Vacuum Technology
for Mass Spec Instruments

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Vacuum For Mass Spectrometry

This Webinar discusses how vacuum technology is a fundamental component Mass Spectrometers.

Beginning with the simple question ‘Why do we need vacuum’ we will move on to discuss the types of vacuum technology typically used on mass specs, and then review the evolution of vacuum subsystems from the 1990’s to the present.
Vacuum for Mass Spectrometry

• Why Mass Spectrometry Needs Vacuum
• Vacuum Technology
  - Measuring Vacuum
  - Rough Vacuum & High Vacuum Pumps
• System Design Concerns
• Vacuum for Mass Spec
  - Evolution of Vacuum Subsystem Design
  - Direction and Future Trends
Why We Need Vacuum for Mass Spectrometry

Avoid Signal Loss
- Avoid signal losses from collisions with residual (background) gas species by creating a long MEAN FREE PATH
  - Rough Vacuum (10^-3 Torr): 5 cm
  - High Vacuum (10^-6 Torr): 50 m
  - Ultra High Vacuum (10^-9 Torr): 50 km

Remove Chemical Contamination
- Avoid signal losses from reactions with residual (background) gas species by creating a CHEMICALLY CLEAN ENVIRONMENT
  - Rough Vacuum (10^-3 Torr): 3E13 part/cm^3
  - High Vacuum (10^-6 Torr): 3E10 part/cm^3
  - Ultra High Vacuum (10^-9 Torr): 3E7 part/cm^3

Prevent Arcing
- Many mass spec elements require the ability to maintain LARGE ELECTRIC POTENTIAL without ‘arching’
  - Electrostatic Lenses (-250 → +250 DC)
  - Quadrupoles (-4K → +4K RF + DC)
  - Ion Detectors (-7K DC → +7K DC)
Rough, High and Ultra-High Vacuum

- **Ultra-High Vacuum**
- **High Vacuum**
- **Rough Vacuum**

- **Molecular Flow**
- **Viscous Flow**
MEASURING ROUGH VACUUM: Atm - 10^{-3} Torr

**Thermocouple Gauges**

- **CONVECTION**: Maintain filament at constant T (above ambient)
  - Pressure $\propto$ Current
  - Atm $\rightarrow$ < 1 x 10^{-3} Torr
- **THERMOCOUPLE**: Maintain filament at constant CURRENT
  - $P \propto T$
  - Slow response time; non linear above $\approx$ 2 Torr

**Thermal (Pirani) Gauge**

- Energy (temperature) loss from a heated filament upsets the balance of a Wheatstone Bridge (typ) circuit
  - Pressure $\propto$ Voltage (atm – 10^{-3} Torr)
  - Gas type dependent (based on Thermal Capacity)
  - Extremely non-linear above 1 Torr): Caution when measuring Argon in this range

**Capacitance Manometer**

- Pressure differential distorts metal diaphragm, changing capacitance of a calibrated circuit
  - Pressure $\propto$ Capacitance
  - Full range from 1000 Torr to 0.1 Torr (approx. 3½ decades/gauge)
  - Fastest response, most accurate, gas type independent

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https://cds.cern.ch/record/455555/files/p75.pdf
MEASURING HIGH VACUUM: $10^{-3} - 10^{-8}$ Torr

**Hot Ionization (BA) Gauge**

- Electrons from filament (accelerated by e-field on spiral grid) strike ‘background’ gas molecules creating $M^+$ ions
  - Ions accelerated towards central collector – resulting ion current $\propto$ to gas density (pressure!)
  - Gas Type Dependent (Ionization Potential)
    - Accuracy: $\pm 20\%$ f.s. (typical)
    - $10^{-3}$ to $10^{-12}$ Torr Operating Range

**Inverted Magnetron Gauge (IMG)**

- **Agilent (Varian) invention**
- Electrons from plasma inside small metal cylinder (accelerated by magnetic field) create $M^+$ ions
  - Coulomb attraction drives M+ ions into end plates – resulting ion current $\propto$ to gas density (pressure!)
  - Gas Type Dependent (Ionization Potential)
    - Accuracy: $\pm 50\%$ f.s (typical)
    - $10^{-3}$ to $10^{-11}$ Torr Operating Range

**Wide Range Gauges**

- Combination Gauges combine complementary technologies to produce Wide Range Gauges
CREATING VACUUM 20 – 0.01 Torr

Oil-Free Roots Pumps
- High durability ‘roots’ pumping mechanism ideal for aggressive gas species
  - Oil free
  - Typically water cooled
  - Typically > 100 m³/hr

Dry Scroll Pumps
- Dual or Single stage reciprocating scroll sets seal without oil!
  - Clean, quiet alternative to oil-sealed RVPs
  - 3 m³/hr to 30 m³/hr pumping speeds
  - Low millitorr ultimate pressure

Oil Sealed Rotary Vane
- Trend towards Large Capacity (> 40 m³/hr) Single Stage pumps to evacuate Interface region (few Torr) AND back Turbos
CREATING VACUUM $10^{-3} – 10^{-8}$ Torr

**Vapor Jet (Diffusion) Pumps**
- High velocity Oil Jet (vapor) from diff-stack strikes gas molecules:
  - Oil mist condenses at (water) cooled body of the pump
  - Cryo-cooled baffles at pump inlet can reduce oil mist from entering process chamber

**Turbo-molecular Pumps**
- High speed (35K+ rpm) blades strike gas molecules directing them towards pump base
  - Molecular Drag stage (ie. TwisTorr stage, shown) compresses gas $10^4 \times$ & create torturous path for light gases
  - Mechanical, hybrid, or mag-lev bearings

**Cryo-Pumps**
- He recirculation system keeps large 1st stage array at 77K (water) & smaller 2nd stage array at < 15K ($N_2$, $Ar$, $CO$, $O_2$)
  - $H_2$, $He$, $Ne$ pumped by charcoal on underside of 2nd stage
  - 800 – 60K l/s pumping speed
  - $10^{-3} – 10^{-10}$ Torr pressure
  - Requires periodic ‘regeneration’
Turbo-Molecular Pump: Blade Section

• How it Works:
  • Large INLET coupled directly to chamber allows highest probability of a particle entering the pump
  • BLADES transfer momentum to particles; STATORS have complementary angle to reflect the particle
  • Blade/Stator ANGLE decreases to prevent back-migration
Turbo-Molecular Pump: Macro-Torr Drag Stage

After particles exit the lowest blade stage (approx. $10^{-3}$ Torr) Spinning Disc transfers momentum to them during residence time on the disc.

- Moving Wall with Speed $V$
- Imparts Forward Momentum
Turbo-Molecular Pump: Molecular Drag Stage

• How it Works:
  • Molecular Drag Stage transfers momentum to particles during residence time on a rotating element and directs the motion in a confined channel
    - Rotating blade design can only compress gas to $\approx 10^{-3}$ Torr (max) & has poor pumping for light gases

![MacroTorr ('Gaede')]  ![TwisTorr ('Siegbahn')]  ![Holweck]

- Stripper Surface
- Disk Rotor Impeller
- Channel
- Gas flow in centripetal and centrifugal direction through TwisTorr channel
- Drum Rotor Impeller
- Helical Groove Channel
Turbo-Molecular Pump: Molecular Drag Stage

• How it Works:
  • TwisTorr® - Agilent’s most advanced Turbo-Drag Stage! The MacroTorr’s ‘stripper surface’ is replaced with stators featuring complex channels to guide and compress gas molecules through centripetal and centrifugal motion.
Turbo-Molecular Pump: Rotor Suspension

**Mechanical Bearing Turbos:**
- Ceramic Balls in SS housing
- Grease or Oil-lubricated
- Pre-load, alignment and shock resistance are keys
- Some designs use 1x Mechanical Bearing and 1x Permanent Magnetic Bearing (‘Hybrid’ suspension)

**Magnetic Levitation Turbos:**
- 4 Separate magnetic bearings support and position the ‘floating’ rotor
- ‘Safety’ or ‘Touch-down’ bearings support rotor during ramp-up and shut-down
- Reduced electronics & computing cost help!
Getting to near-UHV Pressures (Time-of-Flight Section)

Consider the shape of the pumpdown curve

Pressure Decay

- Chamber Free Volume
- Desorption
- Diffusion
- Permeation

Consider ALL potential gas sources

- Permeation
- Desorption
- Diffusion
- Virtual Leaks
- Real Leaks
- Backstreaming

Pressure Decay:

\[ \begin{align*}
10^{+3} & \quad \text{Torr} \\
10^{-0} & \\
10^{-3} & \\
10^{-6} & \\
10^{-9} & \\
\end{align*} \]

Time:

Pressure (Torr):

- \(10^{-9}\)
- \(10^{-6}\)
- \(10^{-3}\)
- \(10^{-0}\)
- \(10^{+3}\)

Materials:

Leaks:

Virtual Leaks

Real Leaks

Backstreaming

Diffusion

Permeation

Desorption

Process

Chamber Free Volume
Challenges in Reaching HV and near-UHV Pressures

Material Selection

• “If you don’t want to pump it out, Don’t put it in!”
• Choose materials based on the following criteria:
  Permeation Rates
  Bake-Out Temperature
  Outgassing Rates
  Chemical Compatibility
  Mechanical Properties
  Electrical Properties

Outgassing & Diffusion

• TEMPERATURE is the single most effective way to increase the outgassing and diffusion rates of components inside the vacuum chamber, allowing our HV pumps to remove the gas (mostly water)
  ➢ Heating (and Cooling!) must be done uniformly
  ➢ Effectiveness is proportional to Time (linear) and Temperature (exponential!)
  ➢ Getting the heat to internal parts a challenge!

• CERN ‘Recipe’ for cleaning parts for UHV:
  https://pdfs.semanticscholar.org/fae5/6ae03f7c841f7aae5462a2631b8bbed529f.pdf
Vacuum System Troubleshooting

Pumpdown Curve

- Slope \( \Delta p/\Delta t \) can help determine if vacuum leak is ‘Real’ (outside-in) or ‘Virtual’ (outgassing or desorption)

Leak-Up Rate

- Slope \( \Delta p/\Delta t \) can help determine if vacuum leak is ‘Real’ (outside-in) or ‘Virtual’ (outgassing or desorption)

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# Helium Leak Detector

## Spray Helium Application

- **Large Vacuum Chambers**
  - HLD connected to SUT (system under test) between High Vacuum and Rough Vacuum Pumps

- **Small Vacuum Chambers**
  - HLD connected directly to chamber: Leak Detection is a Rough Vacuum (Viscous Flow) process

## Sniffing Application

Component or Vacuum System *PRESSURIZED* with He:
- HLD operated in ‘Sniffing’ mode to detect He escaping from the part or chamber
- ‘Bombing’ is a multi-step process where components are pressurized then sniffed

<table>
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<tr>
<th>Method</th>
<th>Min. Detectable Leak (atm. cc/sec)</th>
<th>Notes</th>
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</table>
| Rate of Rise | $\geq 10^{-5}$                    | • Qualitative: Real or Virtual  
  • Time Consuming                                 |
| Radioisotope | $\geq 10^{-10}$                   | • Good for Small Sealed Parts  
  • Special Equipment & RSO, High Cost            |
| He Mass Spec | $\geq 10^{-12}$                   | • Fast, Non-Destructive  
  • No Operator Judgement  
  • Low Equipment Cost                              |
Simple Magnetic Sector Mass Spec Vacuum System

**System Design Features**

- **Turbo Molecular Pump** evacuates Spectrometer tube to $10^{-5}$ Torr Pressures
- **Contra-Flow Design** makes use of Turbo Pump’s (relatively) poor He compression
- **Dual Stage RVP** or **Scroll** improves Foreline compression (prevent He backflow)
- **Single Stage Scroll** can be backed by small Diaphragm Pump to achieve same result
Double Focusing Magnetic Sector Mass Spec Vac System

System Design Features

- **Large Capacity Turbo** or **Dual Stage Primary Pump** required to reduce inlet gas load
- **Dedicated Backing Pump** for Mag Sector Section maximizes compression
- **System layout** complicates move to Multi-Inlet Turbo Pumps

Trends

- Movement to Oil-Free Scroll pumps vs RVPs (contamination)
GC Quadrupole or Ion Trap Mass Spec Vac System Design

**System Design Features**

- Single inlet Turbo Pump (typ. With Drag Stage) backed by Oil-Sealed RVP or Multi-Stage Diaphragm Pump

**Trends**

- Movement to Oil-Free Scroll pumps vs RVPs (contamination) or Diaphragm Pumps (maintenance)
- Fewer Diffusion Pump systems produced
- Improved Vacuum Sub-System Cost & Reliability

In Trapping Mode, ions coalesce towards the center of the trap.
- Ramping voltages selectively ejects Mass Species from the trap.
Traditional Quadrupole LC-MS Vac System Design

System Design Features

- **Dedicated, Dual Stage RVPs** required to ‘back’ non-compound Turbo Pumps
- **28 m³/hr RVP on Interface**: The Speed of Light for Single $f$ Power
- **Large Gas Load** in $10^{-3}$ Torr (Transition Flow) region required large pump inlet sizes
- Vacuum Sub-System Cost
- Vacuum Sub-System Reliability
  - Ceramic Ball Bearing Turbos
  - Optimized Cooling Systems
Modern Quadrupole LC-MS Vac System Design

System Design Features

- **Drag Stage Turbos** eliminate need for dedicated dual-stage RVPs
- **Inverter Driven RVPs** obliterate 28 m³/hr limit on Interface/Backing Pump Size
- **Large Gas Load** in $10^{-3}$ Torr (Transition Flow) region require large pump inlets
- **Large Single Stage RVPs** reduce cost

Trends

- Larger size (or multiple) Single Stage RVP backing Pumps (up to 120 m³/hr!)
- Some movement to Oil-Free Pumps
System Design Features

- **Dual-Inlet Turbo Pump** evacuates two independent regions to different vacuum levels
  - Pump custom designed to match customer gas loads
- Restricting $10^{-3}$ Torr Region inlet has SOME impact on pumping speed (**Transition Flow**)
- Vacuum Sub-System Cost (MS RVPs)

**Trends**

- Insertable Pumps Optimize Conductance & Reduce Cost
- Some movement to Oil-Free Pumps
Triple Inlet Turbo LC-MS Vacuum Sub System

System Design Features

- **TRIPLE Inlet Turbo Pump** has direct inlet to drag stage (0.1 – 0.5 Torr) – allowing higher inlet gas flows

**Trends**

- Insertable Pumps Optimize Conductance
- Some movement to Oil-Free Pumps
Quadrupole Time-of-Flight Vacuum Sub System Design

System Design Features

- **Multi-Pass** TOF designs replacing single- or dual-pass systems (reduces system size; complicates Turbo placement)
- **Dedicated Turbo Pump** for TOF Section

**Trends**

- Other vacuum regions used to ‘back’ TOF section Turbo (maximize compression/ negligible gas flow)
- Movement to **Oil-Free Pumps**
Summary

• **Vacuum Measurement**
  - Balance between system cost & precision required
  - Reducing #gauges impacts diagnostic capability

• **Pump Technology**
  - Single Stage RVPs and Compound Turbo Pumps dominate
  - Move to Oil-Free Scroll Pumps driven by sample integrity and maintenance simplicity
  - Lower vacuum pressures (near-UHV) prompt some use of ION PUMPS

• **Vacuum Sub-System Design**
  - “Bespoke” Multi-Inlet Turbos represent the ultimate in optimization but limit vendor selection (especially with insertable ‘cartridge style’ pumps)
  - Vacuum vendors need to be seen at technology partners vs commodity providers
Agilent Vacuum Education Programs

To learn about more Agilent Vacuum Technology Education programs, including:
- Basic Vacuum Practice and Introduction to Leak Detection classes
- On-site Vacuum Technology Seminars
- Custom multi-day classes at your site

…please contact Agilent Vacuum Customer Care (800-882-7426 – Option 3) or e-mail Robin Arons (robin.arons@agilent.com).

Click HERE to access Agilent’s excellent video series on Care and Use of Turbo Pumps.

Click HERE for more information on Helium Leak Detection, including details on Agilent’s new HLD instrument.