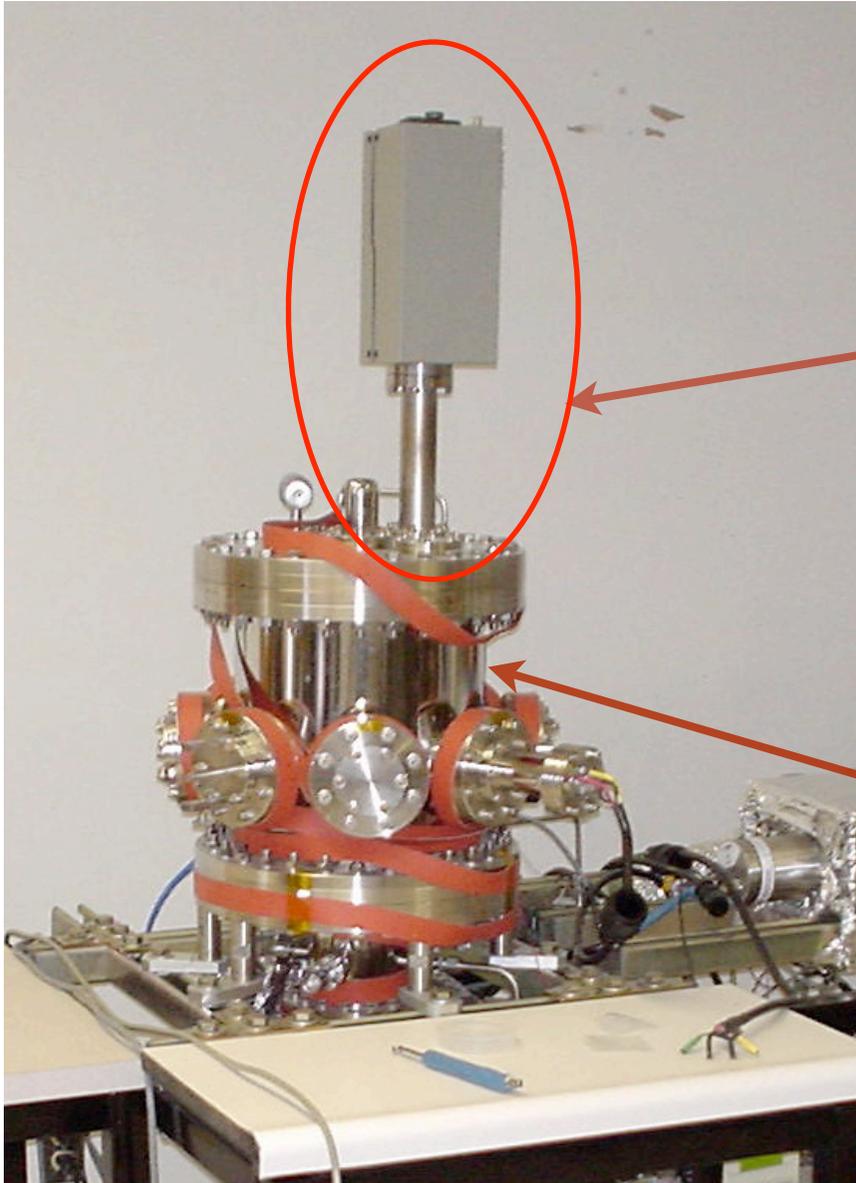
**Complete characterization of a vacuum environment requires the analysis of the gases present, as well as measurement of the total pressure.**

**The instruments used for this purpose are called Residual Gas Analyzers or Partial Pressure Analyzers**

# What is an RGA?

RGA = Residual Gas Analyzer

An RGA is a mass spectrometer of small physical dimensions that can be connected to a vacuum system and whose function is to analyze the gases inside the vacuum chamber



# How does it work?

An RGA is a low resolution mass spectrometer

A small fraction of the gas molecules are ionized (+ ions), and the resulting ions are separated, detected and measured according to their molecular masses.

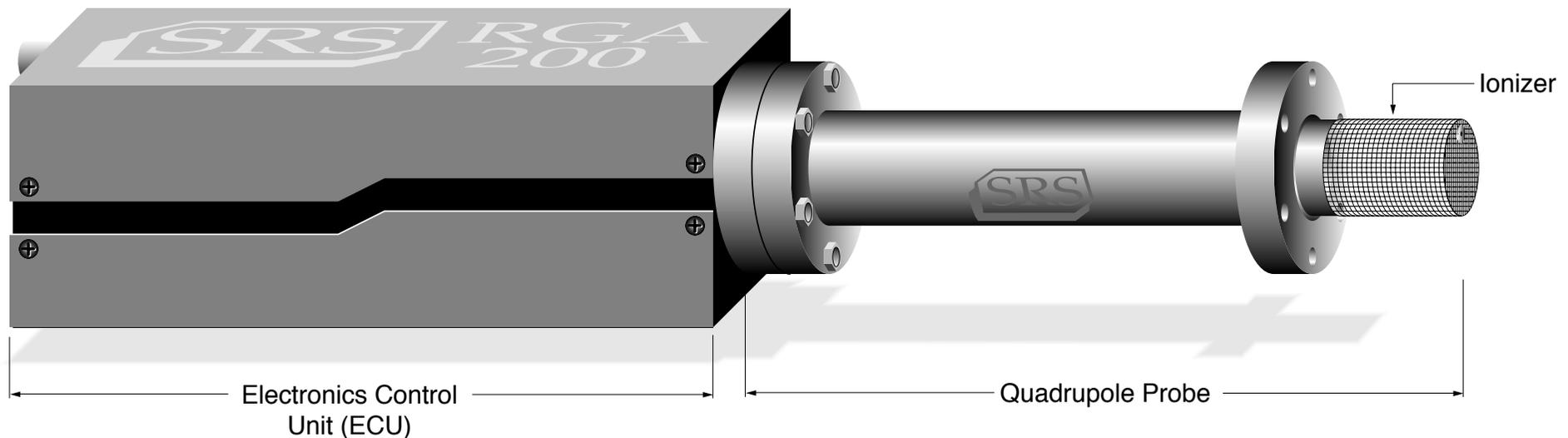
Most RGAs are quadrupoles and separate the ions based on their mass-to-charge ratios

# SRS RGA

The SRS RGA is a mass spectrometer consisting of a **quadrupole probe** and an **electronics control unit (ECU)**.

The probe mounts directly on the vacuum chamber.

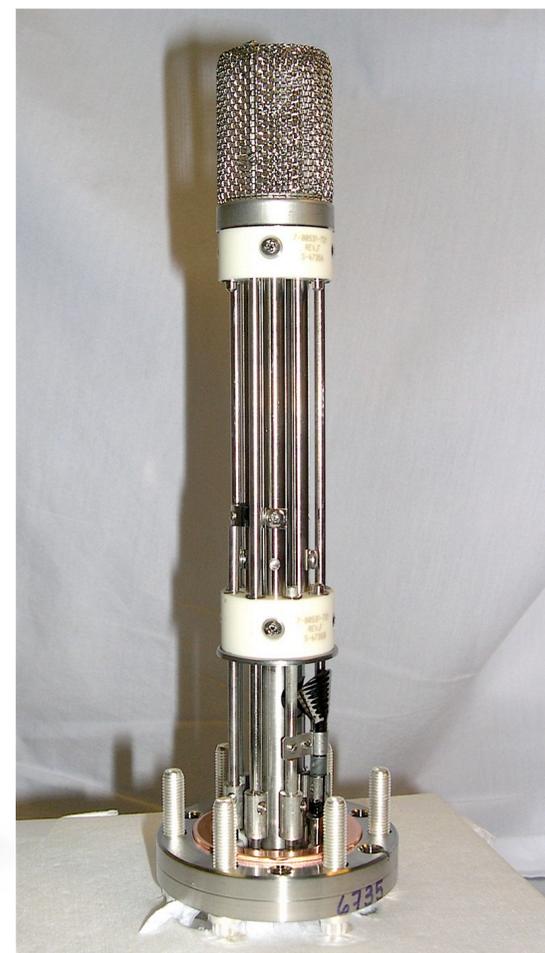
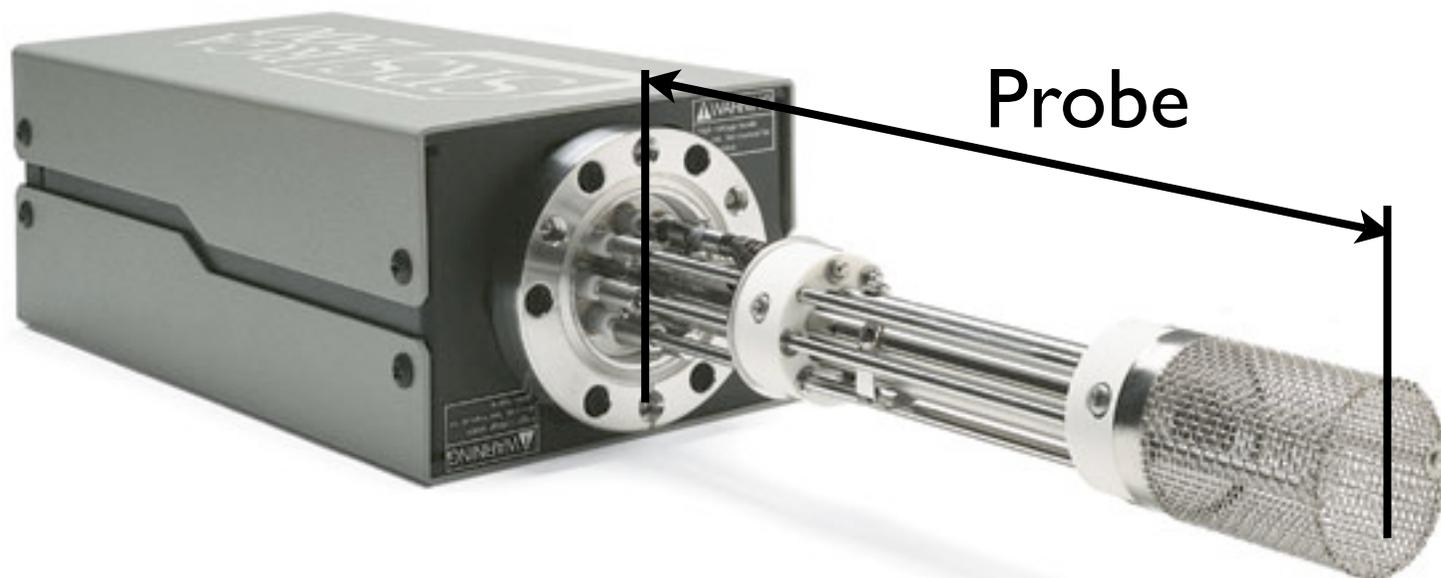
The ECU mounts on the probe and contains all the electronics required to operate the instrument.



RGA Windows completes the package

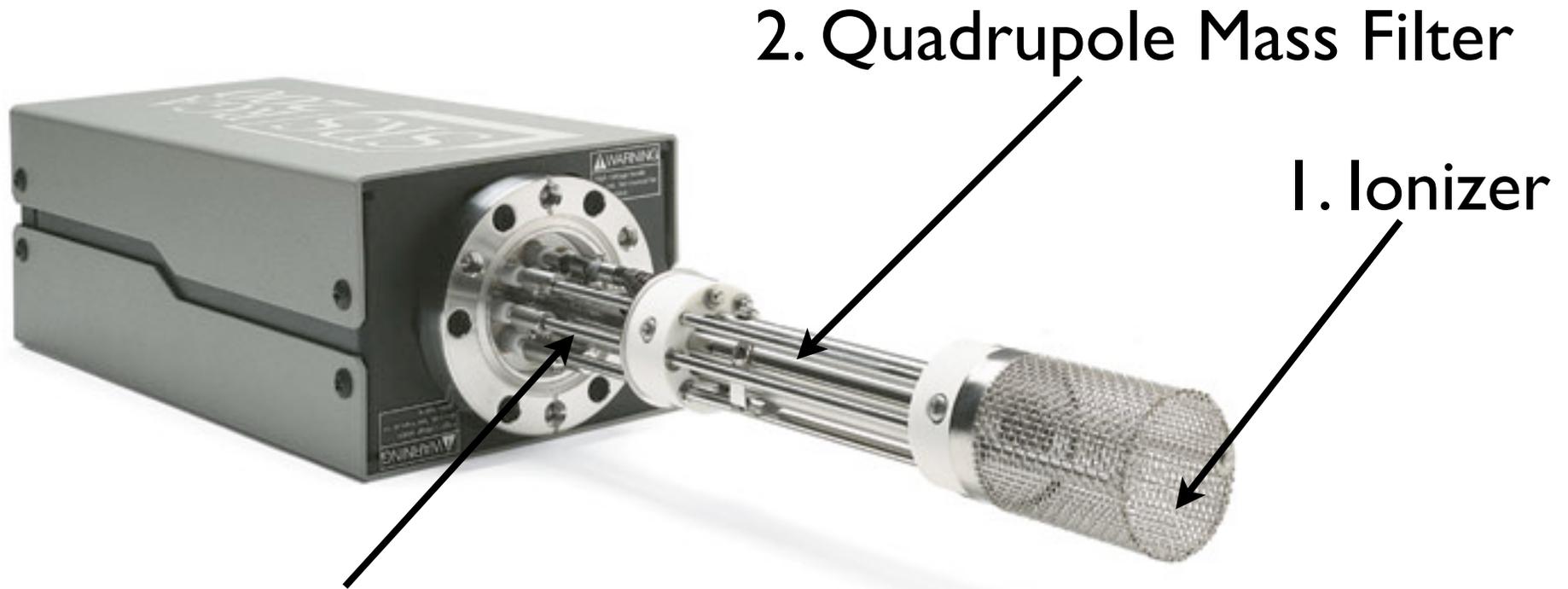
# Quadrupole Probe

The probe is the place where the gas molecules (analyte) are ionized, separated based on their mass/charge and detected and measured as an ion current



# Probe Components

The total probe equipment consists of three parts:



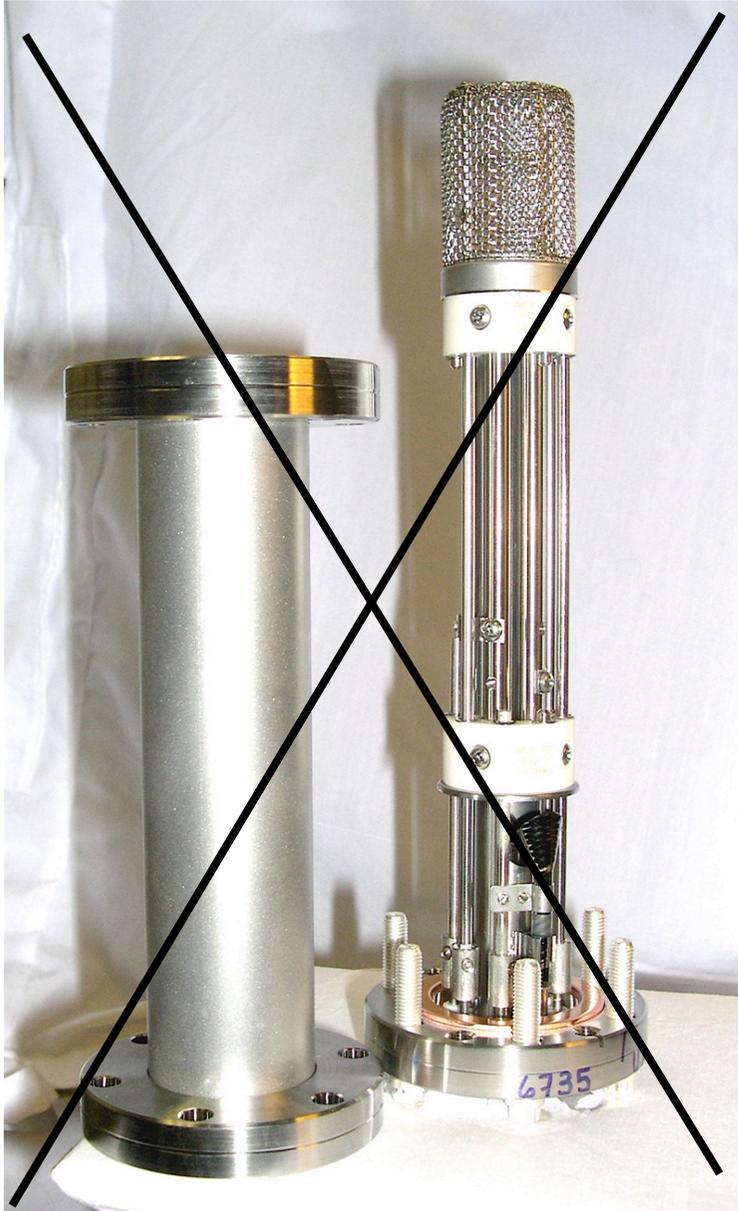
2. Quadrupole Mass Filter

1. Ionizer

3. Ion Detector

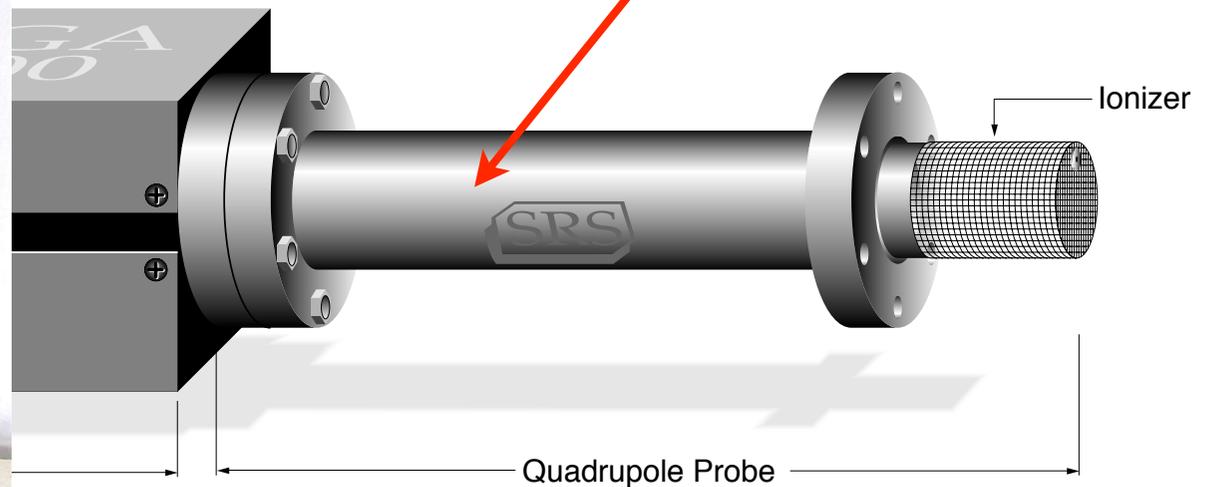
All three parts reside in the vacuum space where the gas analysis measurements are made.

# RGA Cover Nipple



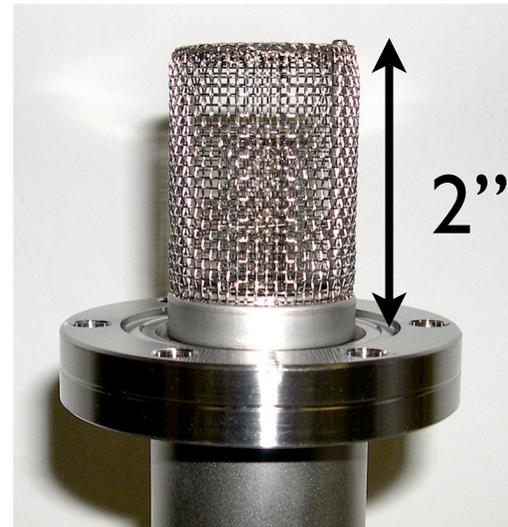
A stainless steel tube (RGA Cover Nipple) covers the probe assembly with the exception of the ionizer.

**Do not remove!**



# Insertion Volume

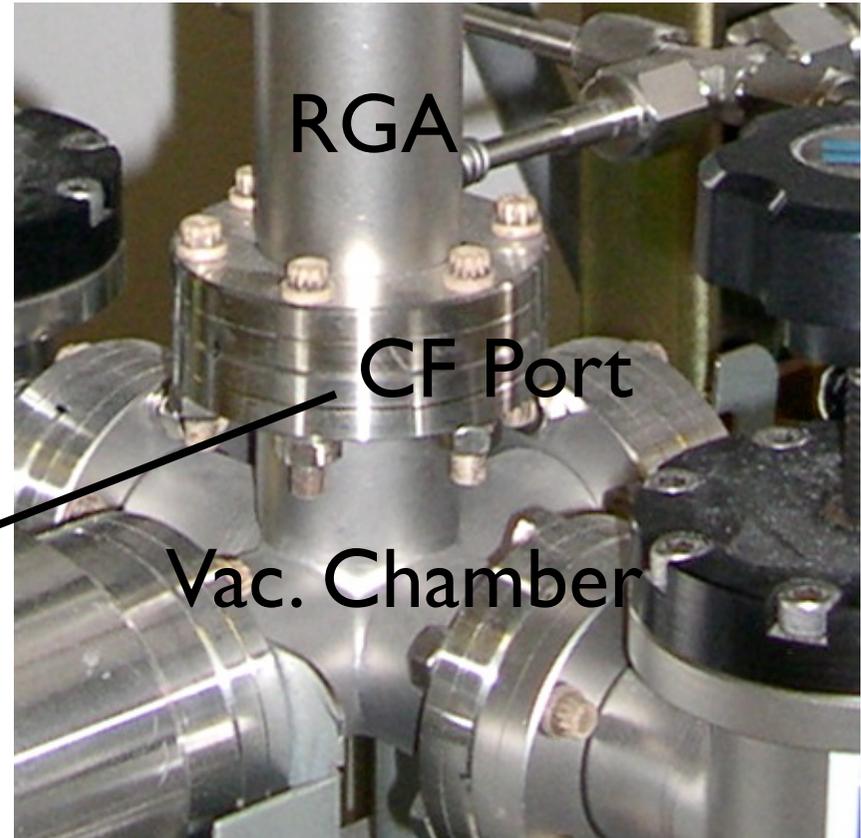
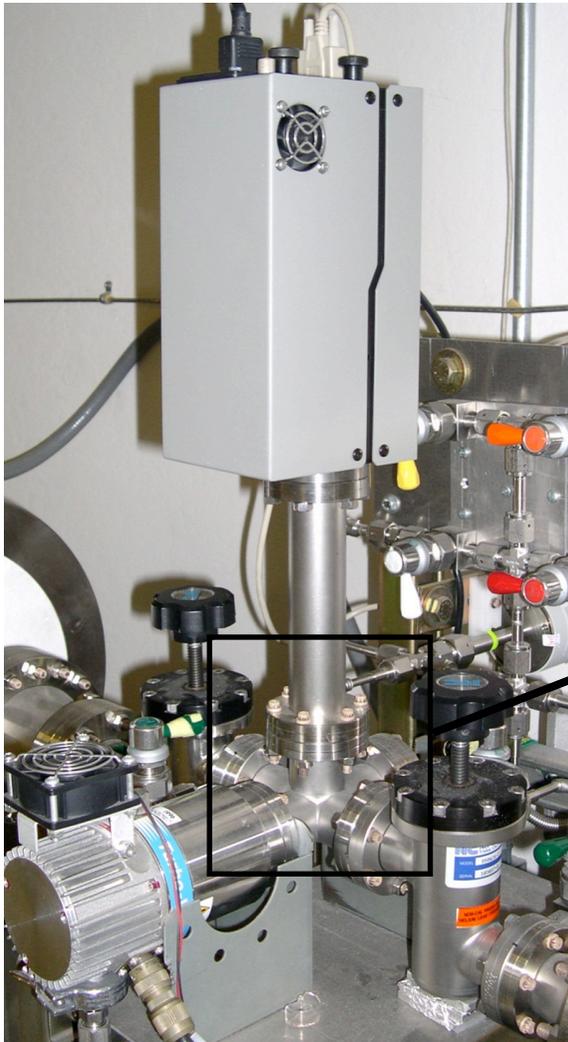
A 2.75" CF port with at least 1.375" ID and 2.5" depth is required to provide clearance for insertion of the ionizer into the vacuum system.



This insertion volume is comparable to that taken by a standard B-A ionization gauge.

# CF Mount

Attach the RGA Probe directly to your vacuum system.  
Make sure to use OFHC/Cu gaskets.



# Good Vacuum

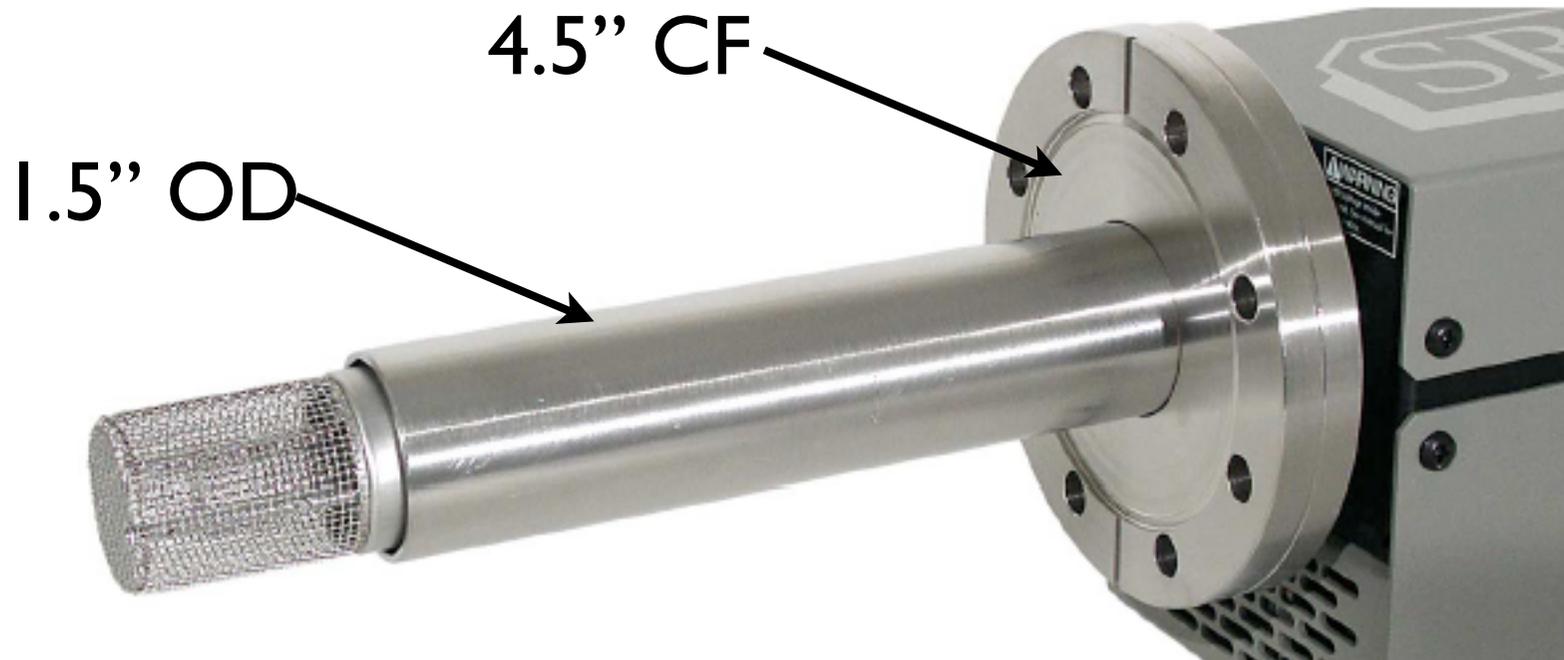
A good vacuum environment -  $10^{-4}$  Torr or less- is required for reliable operation

The vacuum range is slightly higher if you are just looking at light gases.

# Max Insertion Nipple

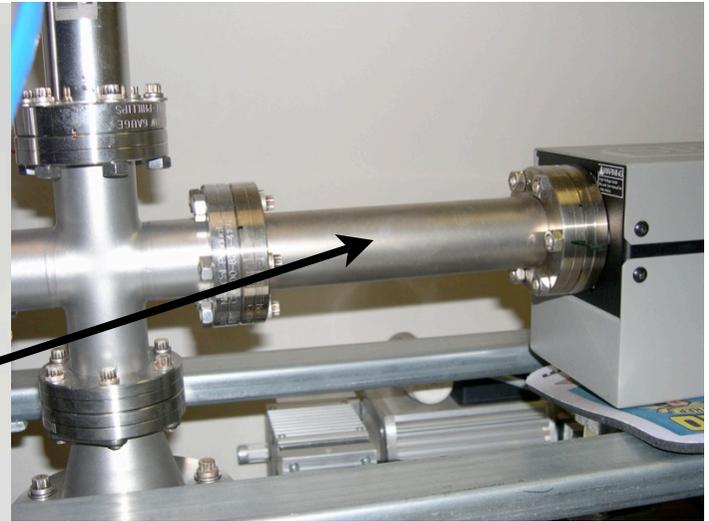
O100MAX

Use the Max Insertion Nipple if you need to bring the ionizer into the chamber.



# Heating Jacket

O100HJR  
200 Celsius



Remove ECU  
during  
bakeouts!

# Probe Bakeout

Bakeout is recommended:

- After installation into the vacuum chamber
- After exposure to air
- When background contamination is evident in the spectra
- When performance degradation due to contamination is evident.

# Bakeout Procedure

- Turn off the RGA and disconnect the ECU
- Attach the heating jacket (O100HJC)
- Make sure the pressure is below  $10^{-5}$  Torr
- Establish a slow flow of dry N<sub>2</sub> gas if desired (scrubber).
- Bake the probe for at least 12 hours (i.e. overnight)
- Wait for the unit to cool down
- Mount the ECU back on the probe
- Run analog spectra to check the integrity of the spectrometer and measure contamination levels.

# Ionizer



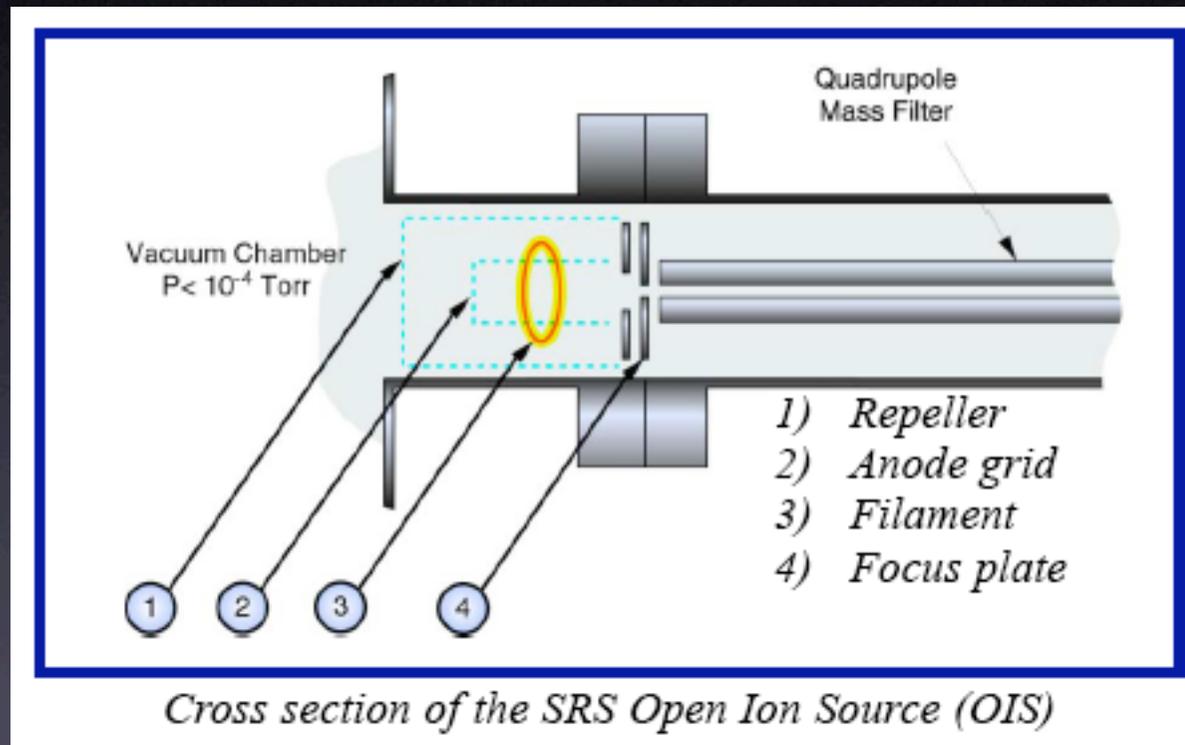
## Open Ion Source

The pressure at the ionizer is the same as in the rest of the analyzer.

Residual gas molecules flow freely through the grid assembly

# Ionizer

## Cross-section View



# Open Ion Source

- Wire Mesh Construction
- Cylindrical symmetry
- Coaxial with quadrupole filter



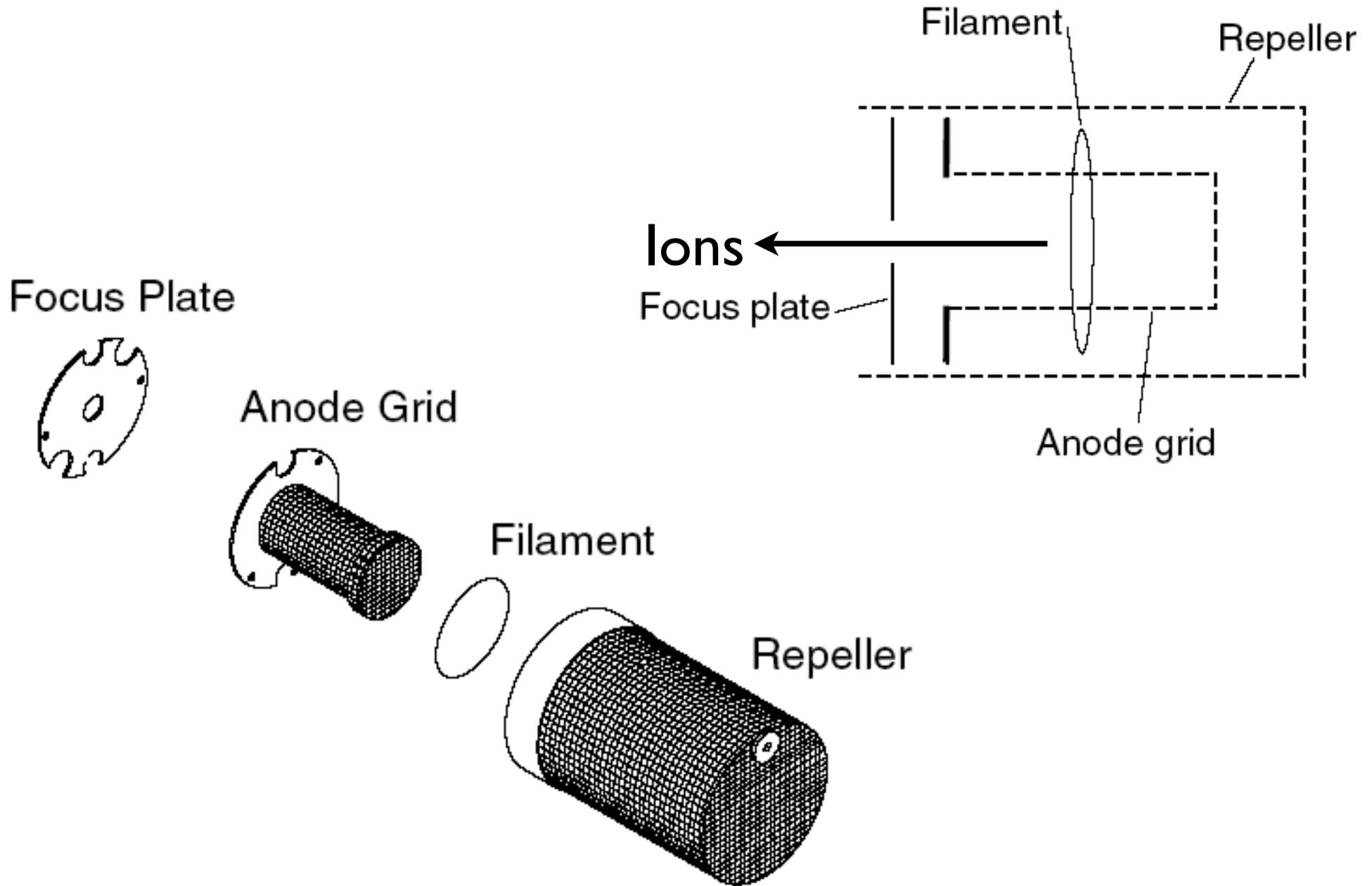
SS 304 Construction

Self aligning

Low outgassing

Field Replaceable  
(O I 00RI)

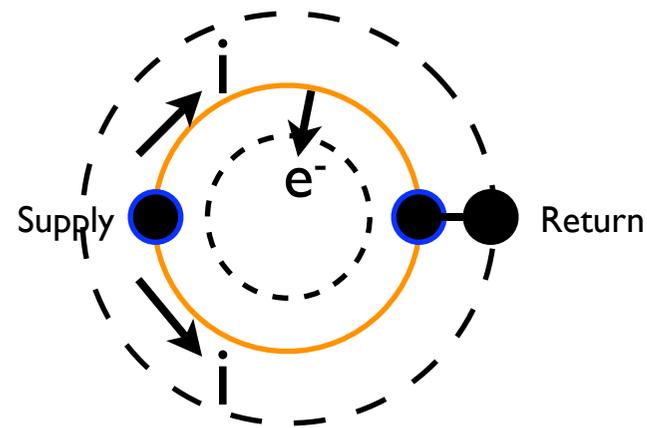
# Ionizer Parts



# Filament

The filament is the source of the electrons used to ionize the gas molecules

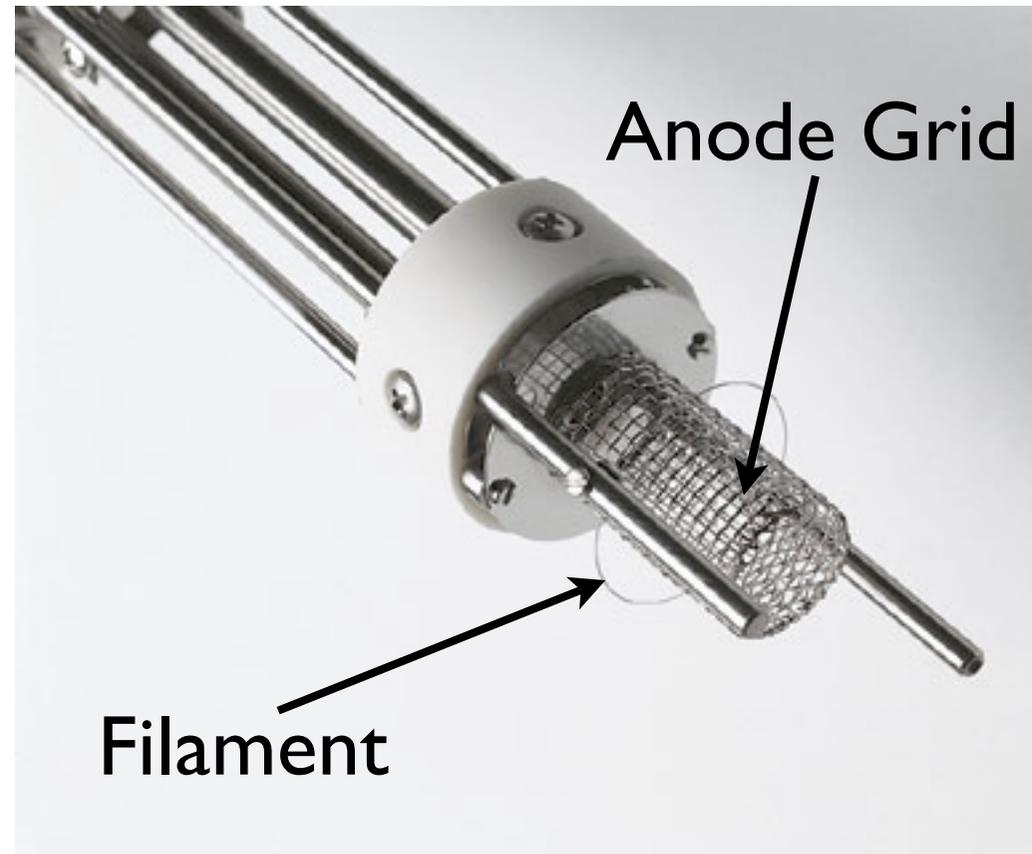
- Circular design
- $\text{ThO}_2\cdot\text{Ir}$  material
- Negative potential
- Resistive heating
- Closed-loop on emission current
- Dual filament operation
- Filament protection



# Anode Grid

The thermionically emitted electrons are accelerated towards the anode grid, which is positively charged relative to the filament.

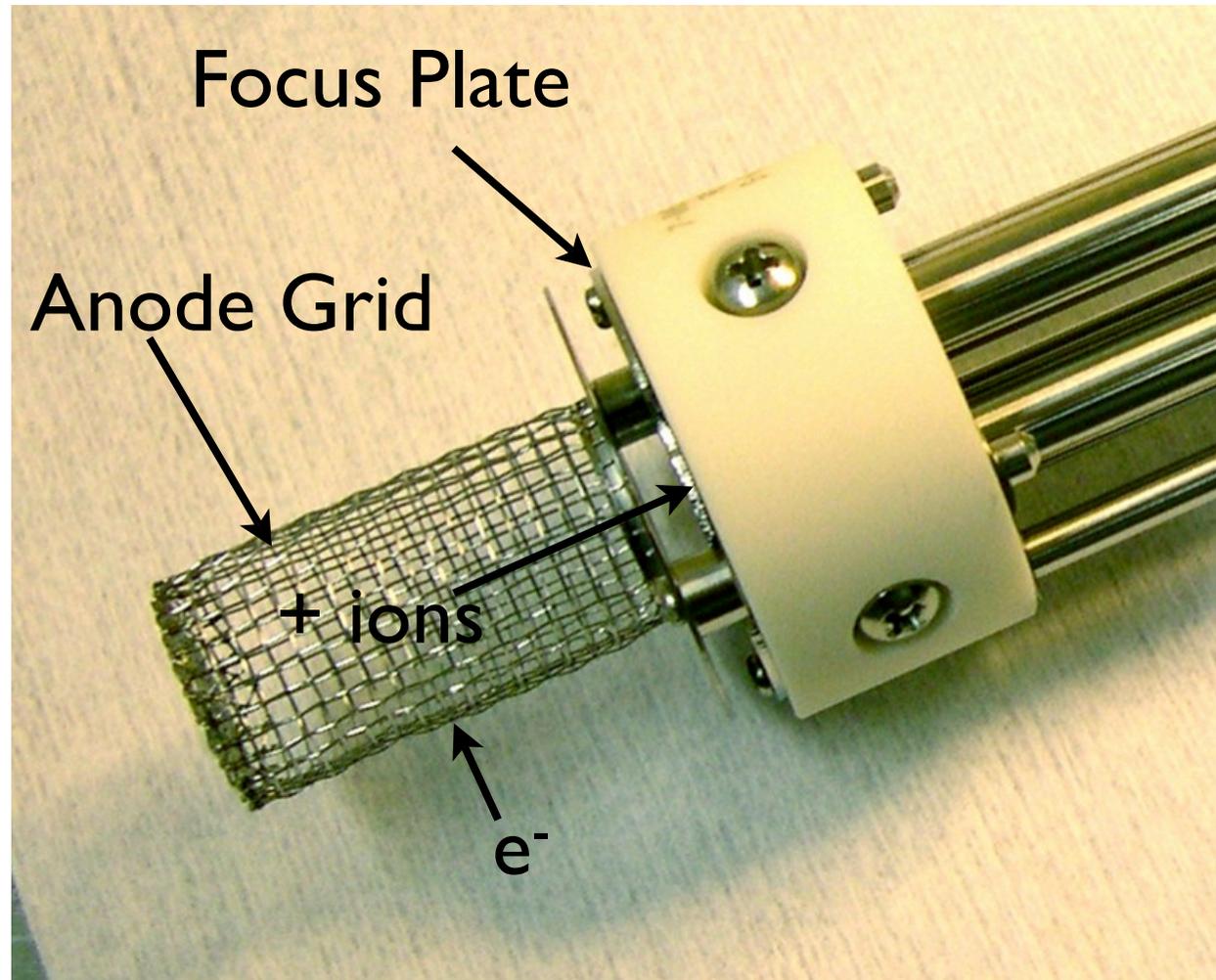
Because of the open design of the anode grid cage, most electrons do not strike the anode immediately, but pass through the cage where they create ions through electron impact ionization (EII)



# Focus Plate

The ions produced within the anode grid volume, are extracted from the ionizer by the electric field produced by the difference in voltage bias between the anode grid and the focus plate.

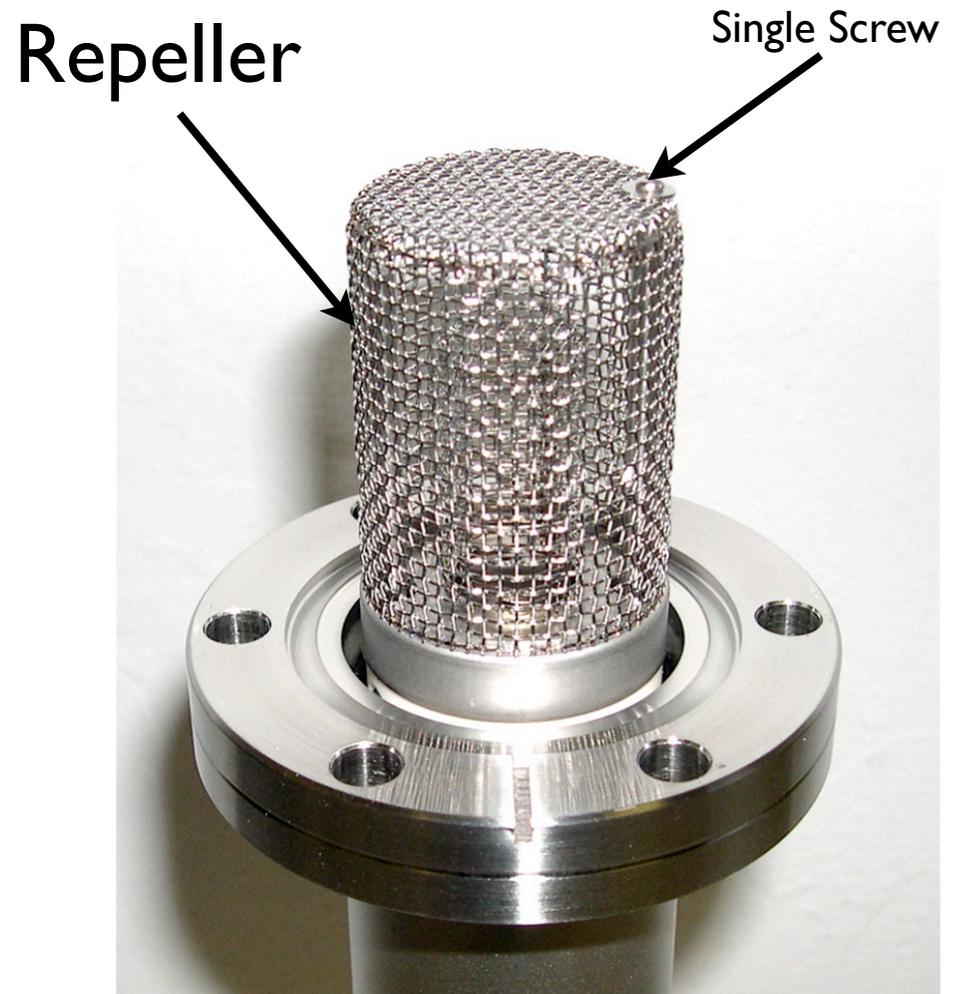
The focus plate is kept at a negative potential and its function is to draw the ions out of the anode cage and focus them into the filter section.



# Repeller Cage

The repeller cage, which completely encloses the ionizer, is biased negative relative to the filament and prevents the leakage of electrons out of the ion source.

It also provides mechanical protection for the delicate filament



# Electron Impact Ionization

## Basic Requirements

- Maximum pressure  $< 10^{-4}$  Torr
- Linearity  $< 10^{-5}$  Torr
- Adjustable bias voltages and electron emission current

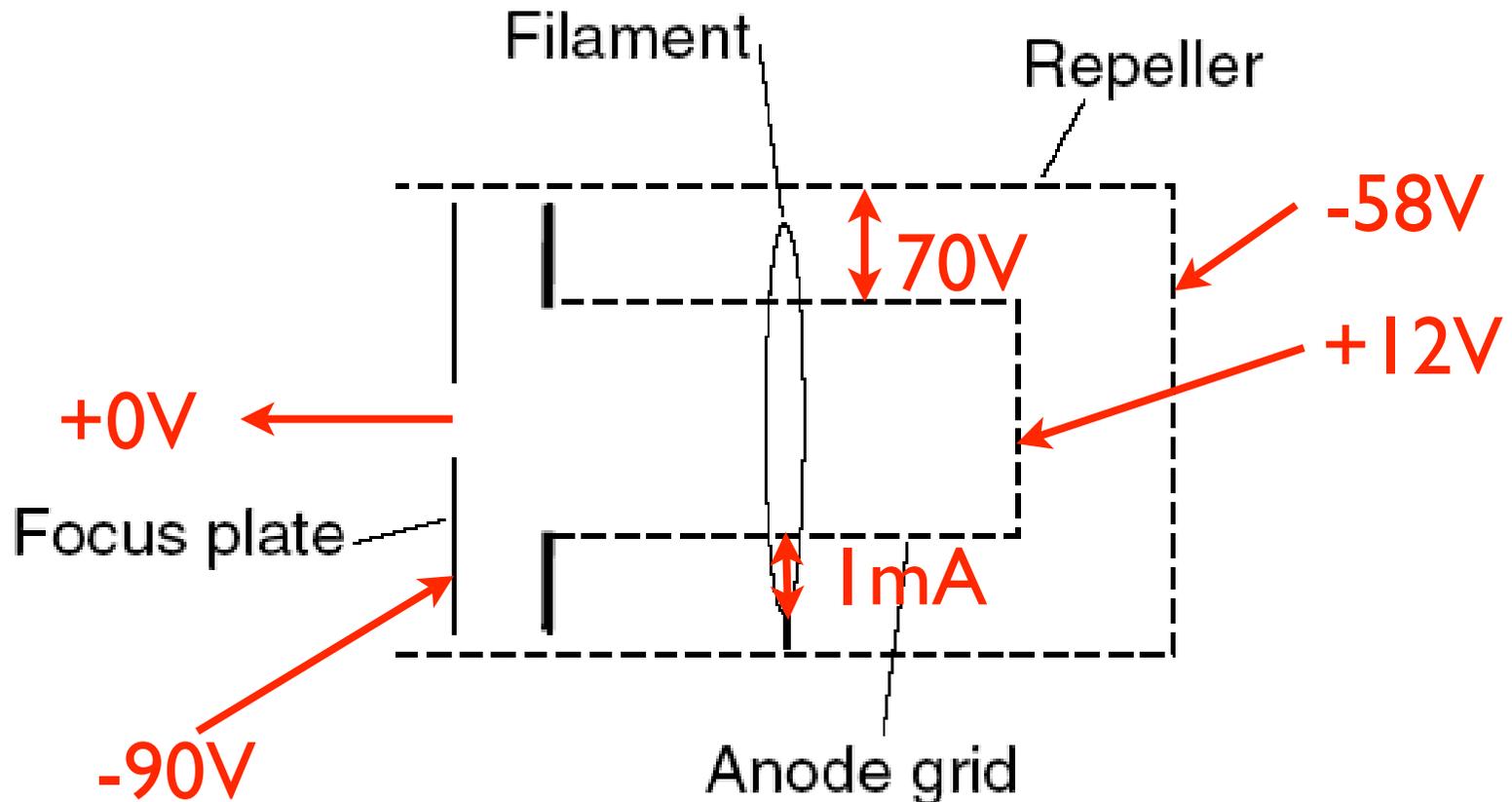
# Ionizer Settings

RGA Windows provides direct access to all ionizer parameters.

	Units	Default	Range	Min. Incr.
Electron Energy	eV	70	25 to 105	1
Emission Current	mA	1.00	0 to 3.5 mA	.02
Ion Energy	eV	12	8 (low) or 12 (high)	NA
Focus Voltage	V	-90	0 to -150	1

# Default Settings

High ion energy (12eV), 1mA emission, 70eV electrons, -90V focus voltage



# Electron Energy

- Voltage difference between the filament return bias and anode grid bias.
- 25 to 105eV, 70eV default.
- Use low energies to reduce multi-ionization, fragmentation and process interference.
- Use higher energies to improve signal/noise
- Use external voltage sources to lower the energy further

# Ion Energy

- Kinetic energy of the ions as they fly through the quadrupole mass filter
- Bias voltage of the anode grid
- Limits the ultimate resolution of the filter (0.3 amu [low], 0.5 amu [high])
- Use High energy for higher signal/noise
- Use low energy for higher resolution

# Focus Voltage

- Range: 0 to -150V, default: -90V
- Adjust to optimize S/N and to eliminate negative background at low masses.
- For Faraday Cup, set 30V more negative than the repeller to eliminate electron leakage into the quadrupole.

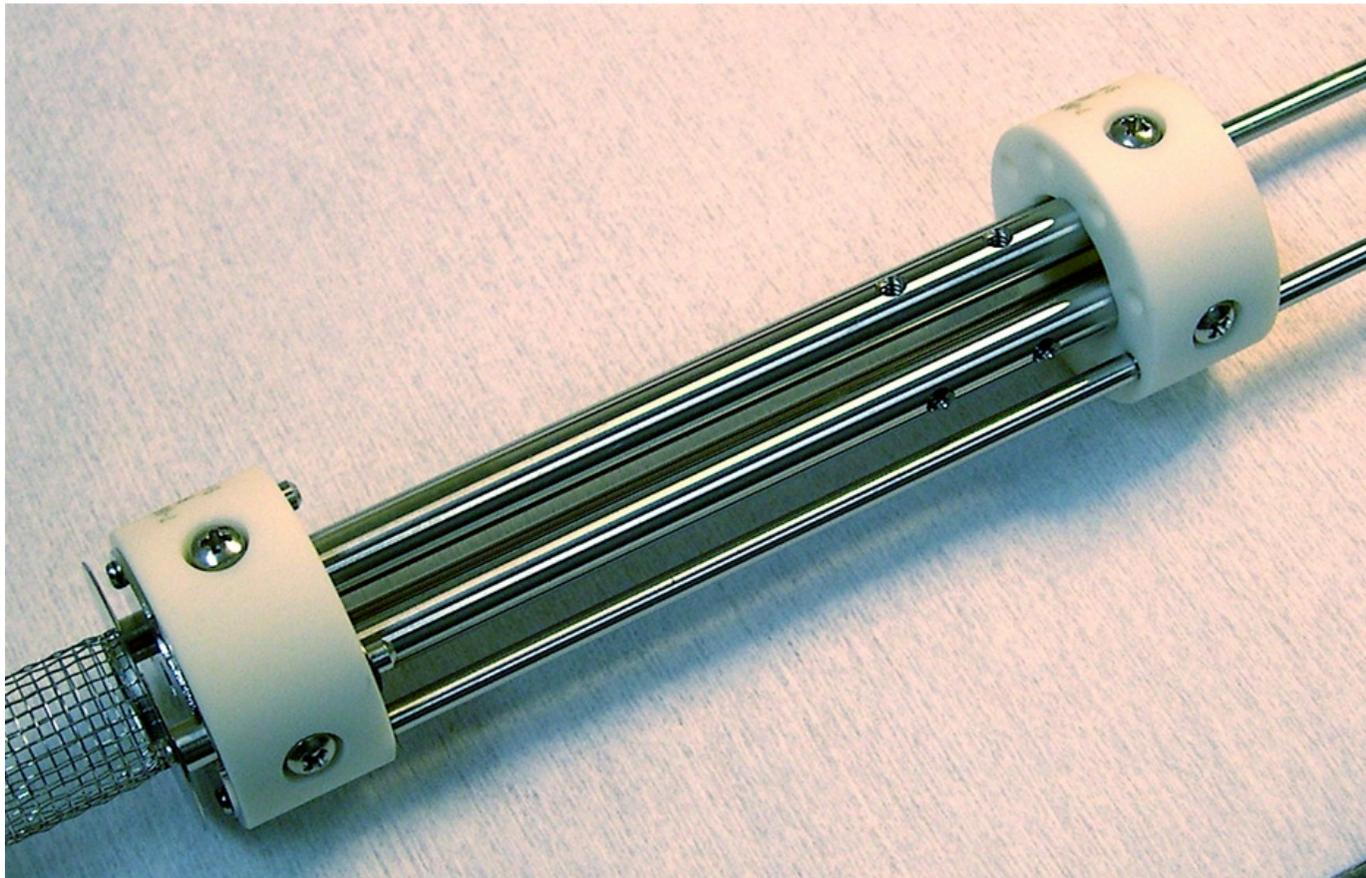
# Electron Emission Current

- Electron current from the filament to the anode grid. Controlled by feedback loop.
- Ion signal directly proportional to electron emission current
- Range: 0 to 3.5 mA, default: 1 mA.
- Use higher values for higher S/N.
- Use lower values for improved pressure range, increased linearity and reduced outgassing and ESD.

# Ionizer Degas

- An ionizer Degas program is built into the RGA Head to clean up the filament and the ionizer by Electron Impact Desorption
- Degassing is a fast way to clean the ionizer but compromises the lifetime of the filament.
- Whenever possible substitute a degas procedure with a proper bakeout
- Let the RGA cool down for at least 10 minutes after a degas
- Try not to use it!

# Quadrupole Mass Filter



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

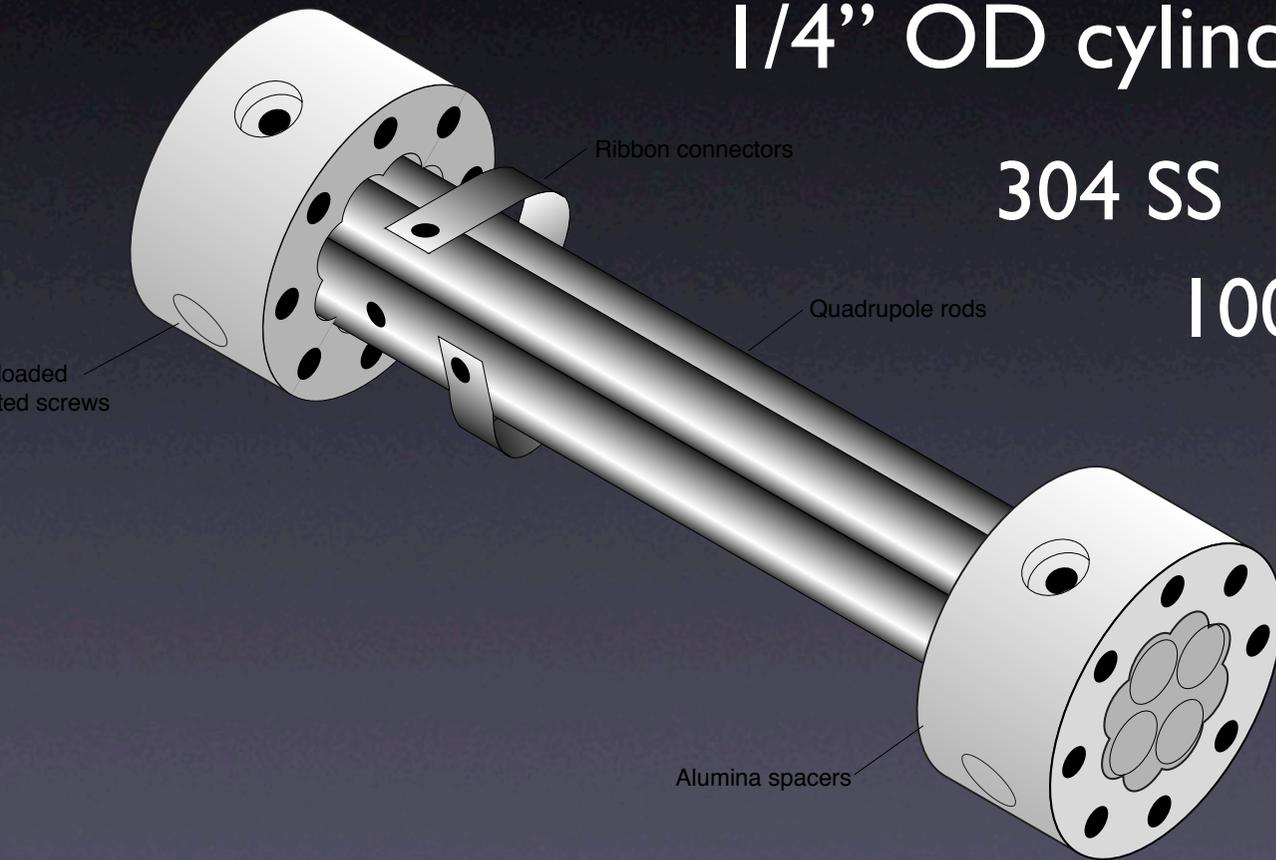
# Mass Filter

1/4" OD cylindrical rods

304 SS

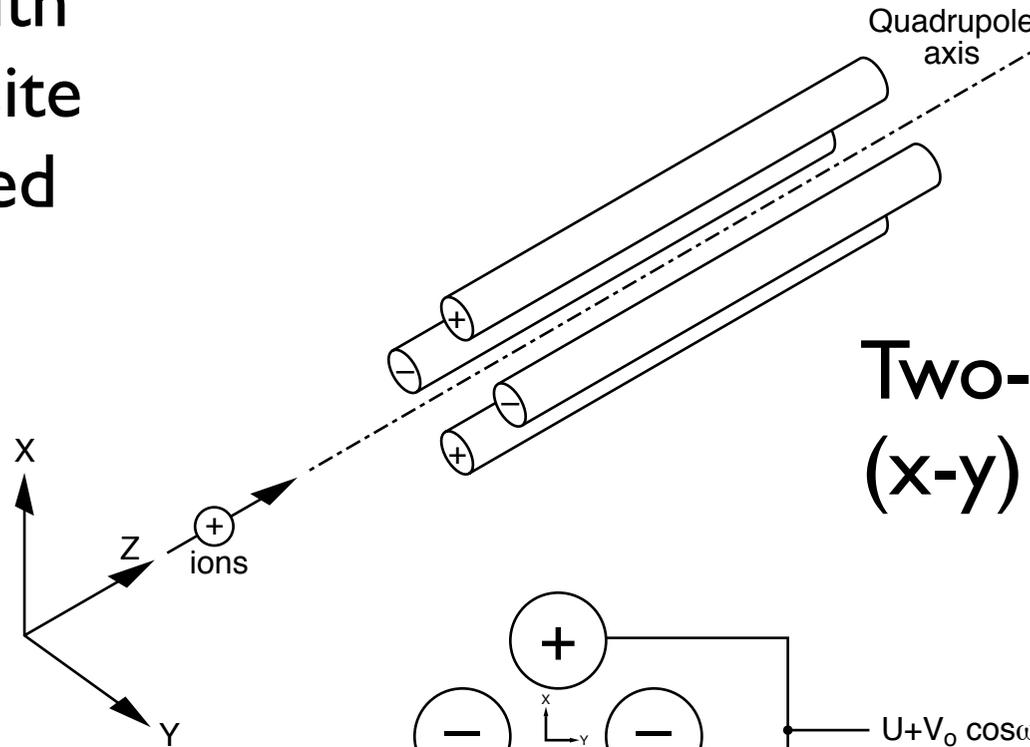
100 micron tolerance

4.5" long

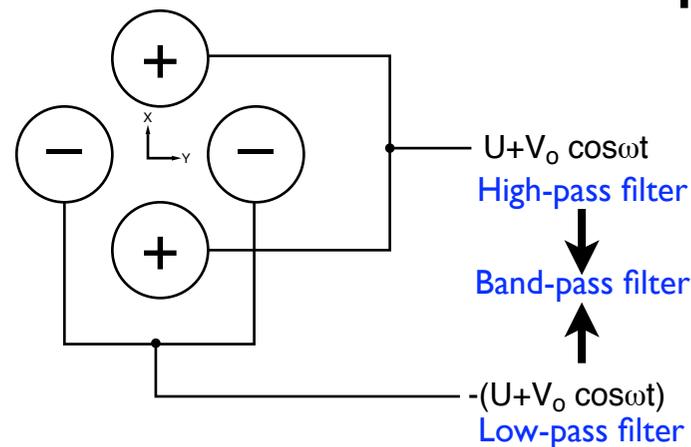


# QMF-Principle of operation

Four rods in perfect quadrature, with the two opposite rods connected together electrically.



Two-dimensional (x-y) quadrupole field.



Ions enter along the z-direction and start oscillating in the X & Y directions.

The ions are separated based on their mass-to-charge ratio by lateral forces resulting from the dynamic electrical potentials applied to the rods.

**A combination of DC and RF voltages control the quadrupole filter operation**

Successful isolation of a specific mass species requires setting the RF/DC such that only the ions of interest have stable trajectories down the rod assembly.

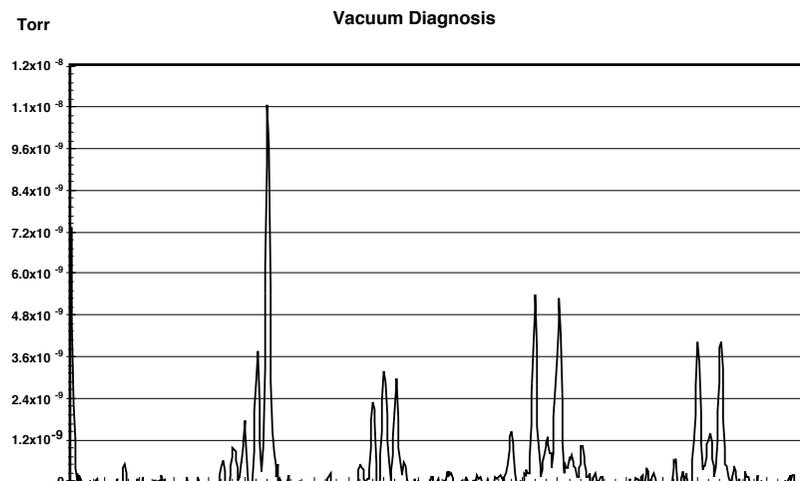
# Rule-of-Thumb

RF amplitude sets the mass

DC/RF sets the resolution

## Mass Spectra

By simultaneously varying the amplitude of the DC and RF voltages, while recording the resulting ion currents, an entire mass spectrum can be scanned.



# Figures of Merit

# Mass Range

Range of masses defined by the lightest and heaviest single charged ions which can be detected by the mass spectrometer

RGA100: 1-100 amu

RGA200: 1-200 amu

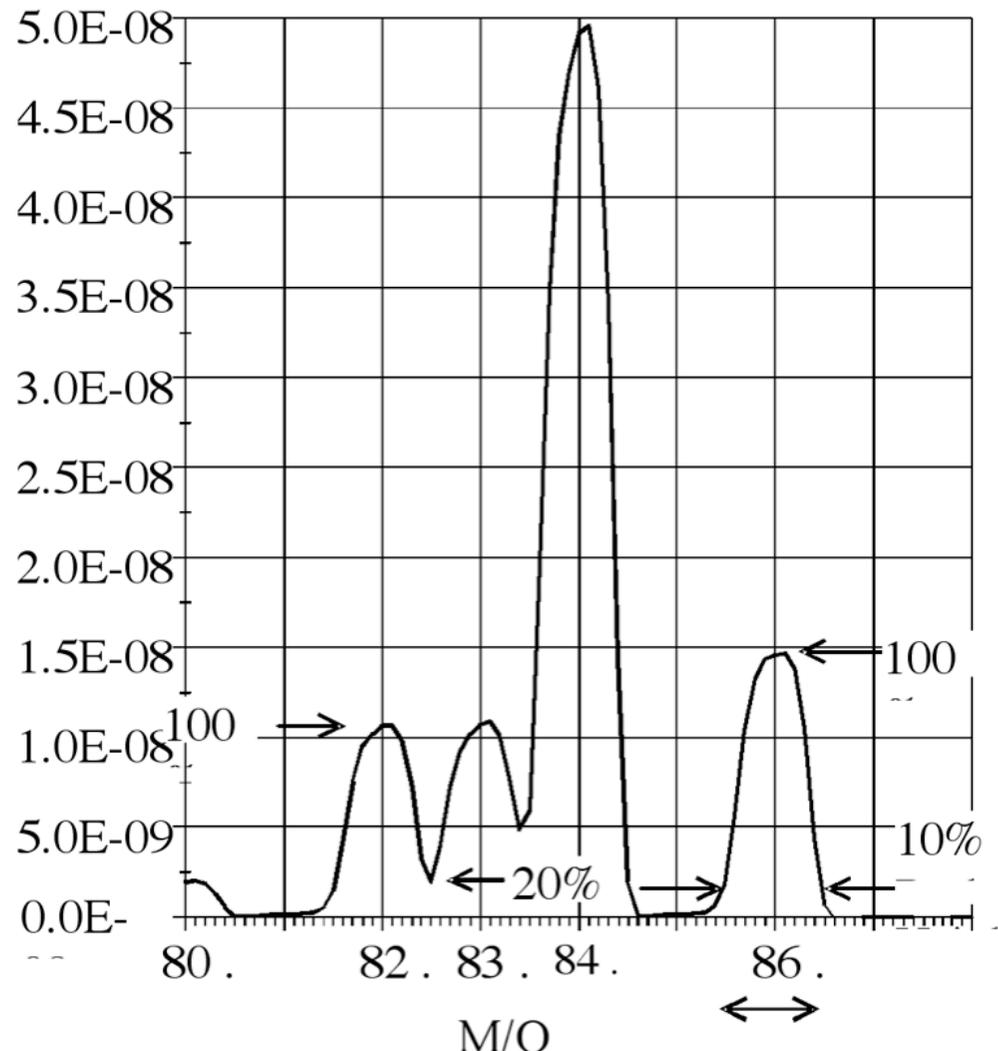
RGA300: 1-300 amu

SRS RGA uses the same probe design and same RF frequency for the three ranges

# Resolution (absolute)

$$\Delta M_{10\%} = 1 \text{ amu typ.}$$

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.



# Resolving Power

$$R = M/\Delta M_{10\%}$$

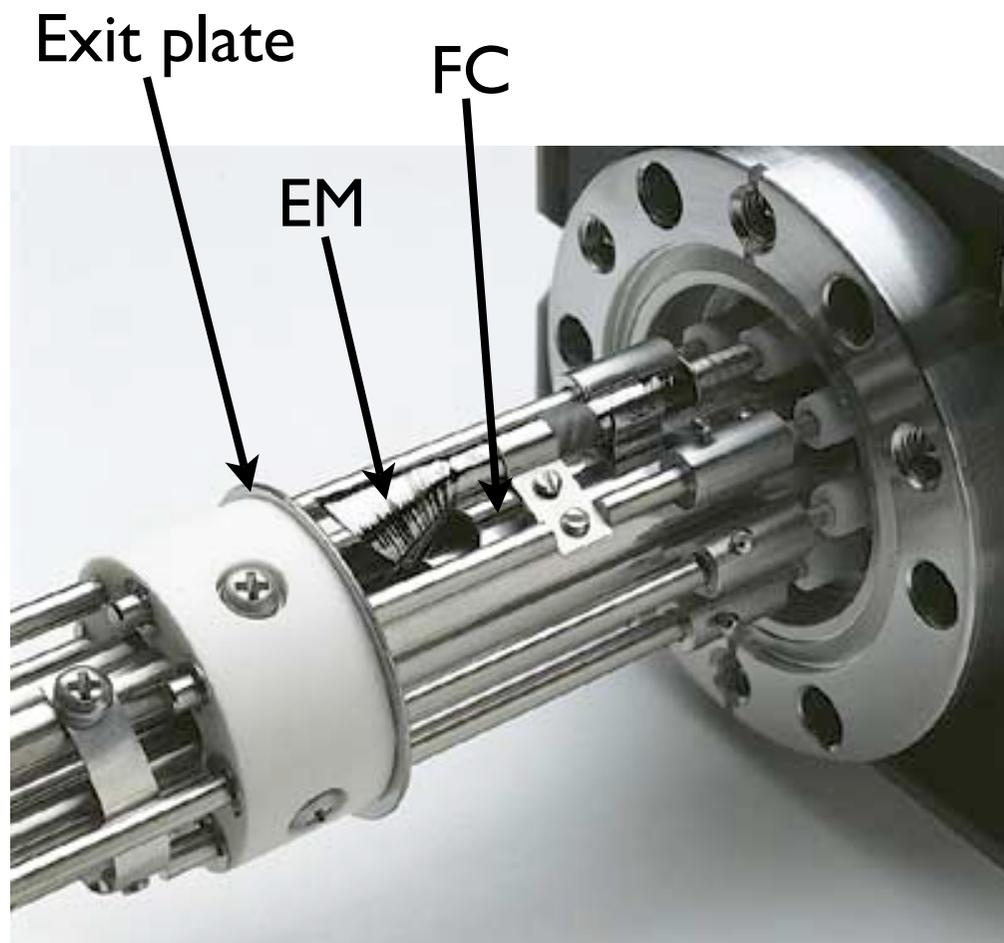
Ratio between a particular mass  $M$ ,  
and the absolute resolution  $\Delta M_{10\%}$  at  
that mass

Sensitivity (throughput) decreases at 1 to 1.5  
times the rate of resolving power increase.

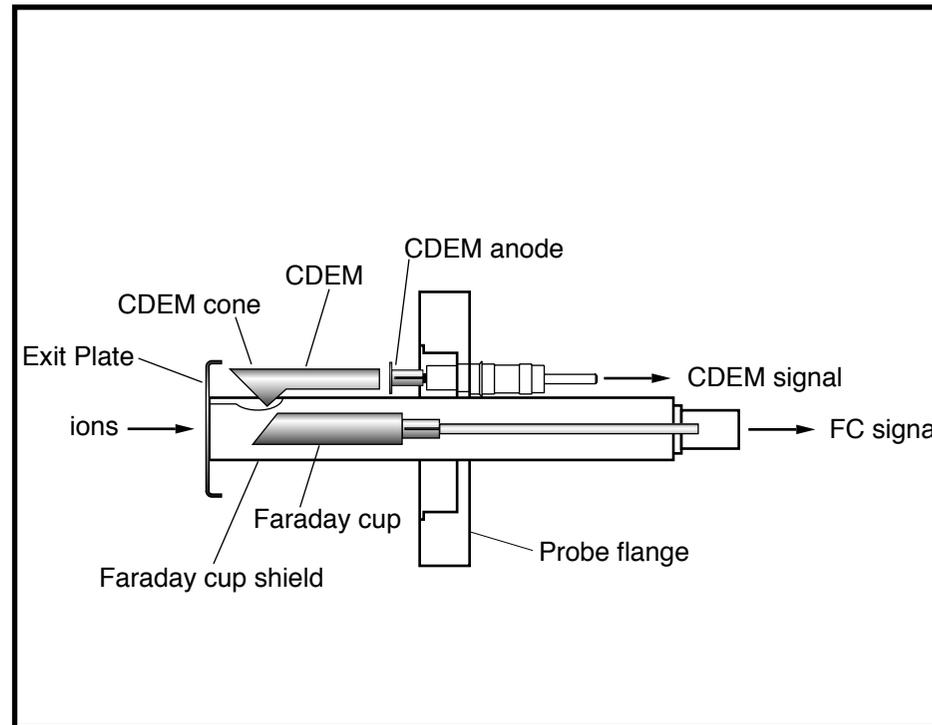
The throughput of the analyzer decreases with  
mass.

# 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.



# Detector Assembly



Detection Limits:

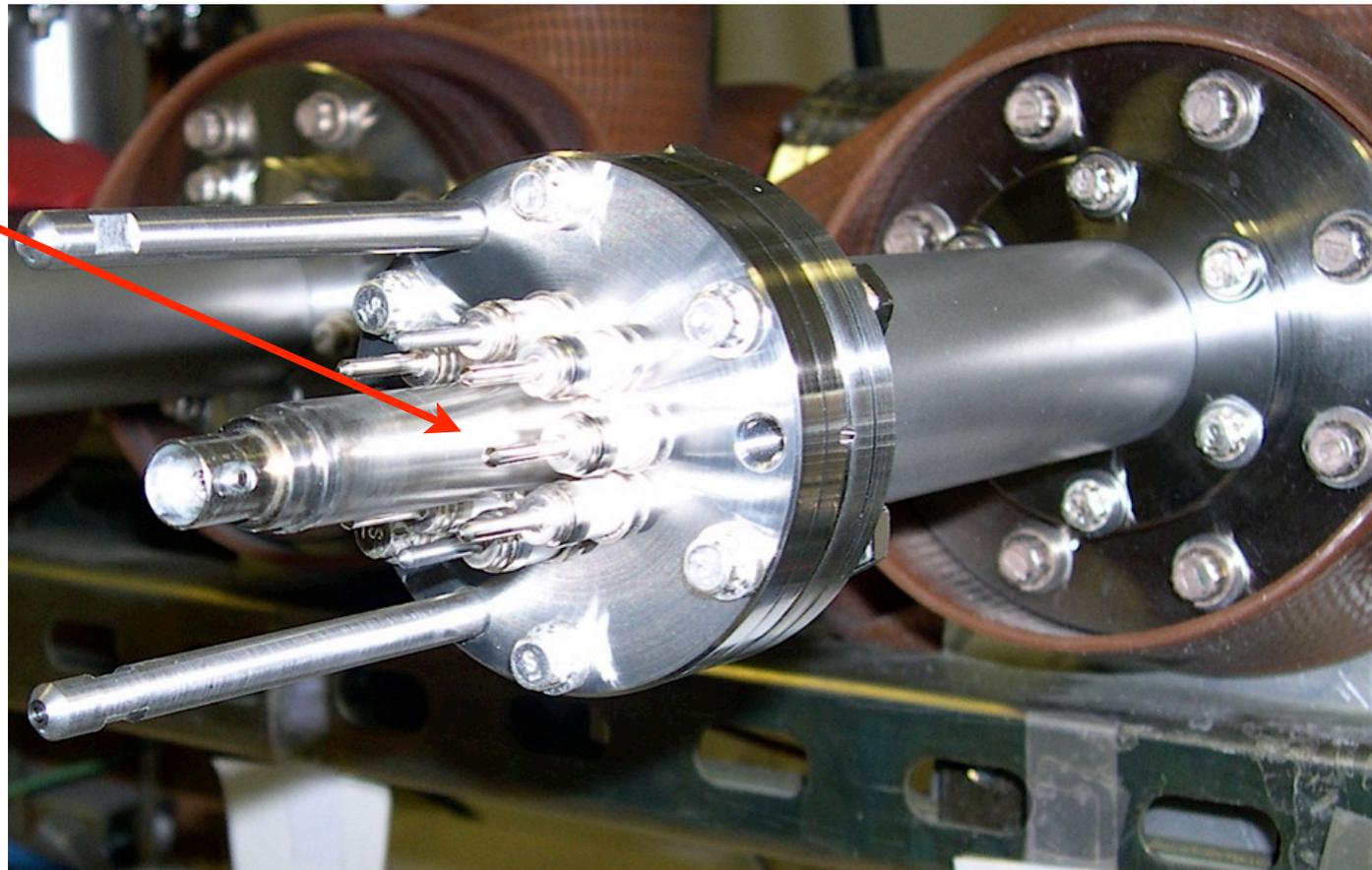
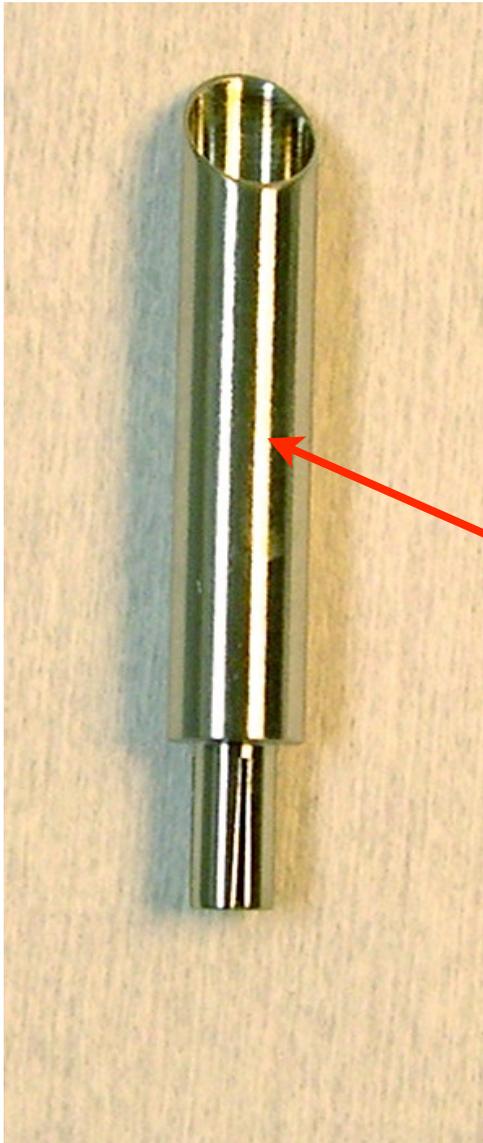
(typ.)

FC:  $10^{-10}$  Torr

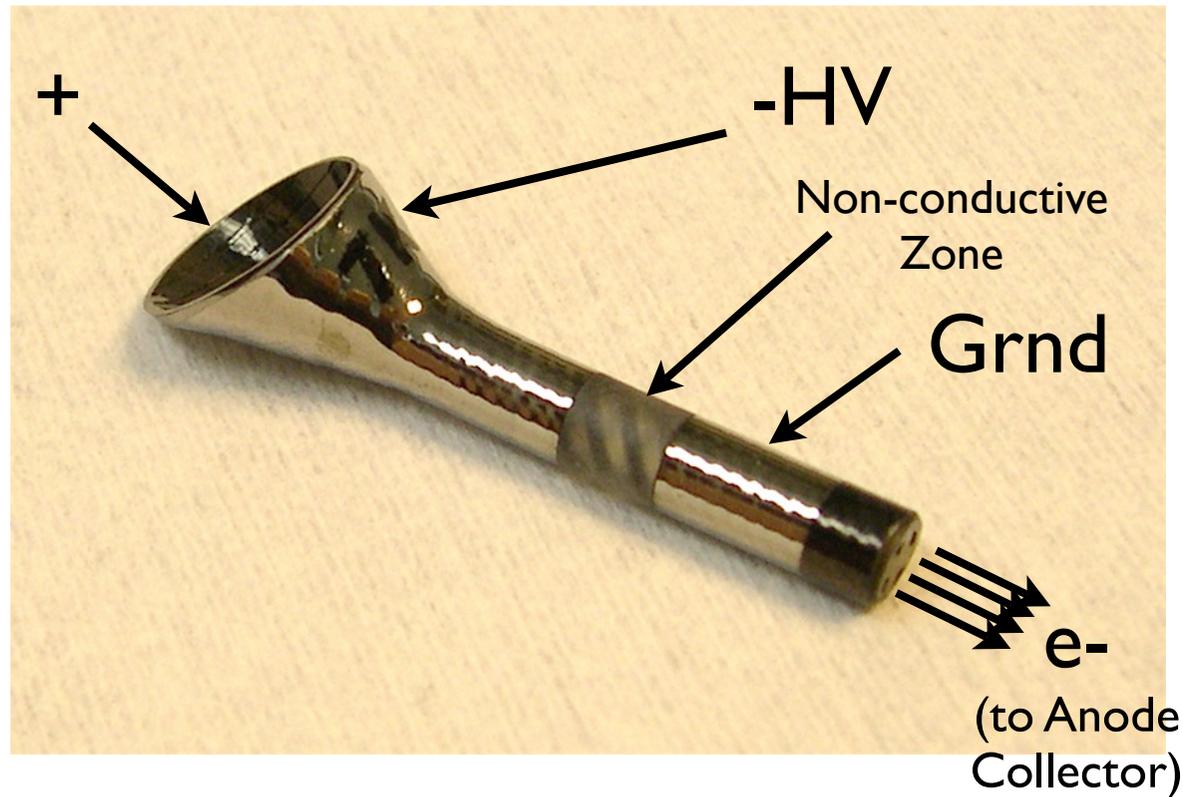
EM:  $10^{-13}$  Torr

# Faraday Cup

Small stainless steel 304 metal bucket located on-axis at the end of the quadrupole. Grounded bias.



# Electron Multiplier



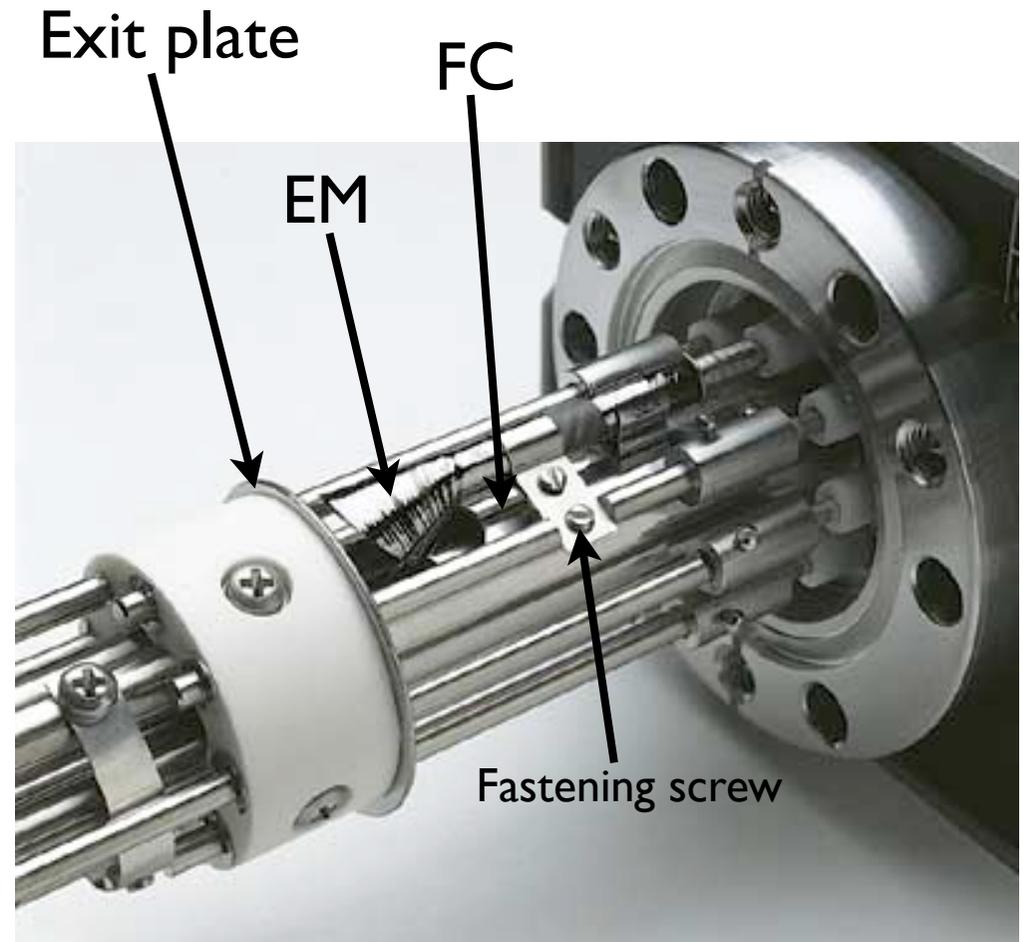
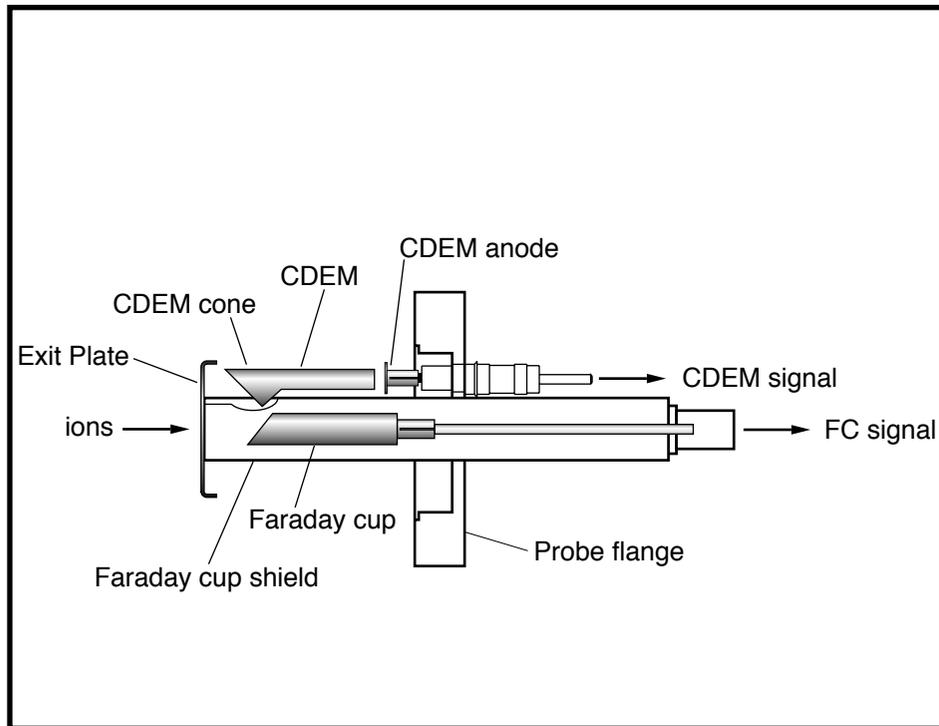
Positive  
Ion  
Detection

Off-axis  
configuration

Multi-channel (4 or 6) Continuous Dynode Electron Multiplier (CDEM).

Straight glass tube with a cone of the same material attached to the front end. Placed upright, next to the FC, and away from the axis of the QMF.

# Off-axis configuration



FC detection is standard in all RGA probes.  
FC units can be upgraded to a CDEM detector (factory)

# Faraday Cup Operation

$$P > 10^{-8} \text{ Torr}$$

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

## Advantages

Simplicity

Stability

Dynamic range

No mass dependence

No pressure dependence

Detection Limit:

$$10^{-10} \text{ Torr}$$

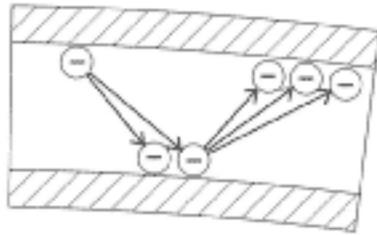
Sensitivity:

$$10^{-4} \text{ Amps/Torr}$$

(N<sub>2</sub> @ 28 amu)

# CDEM Upgrade

An electron multiplier upgrade is recommended when (1) the RGA is operated routinely under  $10^{-8}$  Torr, (2) when faster scan rates are required, (3) when a larger dynamic range is necessary, (4) when peaks need to be pulled out of the noise, and (5) when ion counting is required.



Cascade of electrons produced  
inside a Multiplier Channel  
<http://www.detechinc.com/notes.htm>

# CDEM Operation

## $P < 10^{-8}$ Torr

When the cone is biased negatively (-1000 to -2500V) relative to the FC, positive ions are 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 channels and produce more secondary electrons. Depending on bias voltage, up to  $10^7$  electrons come out at the back end and are picked up by the grounded CDEM anode plate. The resulting electron current is proportional to the incoming ion current and is measured by the same logarithmic electrometer as the FC current.

The gain of the CDEM is a function of the bias voltage and is measured relative to the FC signal.

An automatic Gain Adjustment mode was built into RGA Windows.

# CDEM Operation ctd.

Detection Limit:

$10^{-13}$  Torr

Gain (typical):

100 - 1000x

Gain Ion Counting:

$10^6$  typ.

# CDEM Operation ctd.

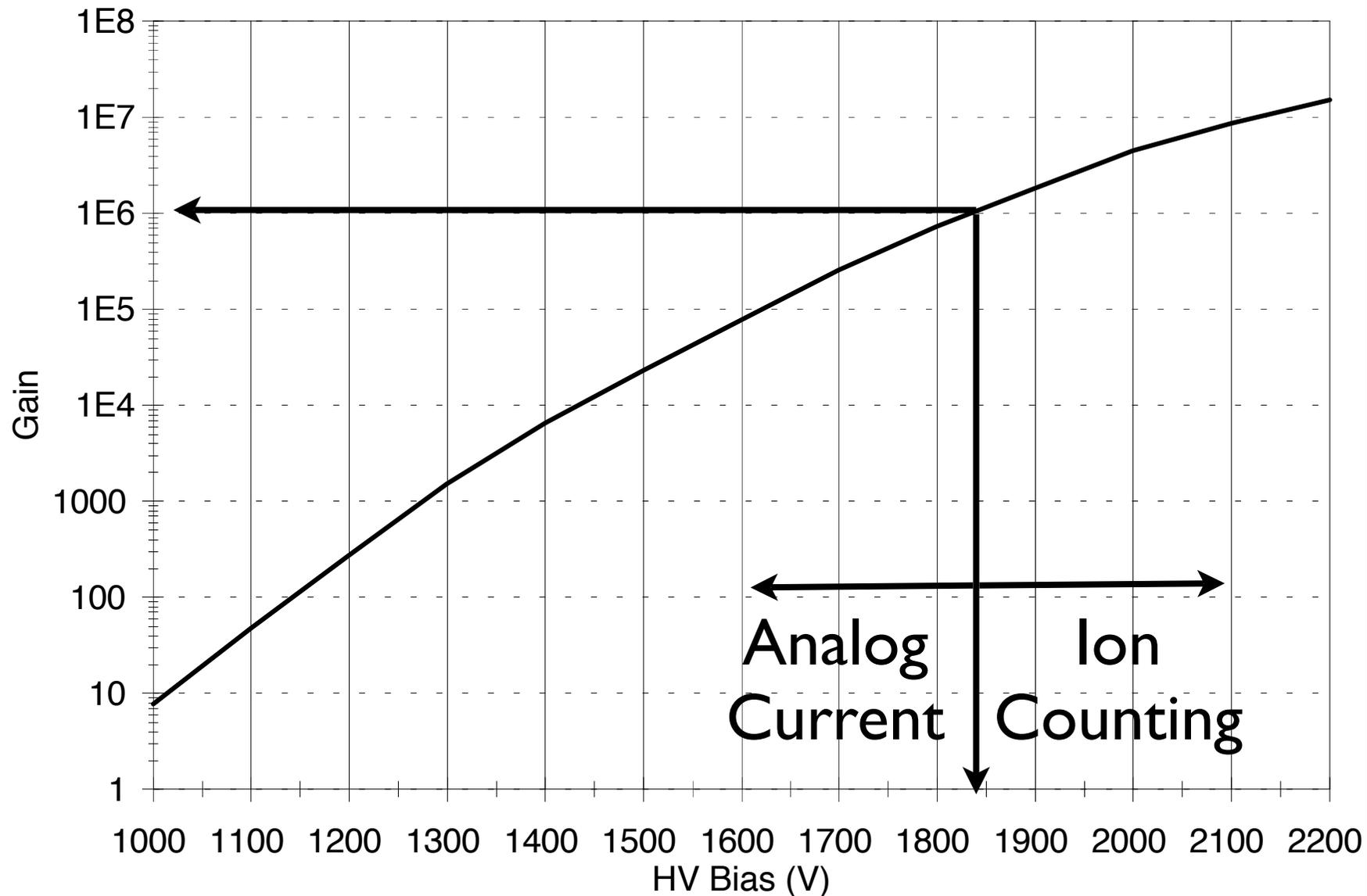
## Advantages

- High gain
- Excellent S/N
- Fast Scans
- Electron discrimination
- Ion Counting (Option 03)
- Field Replaceable

## Disadvantages

- Gain Degradation
- Mass discrimination ( $M^{1/2}$ )
- Current Saturation
- Low pressure limit ( $<10^{-5}$  Torr)
- Bakeout limitation ( $<100^{\circ}\text{C}, 300^{\circ}\text{C}$ )

# CDEM Gain (nominal)



# Gain Adjustment

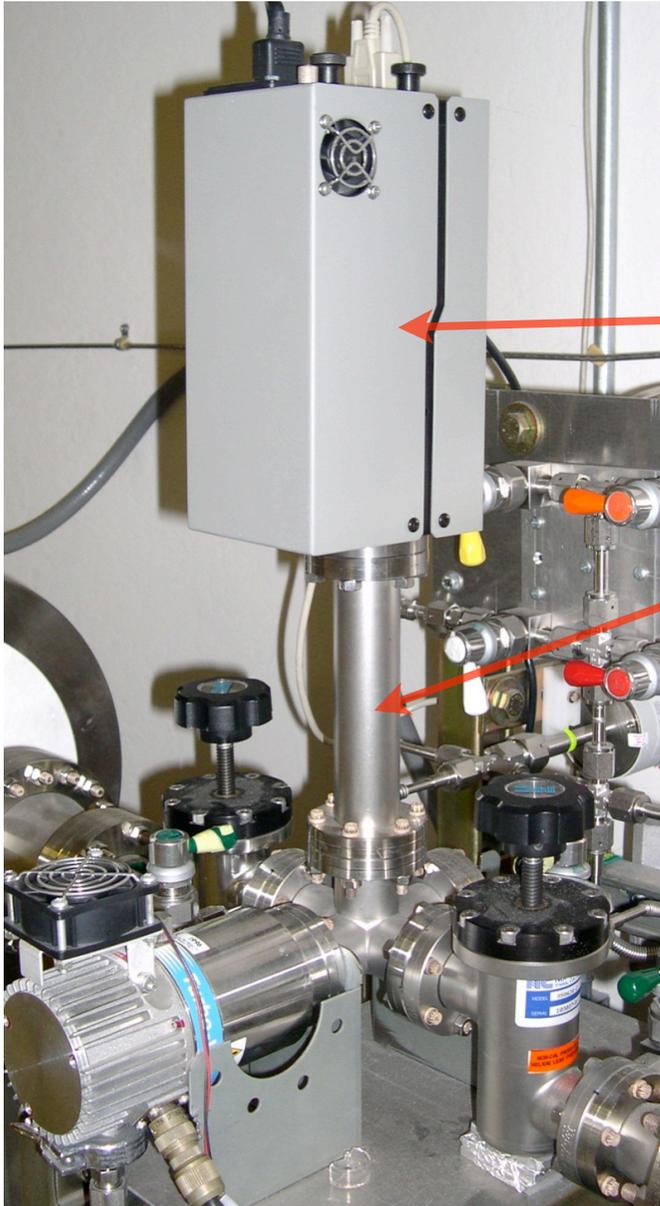
## How does it work?

1. Select RGA Windows / Head Menu/ CEM Settings
2. Enter a Gain (100 or 1000, typical)
3. Enter a Reference Mass ( $M_{Ref}$ )
4. Press Adjust
5. The QMF is parked at  $M_{Ref}$ .
6. The FC ion current,  $I_{FC}$ , is measured.
7.  $I_{FC}$  is multiplied by the Gain to provide the target current:  $I_{FC} \times \text{Gain} = I_{target}$ .
8. The ECU switches to CDEM detection.
9. Starting at a low bias voltage, the CDEM current,  $I_{CDEM}$ , is measured.
10. Compare  $I_{CDEM}$  to  $I_{target}$ .
11. Adjust the bias voltage until the  $I_{CDEM}$  current matches or slightly exceeds  $I_{target}$ .
12. Save the gain and voltage to the RGA head.

# Probe Maintenance

- Filament Replacement
- Ionizer Replacement
- Probe Cleaning
- Electron Multiplier replacement
- Electron Multiplier Refurbishment
- Mass Tuning
- Sensitivity Tuning
- Leak Rate Tuning

# Electronics Control Unit (ECU)

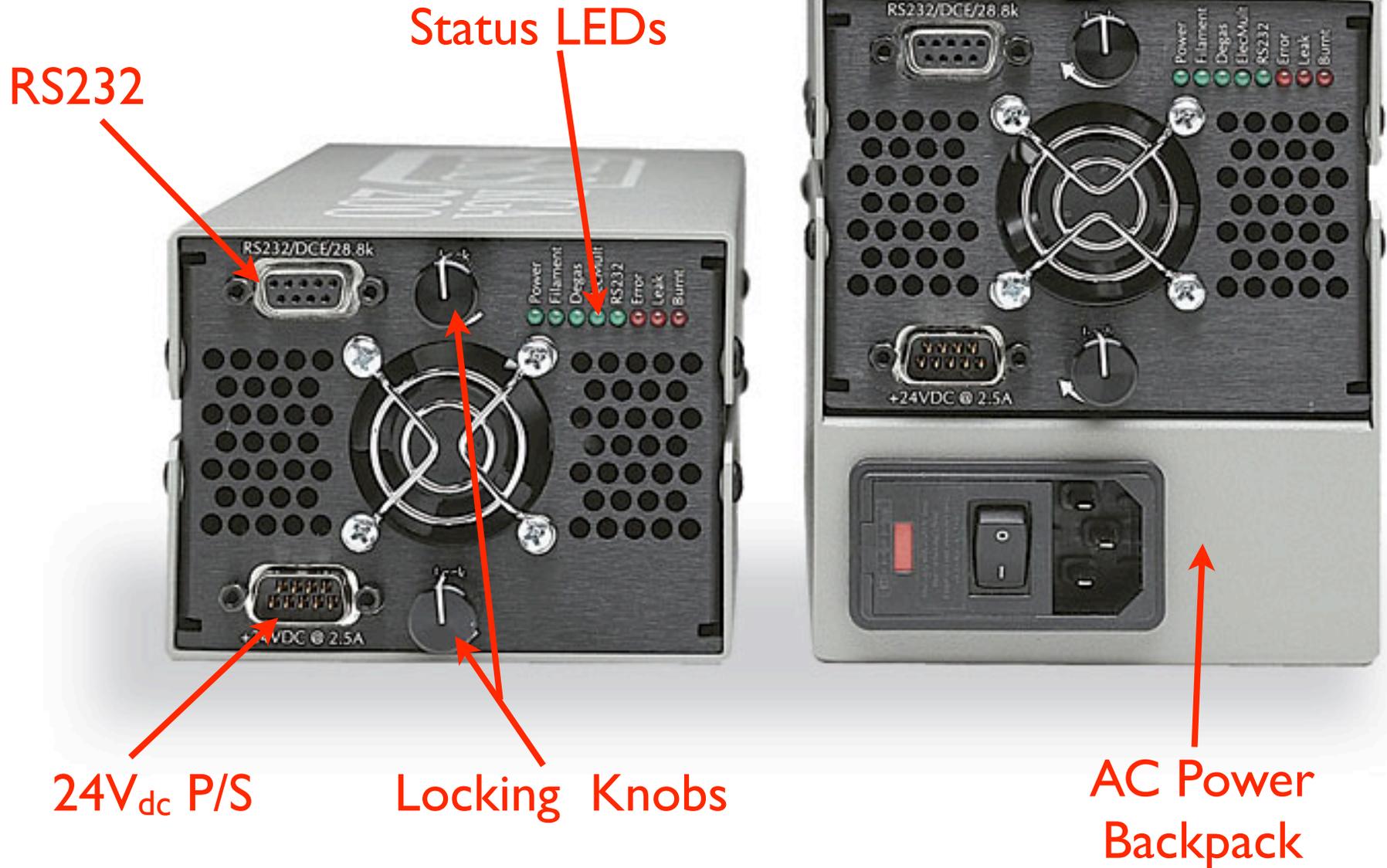


The ECU mounts directly on the RGA Probe and contains all the electronics required to operate the mass spectrometer.

# ECU Basics

The ECU is powered by either an external 24VDC (@2.5 amps) power supply or an optional, built-in power module which plugs directly to an AC outlet.

# ECU-Rear Panel



# Warnings

- Do not operate the ECU above 70 degrees Celsius
- Follow the High Voltage warnings on the box
- Make sure the locking knobs are tight
- Ground the ECU to the Facility ground

# ECU Components

- Regulated power supplies (Ionizer)
- Logarithmic Electrometer (pico-ammeter)
- Current calibrator
- RF & DC supplies (QMF)
- HV P/S (CEM)
- Built-in Microprocessor
- Control Firmware
- RS232 Interface
- Status LEDs
- I/O connectors

# Logarithmic Electrometer

A temperature compensated, logarithmic pico-ammeter built into the ECU box measures the ion currents collected by the FC, and the electron currents delivered by the CDEM.

The output of the electrometer is proportional to the logarithm of the ion current so that several decades of current can be read without any gain switching.

The microprocessor automatically configures the electrometer and connects its input to the correct signal based on the type of detector selected.

The electrometer is auto-ranging and temperature compensated

# Logarithmic Electrometer

- The electrometer measures both positive and negative currents (automatic and autoranging)
- Current range:  $10^{-15}$  Amps to  $1.32 \cdot 10^{-7}$  Amps.
- The electrometer response can be calibrated at any time thanks to a built in current source.
- Use RGA Windows/Head Menu/Calibrate Detector to update the electrometer's calibration
- The electrometer also auto-zeroes at the beginning of each scan.

# Current Averaging

The amount of averaging and the data acquisition rates for scans and single mass measurements are set by the Scan Speed (or Noise Floor) parameter:

(std. dev.)

SS	NF	Scan msec/amu	Single Mass msec/mass	Baseline Noise Amps
1	0	2000	2200	7e-15
2	1	1000	1100	1e-14
3	2	400	440	1.5e-14
4	3	200	220	2.0e-14
5	4	126	139	4.0e-14
6	5	45	50	1.2e-13
7	6	30	33	2.5e-13
8	7	15	16.5	5e-13

Lots of Averaging

No Averaging

# QMF Power Supply

All the electronics required to power the QMF are built into the ECU box.

The RF/DC levels for each mass are set and regulated from the ECU, under strict microprocessor control, and based on internal tuning parameters permanently stored in non-volatile memory.

Tuning parameters are pre-loaded at the factory as part of the test & cal procedure performed prior to shipment (i.e. ready to go out of the box).

# QMF P/S

The RF/DC tuning parameters can be tweaked at any time using the Peak Tuning Procedures supported by RGA Windows.

RF Frequency: 2.7648 MHz

RF Voltage: 8.56 V<sub>pp</sub>/amu

DC Voltage: 1.4V/amu

# Calibration Mixture

The SRS RGA mass axis is calibrated using a mixture of:  
He (lo mass), Ar, Kr and Xe (hi mass).

For 300 amu units PFTBA and perfluorononane are  
used to check the entire range.

Calibrated mixtures are available from third parties for  
routine mass tuning.

# PC Interface

- The SRS RGA includes a complete ASCII command set.
- The SRS RGA includes a RS232 serial communication port.
- SRS provides RGA Windows to interface the SRS RGA with a PC and perform standard measurements.
- SRS also provides a complete LabView Development Kit to implement custom measurement setups.

# Partial Pressure Measurement

Partial pressure signals with the SRS RGA are determined with the help of previously calculated sensitivity factors by reference to the abundance (i.e. peak current) of the individual mass numbers (i.e. principal peaks) attributed to each gas type.

For example: The partial pressure of  $N_2$  is determined measuring the ion peak current at 28 amu and using the corresponding sensitivity factor to scale that number into Torr.

# Partial Pressure Sensitivity

The partial pressure sensitivity of the RGA to a gas  $g$ ,  $S_g$ , is defined as:

$$S_g = (H-H_0) / (P-P_0) , [\text{Amps/Torr}]$$

Where,

- $H-H_0$ : change in principal mass peak height, [Amps], FC currents are used to determine sensitivities.
- $P-P_0$ : change in total pressure due to a change in partial pressure of the gas, Torr.
- $H_0$  and  $P_0$  are background values.

# Partial Pressure Sensitivity

The sensitivity of the RGA varies with:

- Different gases
- Ionizer settings
- QMF settings
- Detector settings
- Aging of the unit

Careful quantitative analysis requires that the sensitivity factor,  $S_g$ , be determined for every gas which maybe a component in a mixture being analyzed. Spectral deconvolution is recommended in the presence of spectral overlaps.

# Quantitative measurements

Even with careful in-situ calibration, independent studies have shown that RGA's should not be expected to deliver better than 3% accuracy for gas analysis measurements.

# SRS RGA Sensitivity

The nominal sensitivity factor for the SRS RGA is:

$$10^{-4} \text{ Amps/Torr}$$

for N<sub>2</sub> at 28 amu and FC detection.

A unit specific sensitivity factor is loaded into each SRS RGA prior to shipment as part of the factory test&cal procedure.

A Sensitivity Tuning procedure is available through RGA windows to adjust the sensitivity factor in the field.

# Nitrogen-Equivalent Readings

A single sensitivity factor is used to calculate all partial pressures in an analog scan. Since the sensitivity factor is based on nitrogen, all partial pressure readings are labeled as: nitrogen-equivalent readings.

# Tuning Procedures

- Sensitivity Tuning
- Leak Rate Tuning (Jefferson Lab)
- Peak Tuning (Mass Tuning)
- Electron Multiplier Gain
- Gas Conversion Factors

# Tuning Procedures

- UHV and XHV systems have very little impact on RGA tuning parameters
- UHV and XHV users should rarely need to perform any of the available tuning procedures described in the SRS RGA Manual.

# Mass Units - amu

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.  $^{12}\text{C}$ ), is the unit of molecular mass most commonly used in mass spectrometry.
- $1 \text{ amu} = 1.660540 \cdot 10^{-27} \text{ kg}$ .

# Mass number

- The mass number,  $M$ , is the sum of the number of protons and neutrons in a molecule.
- To a very close approximation the mass of a molecule, in amu, is equal to its mass number. This is particularly valid for low resolution mass specs such as RGAs.

# Mass-to-charge

- 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  $e^-$ .
- For example,  $^{36}\text{Ar}^{2+}$  and  $^1\text{H}_2^{16}\text{O}^{1+}$ , both have  $M/Q = 18$  and cannot be differentiated from each other by an RGA.

# Mass of an ion

- For singly charged ions, the mass/charge is numerically equal to the mass of the ion in atomic mass units.
- RGA users often use the term “mass-of-an-ion” when they really mean the mass/charge. This convenient way of speaking is strictly valid for singly charged ions only.

# What are RGA's used for?

RGAs are widely used to identify the kinds of molecules present in a residual gas environment.

When properly calibrated, an RGA can be used to determine the concentrations or absolute partial pressures of the components of a gas mixture.

# Areas of application...

- Residual Gas Analysis (HV, UHV and XHV)
- Surface Science (TPD, SIMS, laser ablation, adsorption, etc)
- Materials Science (i.e. outgassing, sorption)
- Gas Analysis (Atmospheric Sampling, MIMS, GC-RGA, IMS-RGA, TGA-MS)

# Specific applications...

- Leak Detection (not just He)
- Package Testing (Inside-out leak testing)
- Cryogenic Dewar testing
- Trapped gas studies (bubbles)
- Molecular beam monitoring
- Vacuum Oven monitoring
- Volcanic gas monitoring
- Underwater sampling
- Environmental testing
- VOC detection
- Laser ablation

# more applications...

- Miniature mass spec development (gold standard)
- Droplet and Aerosol Beam analysis
- Fluid Inclusions (petroleum)
- Cluster Studies
- TGA-MS (evolved gas)
- Pollution studies
- Portable MS systems
- Adsorption studies
- Water analysis
- Plasma monitoring
- Accelerators (XHV)

# Semiconductor applications

- Leak Detection (dynamic and crisis, background check)
- In-situ process monitor (in-chamber)
- Gas delivery monitor (on-line)
- Equipment diagnostics (mass flow controllers, valves, etc)
- Pump-downs
- Rate-of-rise
- End point detection
- Photoresist residues
- MBE beam flux and diagnostics
- Flat panel manufacturing
- “Good process” fingerprinting
- Exhaust management

# What are the Challenges?

- High pressures (differential pumping)
- Nasty Gases (reactive, corrosive, toxic)
- Harsh Environments (plasmas, heaters, guns)
- $< \text{Ppm}$  requirements
- Large pressure range

# Surface Science Applications

John T. Yates, Jr., “Experimental Innovations in Surface Science, A Guide to Practical Laboratory Methods and Instruments”, AIP Press, Springer, 1997.

# High Pressure Sampling

## $P > 10^{-4}$ Torr

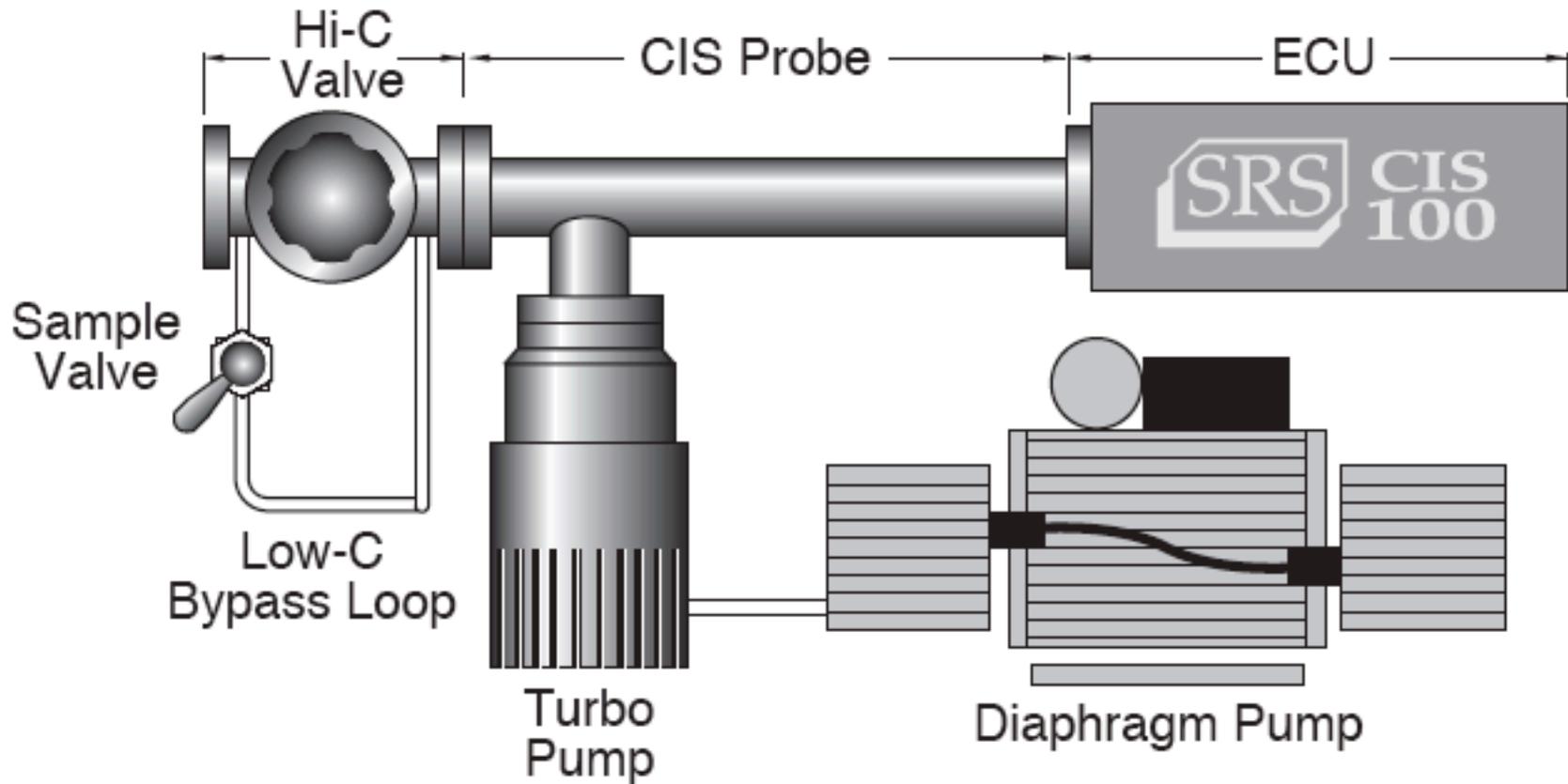
RGAs are routinely used to monitor gases at pressures above their maximum operating pressure of  $10^{-4}$  Torr.

Differential pumping is typically used to introduce the gases into the RGA chamber.

Alternative sampling schemes involve membrane inlet systems.

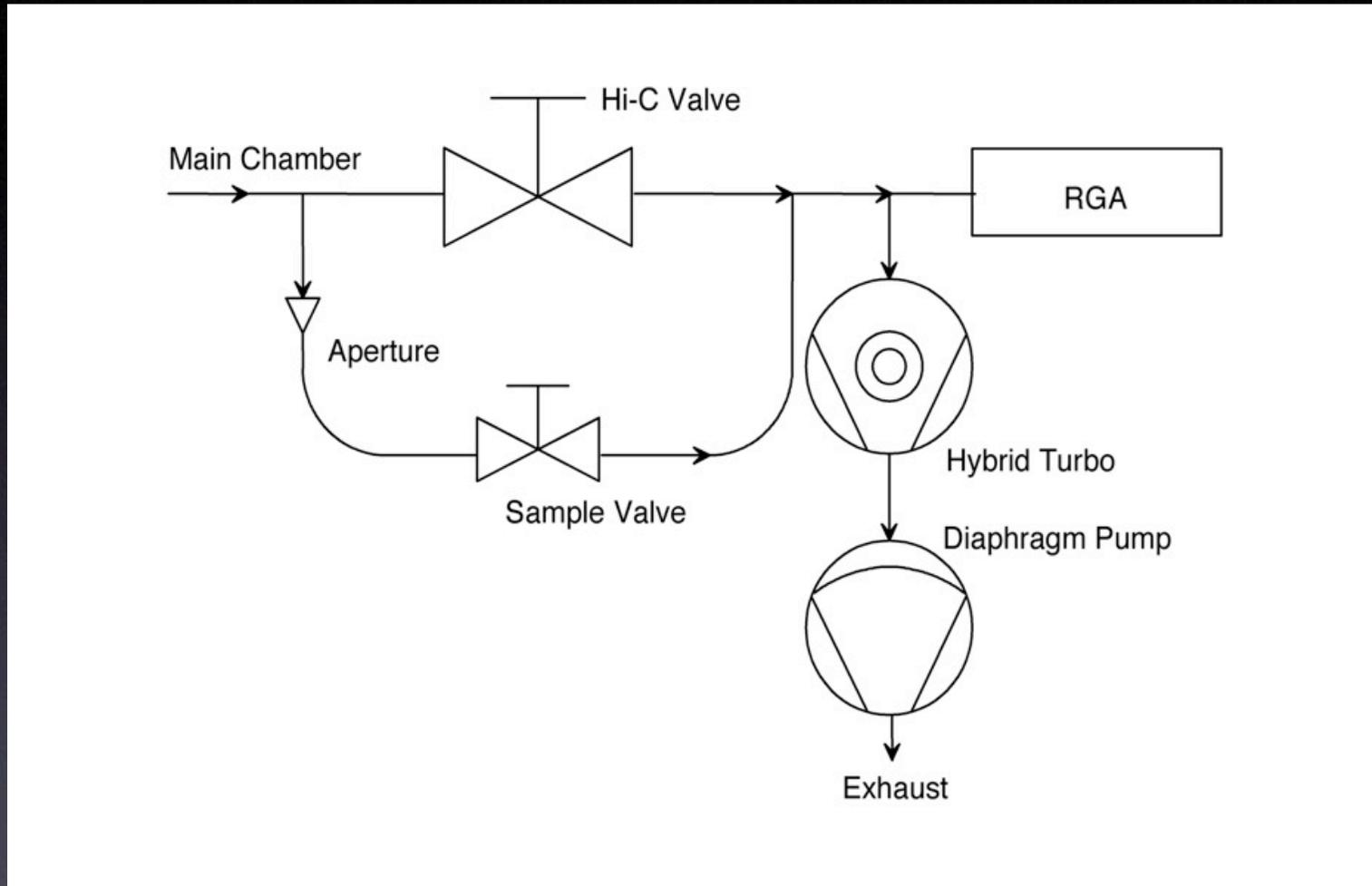
**For pressures <10Torr...**

**Single stage differential pumping - PPR**



*Figure 2: PPR inlet system*

# Single stage differential pumping



## Performance:

Inlet pressure:  $10^{-4}$  to 1 Torr

RGA Pressure:  $3 \cdot 10^{-6}$  Torr

Gas flow:  $\sim 10^{-4}$  Torr L s<sup>-1</sup>

Response time: 20 s @ 1 Torr inlet pressure (scales linearly with pressure)

# PPR...

## ...Advantages

- Low cost
- Simple design
- Flexible design
- Low Maintenance
- Dual Valve
- Low Det. Limit  
( $10^{-10}$ Torr FC)

## ...Disadvantages

- Background interf. ( $H_2O$ )
- Process interf. (Ar)
- > 100ppm water
- Slow response
- History effects
- Wall effects

- 0.34%  $Ar^+$ (36), 350 ppm  $Ar^{++}$ (18) -  
 $10^{-8}$  Torr  $H_2O$  background,  $3 \cdot 10^{-6}$  Torr RGA pressure

# $10^{-4}$ - $10^{-2}$ Torr CIS advantage

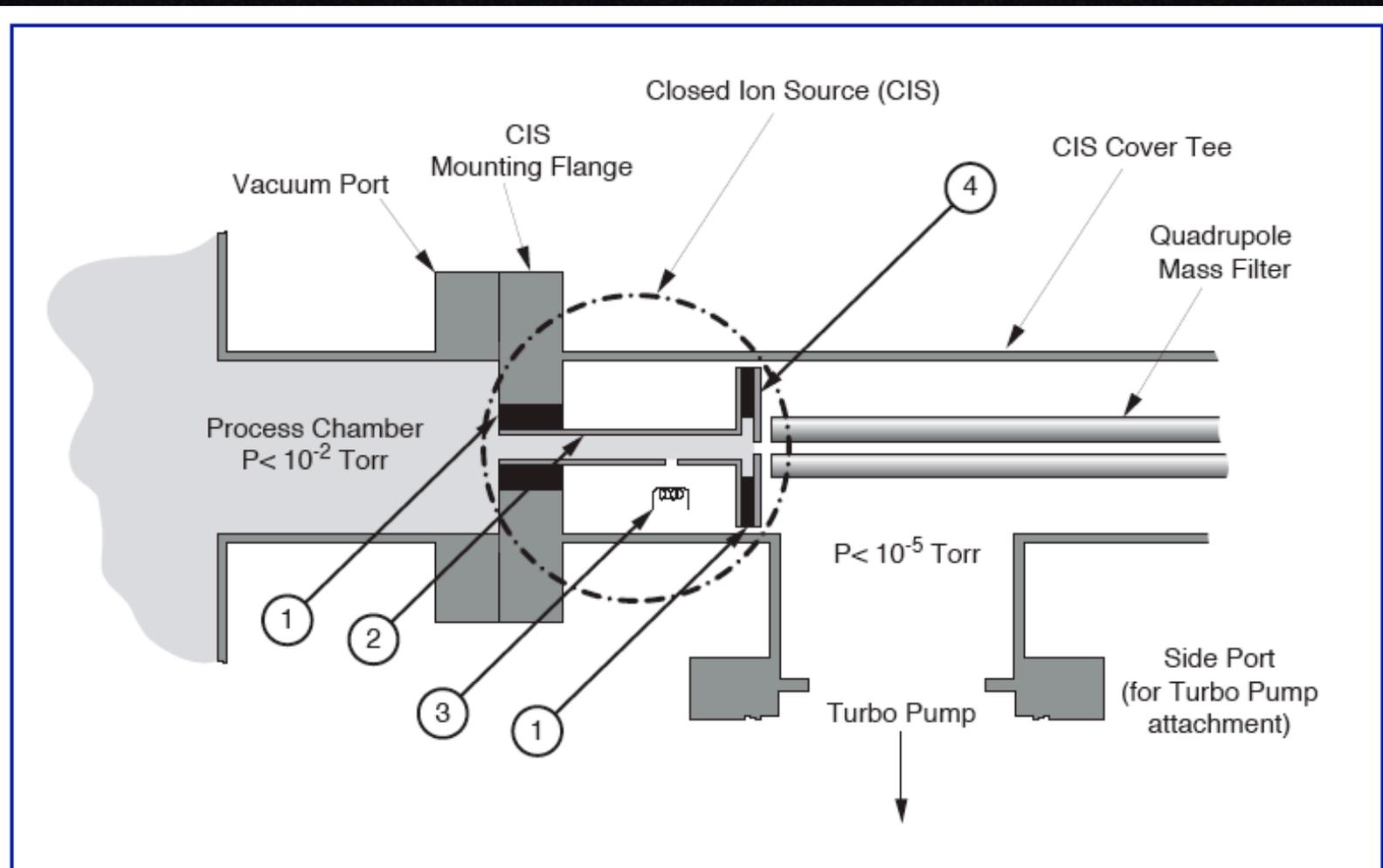


Figure 3: Schematic diagram of the CIS

## Advantages

Direct immersion  
Signal/Background  
CIS/RGA operation  
Low Fil. Emission  
W and ThO<sub>2</sub> fil.  
Low ESD  
CIS35 mode  
1 ppm water  
Faster response

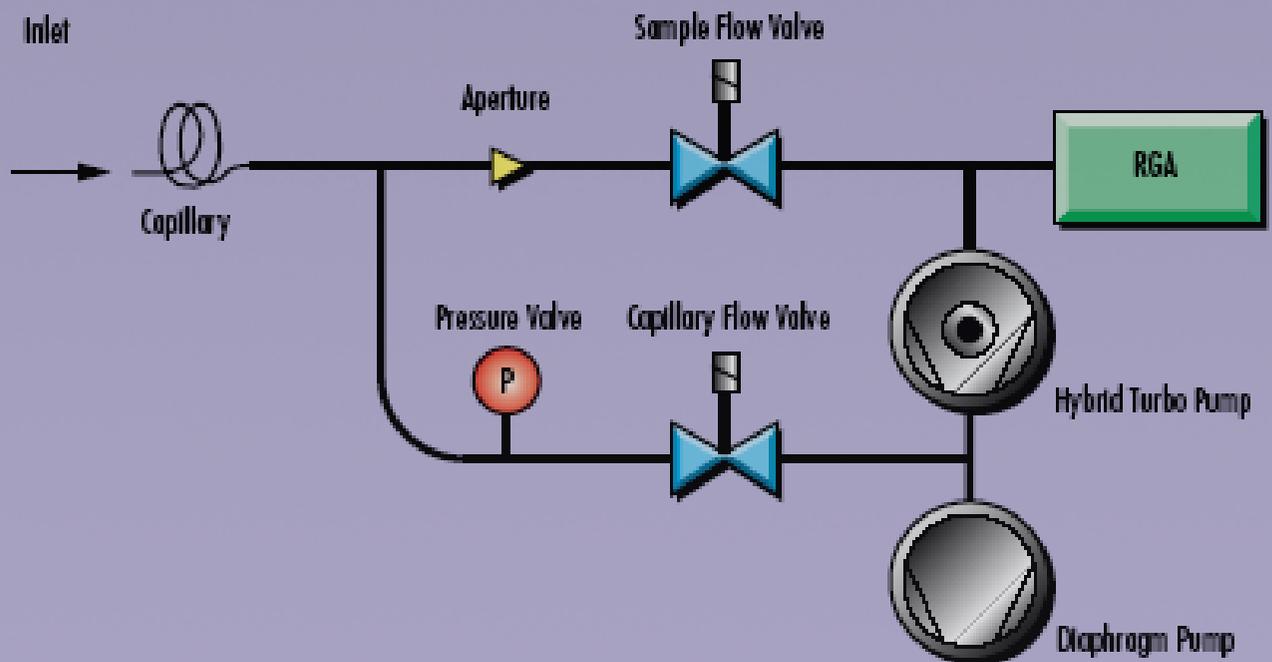
## Disadvantages

Expensive  
More maintenance  
Long term stability  
Filament life  
Contamination  
Degradation

- 0.34% Ar<sup>+</sup>(36), 350 ppm Ar<sup>++</sup>(18) -  
10<sup>-8</sup> Torr H<sub>2</sub>O background, 10<sup>-2</sup> Torr Ionizer pressure

# 10 Torr to Atm...

## Bypass pumping - QMS



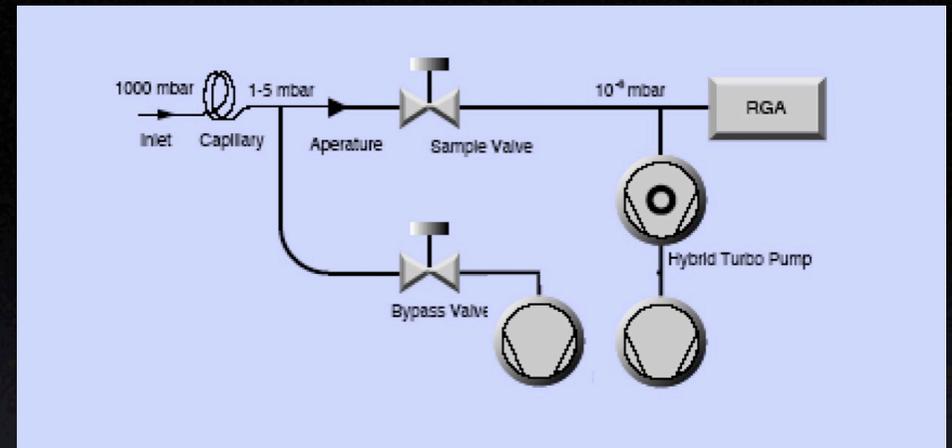
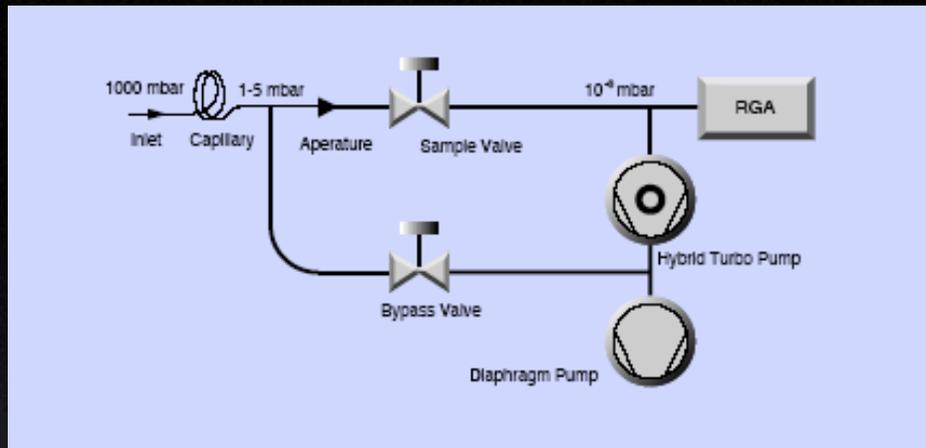
## **Advantages**

Fast response  
Compact  
Rugged  
Closed loop  
Capillary materials  
Closed Loop

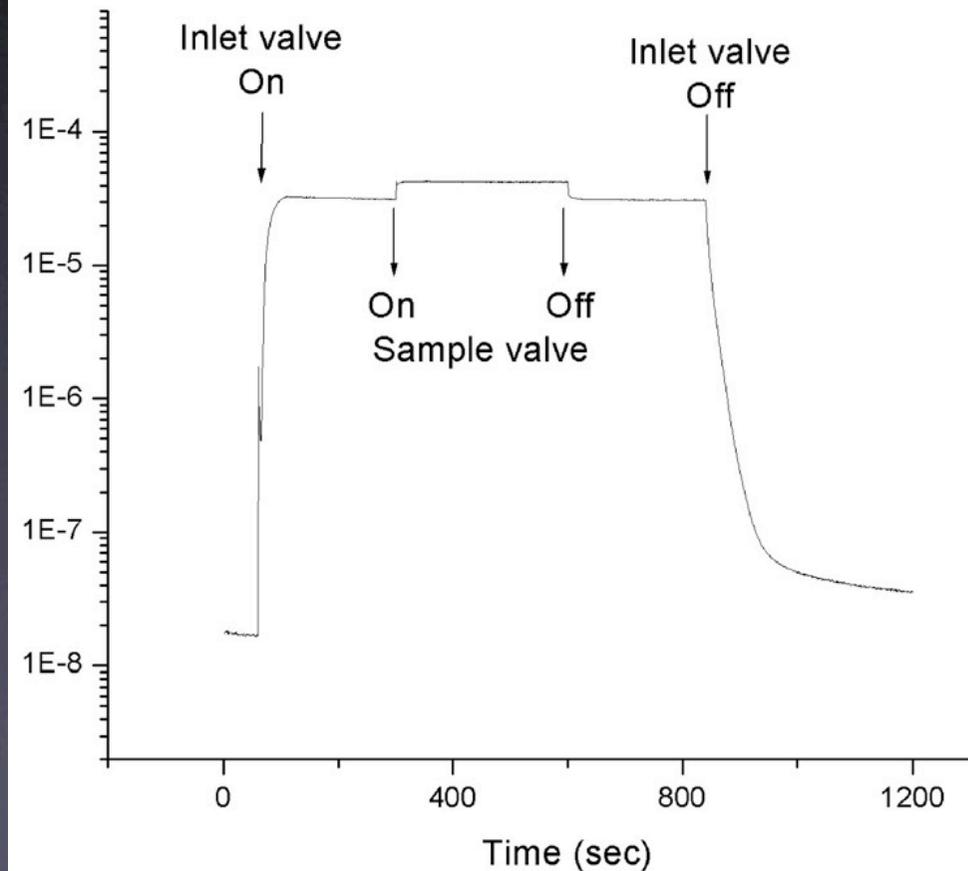
## **Disadvantages**

Light gas backstreaming  
Reactive gases  
Corrosive gases

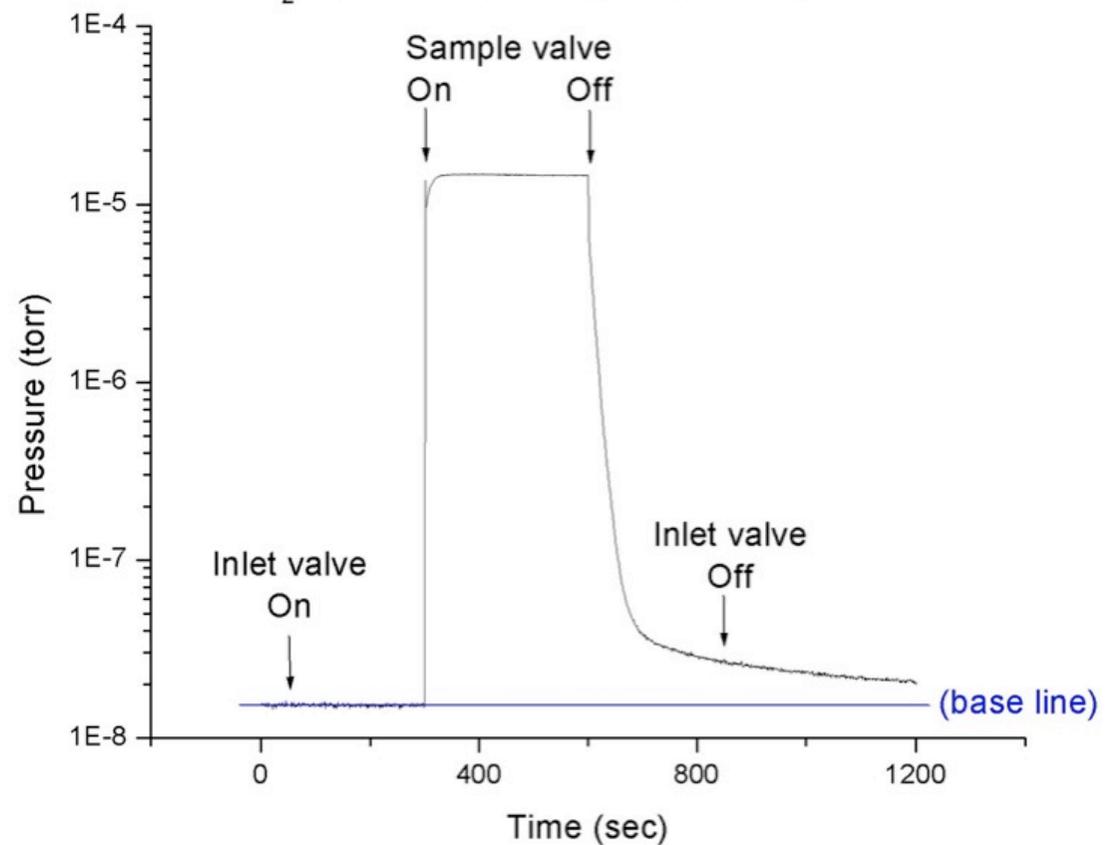
# Light Gases - Hydrogen



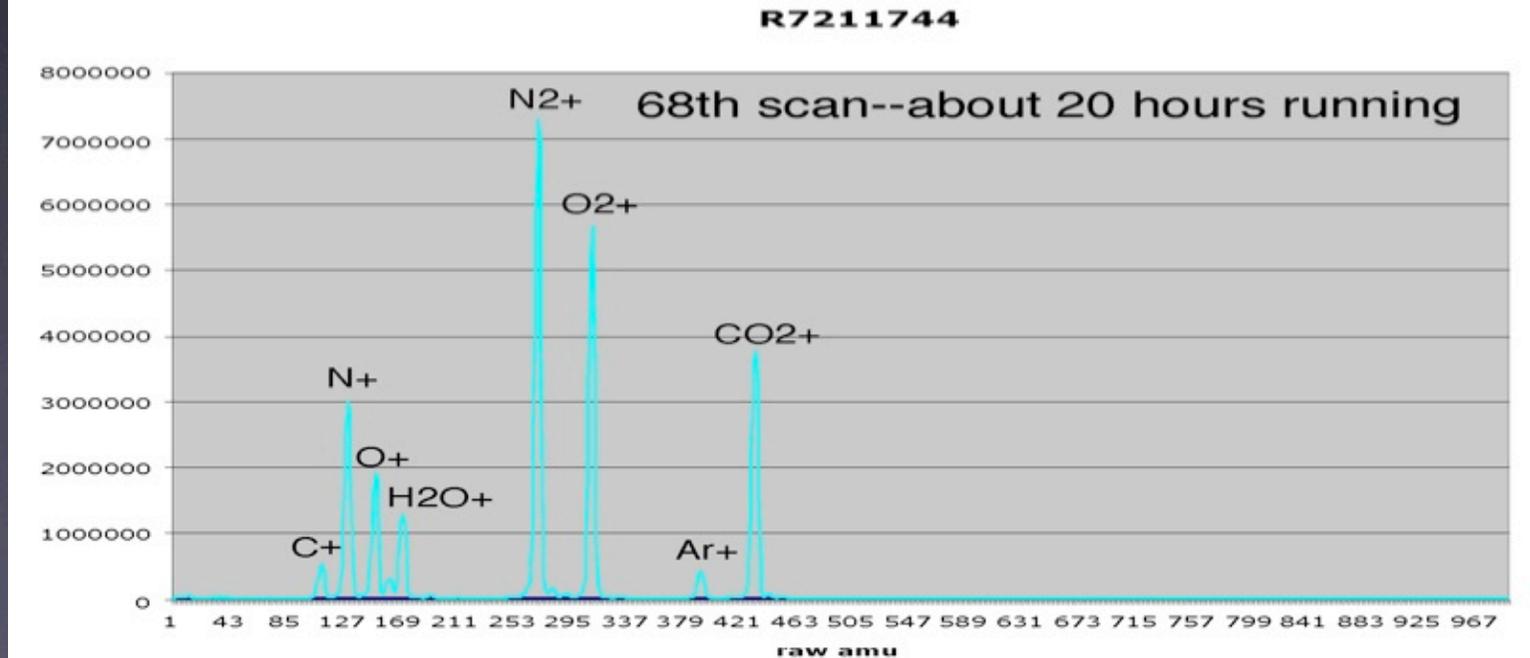
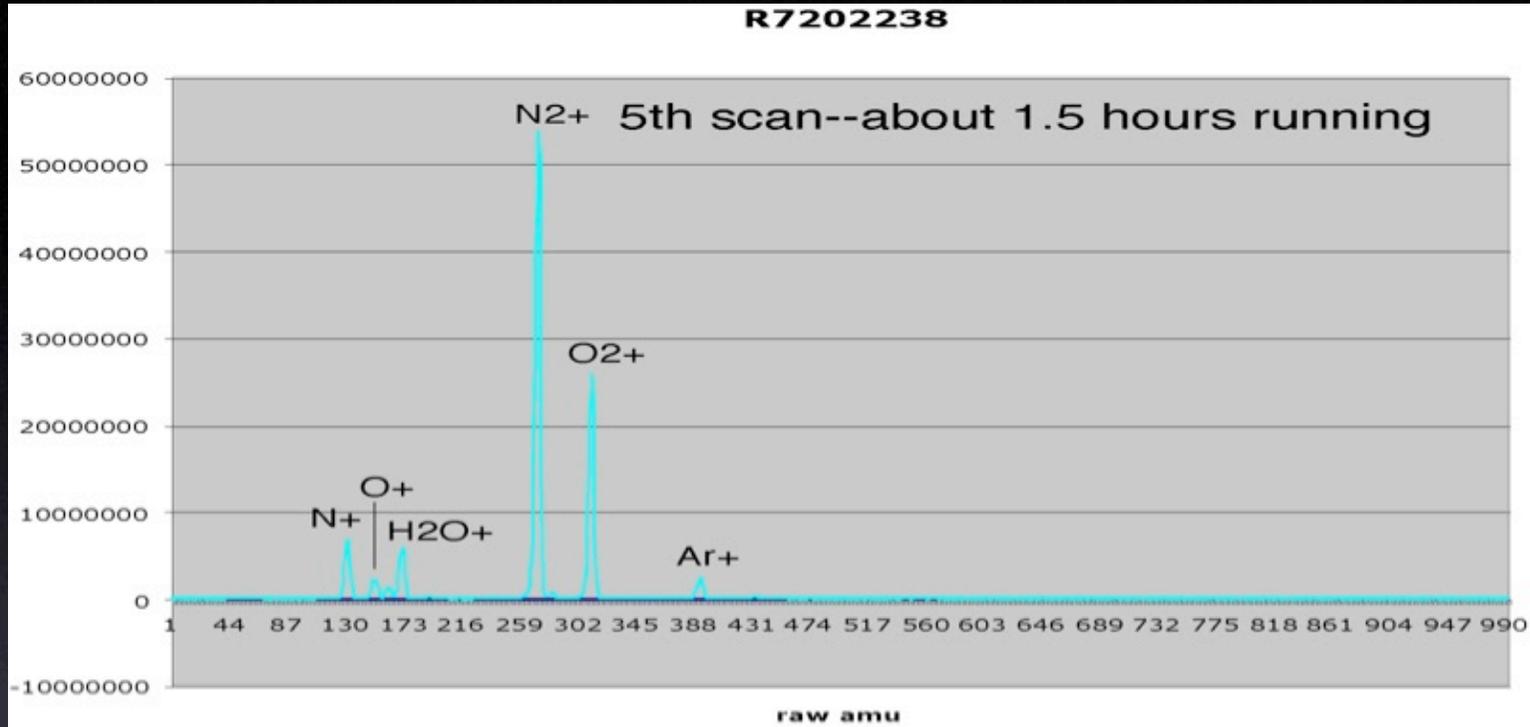
### H<sub>2</sub> gas behavior in QMS



### H<sub>2</sub> behavior in two-pump configuration



# Reactive Gases - No SO<sub>2</sub> Signal



# Corrosive gases

- Corrosion resistant TP
- Corrosion Resistance DP
- Dual Pump Configuration
- Coatings
- Filament Materials

# Basic Operating Modes

The firmware built into the RGA Head, completely controls the operation of the instrument and provides four basic modes of operation:

- Analog Scan
- Histogram (Bar Graph) Scan
- Single Mass Measurement
- Total Pressure Measurement

# PC Interface

- RGA Windows provides easy access to all RGA mass spec functions without any need for computer programming.
- SRS RGA can also be programmed directly using the RGA Command Set supported by its PC interface
- A LabView development Kit is also available from SRS.

# RGA Windows

- RGA Windows simply provides a graphical interface to display the mass spectrometer's data in a format compatible with residual gas analysis needs.
- All the necessary calibration and tuning parameters of the spectrometer are stored in the RGA Head itself. SRS RGA is a true stand-alone mass spectrometer.
- The basic operating modes supported by RGA Windows are based on the four basic operating modes supported by the head's firmware.

# RGA Windows

- Latest version 3.0
- Available as download: <http://www.thinksrs.com/downloads/soft.htm>
- Version 2.3 is officially obsolete!

# What's new?

- 32 bit platform compatible
- Multi-thread support
- Faster response
- New RS232 connection panel
- Full support of Windows 98, 2000, XP
- Support of higher screen resolutions
- New toolbars and buttons
- Improved Help engine - Includes What's New?
- Virtual instrument connection- training

# New Mass Spec Functions

- Scan w/out filament - good for scientists
- Improved “P vs. time” saves on the fly (unlimited saves)
- Larger “P vs time” buffer (256Mbytes)
- Logging for all scan modes (unlimited saves)
- Global settings for Table and “P vs. time”
- Improved Leak Testing
- New Leak Calibration menu
- Running averages for analog scans
- New Scan Setup button
- New Schedule button
- Improved Library Search

# More New MS Functions

- New CDEM toggle button
- New CDEM Settings menu item
- New End Button
- New Progress Cursor in Histogram mode
- New Leak Rate Calibrator option
- New Maintenance Schedule
- Autocomplete gas names in Table setup

# New Graphing Functions

- New Linear/Log Y-axis button
- New Control View Tool Bar - zoom, pan, scale
- Real-time graphical cursor in all modes
- View on/off for all gas traces in P vs time.
- New Active Graph objects - left mouse button
- New legends for “P vs. time”
- Improved Analyze mode
- Analyze logging fully implemented
- Floating Scan and Schedule setup windows

# Analog Scanning

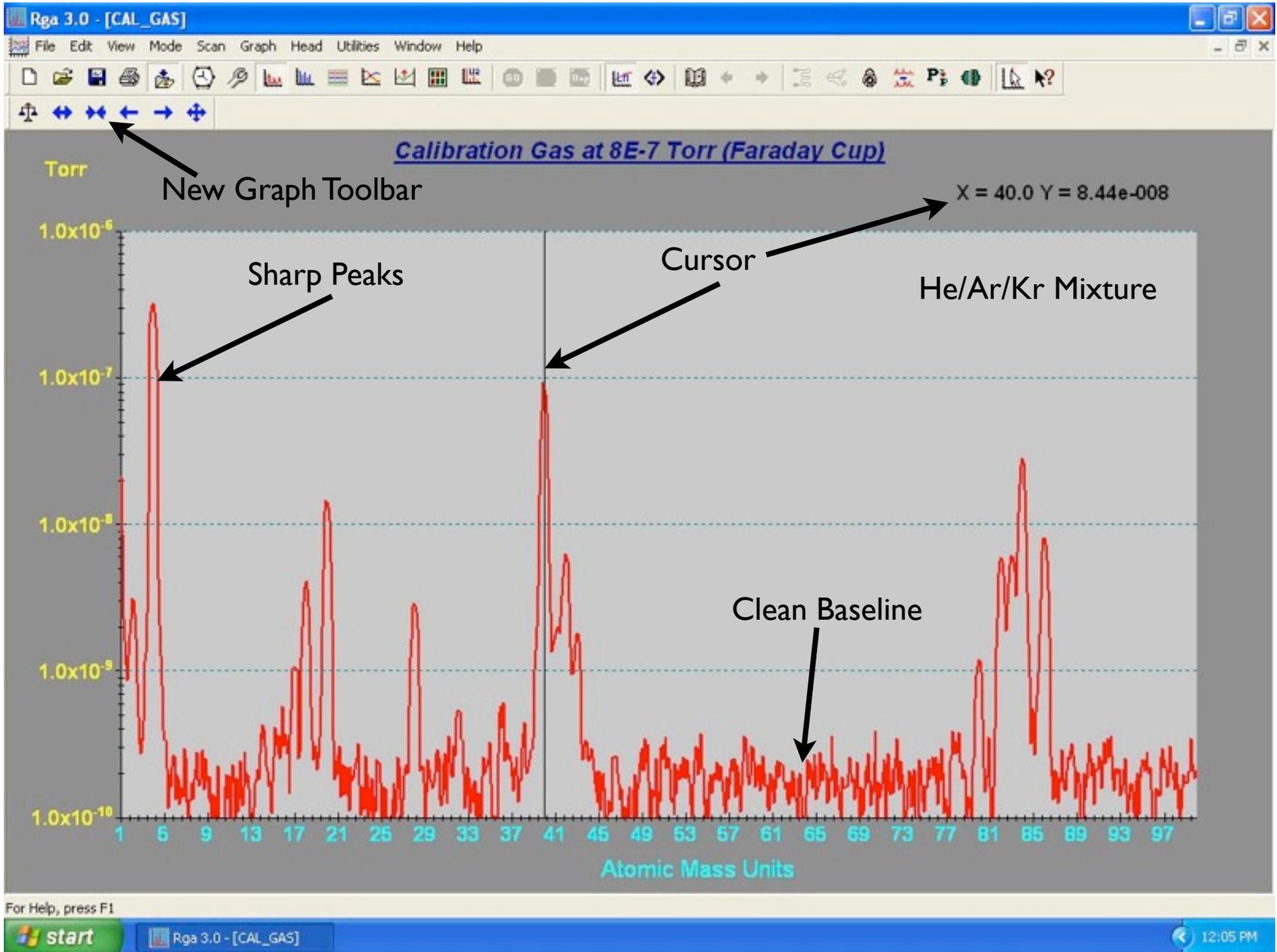
# Analog Scanning

- The most basic mode of operation of the RGA as a quadrupole mass spectrometer
- During analog scanning, the quadrupole mass spectrometer steps at fixed mass increments through a pre-specified mass range. The ion current is measured after each mass increment and transmitted to the host computer over RS232
- RGA Windows collects the data and displays it in a mass spectrum format: ion current (or partial pressure) vs. mass

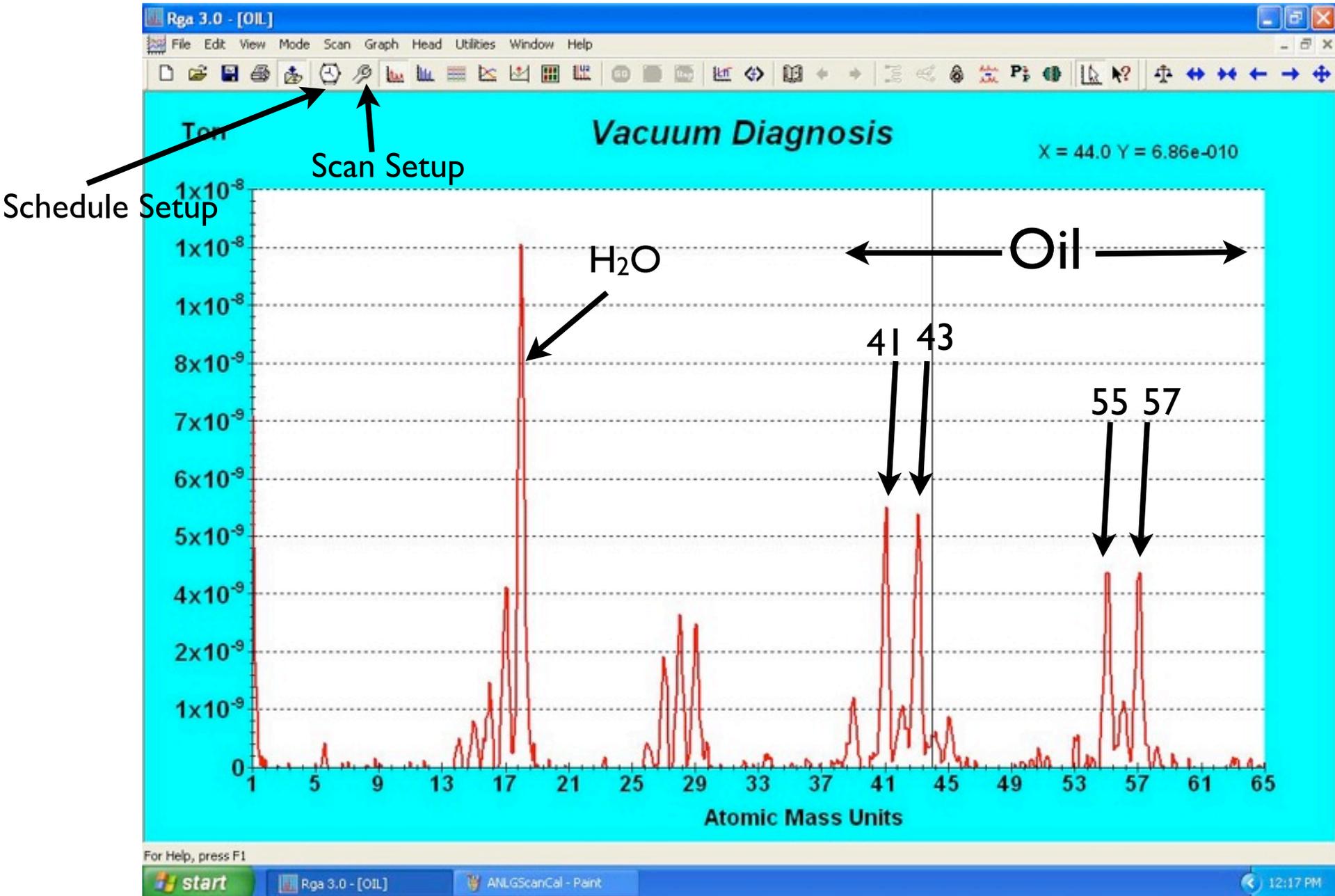
# Main Uses

- Peak tuning
- Fractional mass detection
- Peak shape characterization
- Mass spec performance characterization
- Reality check before “P vs time”
- S/N analysis and Scan Speed optimization
- Blind scan logging

# Analog Scan Example



# Oil Contamination



# Oil Contamination

- Look for clusters at 41/43, 55/57 and so on.
- Clusters in 14 amu increments
- -CH<sub>2</sub>- : 14 amu
- Use scan logging to track oil contamination

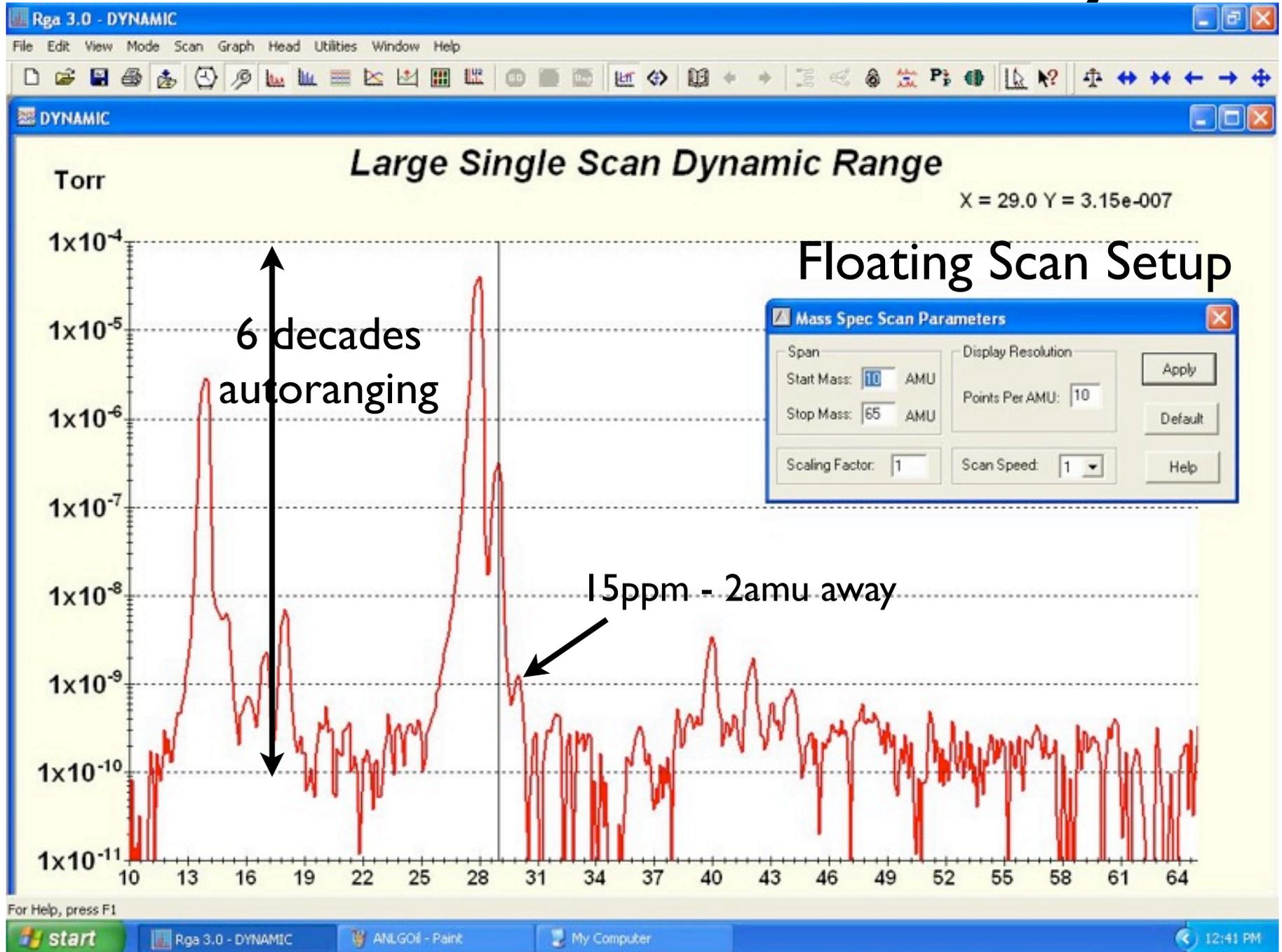
# Oil Contamination

- Bakeout often
- Use traps, cold fingers and water cooled baffles
- Avoid organic cleaning solvents
- Use oil-free pumping systems
- Use water based cleaners
- Avoid elastomers and plastics
- Avoid lubricants and greases
- Clean parts before exposure to vacuum

# Vacuum Cleaning

- Mr. Clean!
- Micro cleaner - from Cole-Parmer
- Branson ultrasonic cleaner
- Luminox- for aluminum parts
- Lots of rinsing....left over detergent outgasses
- Deionized water to rinse
- Hot water is better
- Vacuum baking is always good

# Abundance Sensitivity



# Histogram Scanning

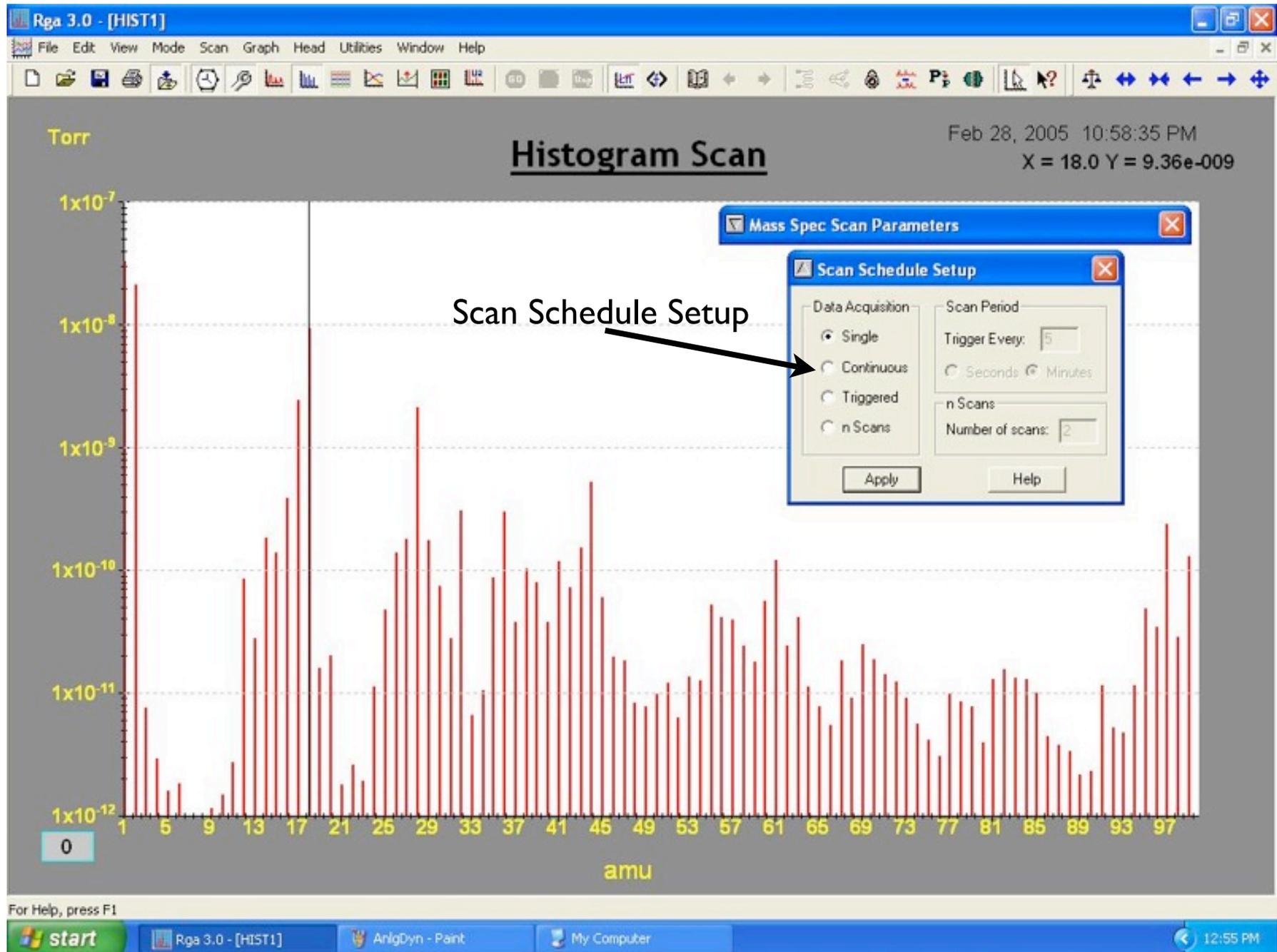
# Histogram Scan

- The Histogram (Bar Graph) scan consists of a succession of individual peak-height measurements over a pre-specified mass range. A single value is used to represent the peak heights at each integer mass within the range.
- Peak height measurements are done based on the Peak Locking (Miniscan) procedure described in Single Mass Measurement.

# Advantages

- Very popular display mode
- Reduced amount of data
- Simplified mass spectrum representation
- Faster spectral interpretation

# Histogram Scan Example



# Single Mass Measurement

# Table Mode

- SRS RGA can measure individual peak heights at any integer mass within its mass range.
- RGA windows uses this measurement mode to collect its data for: Table, Pressure vs. time, Annunciator and Leak Detection modes.

# Peak Locking

- During a single mass measurement the RGA head performs a “Miniscan” around the mass requested and the maximum current value detected is sent out over RS232.
- This scanning procedure is called Peak Locking and is designed to correct against 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.

# Uses of Single Mass Measurement

- Collect data for Table, Annunciator, “P vs. time” and Leak Detection Modes.
- Link the Single Mass data to PLCs
- Reduce the amount of data collected vs. scan logging
- Display “corrected” partial pressures in real time.
- Extend the dynamic range by toggling between FC and CDEM measurements

# Table Mode Setup

**RGA Table Scan**

Ch#	Name	Mass	mBar	Alarm
1	Hydrogen	2	0.3E-08	NO
2	Water	18	3.9E-08	NO
3	Nitrogen	28	9.4E-09	NO
4	Oxygen	32	1.8E-09	NO
5	CO2	44	4.4E-09	NO
6	Oil	55	6.6E-09	NO
10	Floor	21	1.0E-09	NO

**Floating Setup Dialog:**

View	Enable	Name	Mass	Scaling	CEM	Speed	Alarms
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 1 Hydrogen	2	1	<input type="checkbox"/> ON	3	Alarm 1
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 2 Water	18	1	<input type="checkbox"/> ON	3	Alarm 2
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 3 Nitrogen	28	1	<input type="checkbox"/> ON	3	Alarm 3
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 4 Oxygen	32	1	<input type="checkbox"/> ON	3	Alarm 4
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 5 CO2	44	1	<input type="checkbox"/> ON	3	Alarm 5
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 6 Oil	55	1	<input type="checkbox"/> ON	3	Alarm 6
<input type="checkbox"/>	<input type="checkbox"/>	Ch 7	1	1	<input type="checkbox"/> ON	8	Alarm 7
<input type="checkbox"/>	<input type="checkbox"/>	Ch 8	1	1	<input type="checkbox"/> ON	8	Alarm 8
<input type="checkbox"/>	<input type="checkbox"/>	Ch 9	1	1	<input type="checkbox"/> ON	8	Alarm 9
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Ch 10 Floor	21	1	<input type="checkbox"/> ON	3	Alarm 10

**Global Settings:**

All Channels

CEM:  ON

Speed: 8

Auto-complete gas names

For Help, press F1

Windows Taskbar: start | Rga 3.0 - TAB1 | Tabledosed - Paint | My Computer | 2:04 PM

# Table Mode Setup

The screenshot displays the Rga 3.0 - TAB1 software interface. The main window is titled "RGA Table Scan" and contains a table with the following data:

<u>Ch#</u>	<u>Name</u>	<u>Mass</u>	<u>mBar</u>	<u>Alarm</u>	<u>Speed</u>	<u>Cal</u>	<u>CEM</u>
1	Hydrogen	2	9.3E-08	NORMAL	3	1.00	OFF
2	Water	18	3.9E-08	HIGH	3	1.00	OFF
3	Nitrogen	28	9.4E-09	LOW	3	1.00	OFF
4	Oxygen	32	1.8E-09	NORMAL	3	1.00	OFF
5	CO2	44	4.4E-09	NORMAL	3	1.00	OFF
6	Oil	55	6.6E-09	NORMAL	3	1.00	OFF
10	Floor	21	1.8E-09	NORMAL	3	1.00	OFF

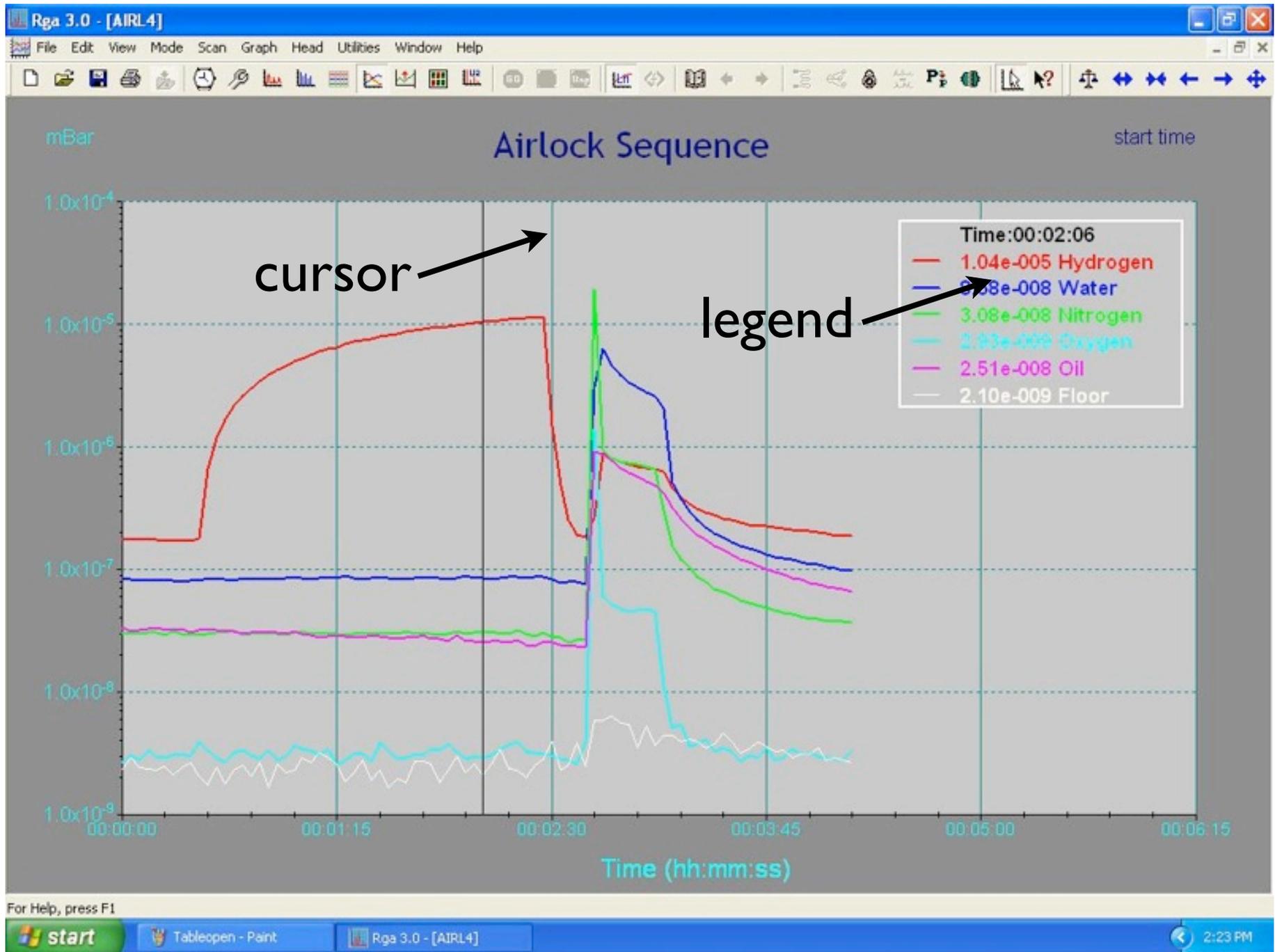
The software interface includes a menu bar (File, Edit, View, Mode, Scan, Graph, Head, Utilities, Window, Help) and a toolbar with various icons. The Windows taskbar at the bottom shows the Start button, open applications (Rga 3.0 - TAB1, Histogram - Paint, My Computer), and the system clock (2:03 PM).

# Pressure vs. Time

# Pressure vs. time

- Use the single mass measurement capability of the SRS RGA to follow the concentrations of up to ten gases as a function of time.
- RGA Windows displays the data in colors so the different gases can be easily visualized.
- Cursors and legends provide real time partial pressure values
- Perform process analysis, rate-of-rise and pump-down measurements.

# P vs. time



# Leak Test

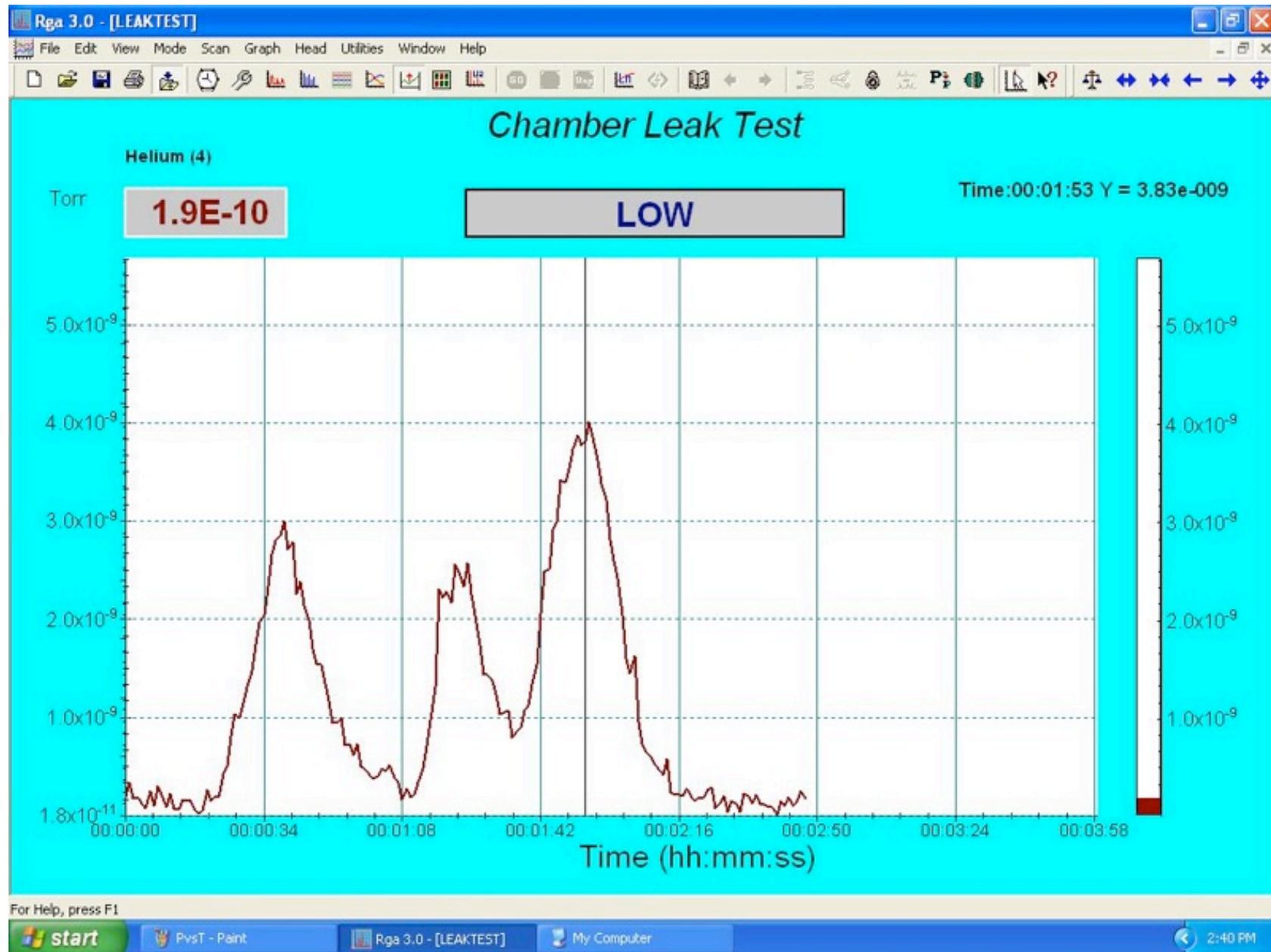
# Leak Test Mode

- The single mass measurement capability of the SRS RGA is used to monitor an individual gas in real time.
- Any gas within the mass range can be monitored
- Sound alarms and pressure-proportional tones provide audio feedback
- Gas levels can be displayed in Amps, Torr or  $\text{Torr}\cdot\text{L}\cdot\text{s}^{-1}$
- A built in leak valve can be used to calibrate the RGA in-situ - Leak Rate Tuning

# Leak Test Mode

- The most common use of Leak Test mode is to detect gas leaks in vacuum systems.
- RGAs are excellent leak detectors!

# Leak Test Example

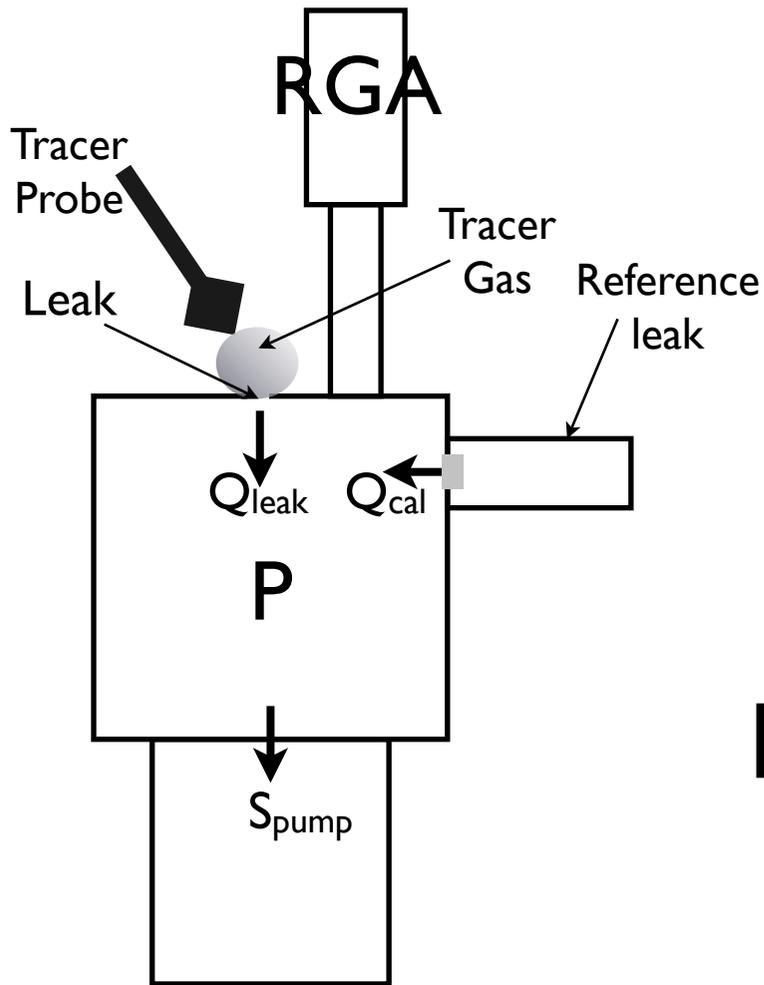


# Why Leak Test with an RGA?

- Use any gas, not just Helium!
- Dynamic Testing
- Inside-out-leak testing
- Leak rate display
- Process control as well!
- Distinguish between outgassing, virtual leaks and real leaks
- Cheaper
- Lower maintenance
- No more sharing. Dedicated RGA!

# Leak Rate Tuning

$$Q_{\text{cal}} = P \cdot S_{\text{pump}}$$



$Q_{\text{cal}}$ : reference leak rate,  
 $\text{Torr} \cdot \text{L} \cdot \text{s}^{-1}$

$P$ : partial pressure reading,  
 $\text{Torr}$  (tracer gas)

$S_{\text{pump}}$ : leak rate factor,  
 $\text{L} \cdot \text{s}^{-1}$

# Leak Rate Tuning

1. Enter reference leak rate

Torr **0.0E-00**

**NORMAL**

2. Enter mass of the tracer gas

3. Press the Measure button

4. The Leak Rate Factor ( $S_{\text{pump}}$ ) is calculated and stored within RGA Windows.

**Leak Rate Tuning**

Calibrated Leak (Q)

Reference Leak Rate:  Torr\*L/s

Leak Rate Factor

Partial Pressure Reading (P):  Torr

Leak Rate Factor Q/P:  L/s

Mass Selection

Mass used for Partial Pressure readings:  AMU

# Next.....

- Hands-on RGA demo
- RGA Windows tutorial
- Learn how to use your RGA as a leak detector
- Discuss your specific applications
- “Questions & Answers” session

**What did I leave out?  
Any questions?**

Thank you....

See you later...