# Vacuum Technology
## for Mass Spec Instruments

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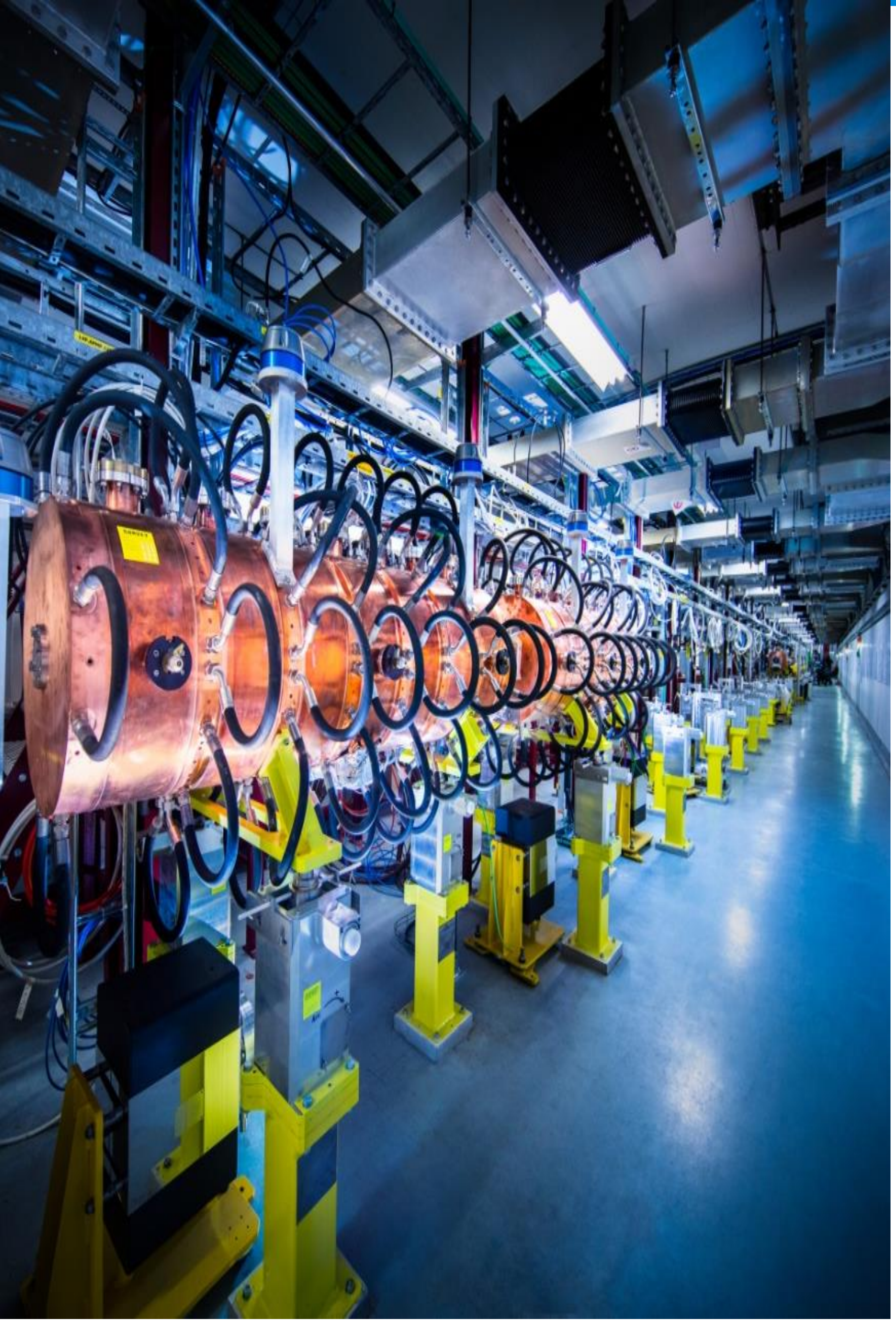


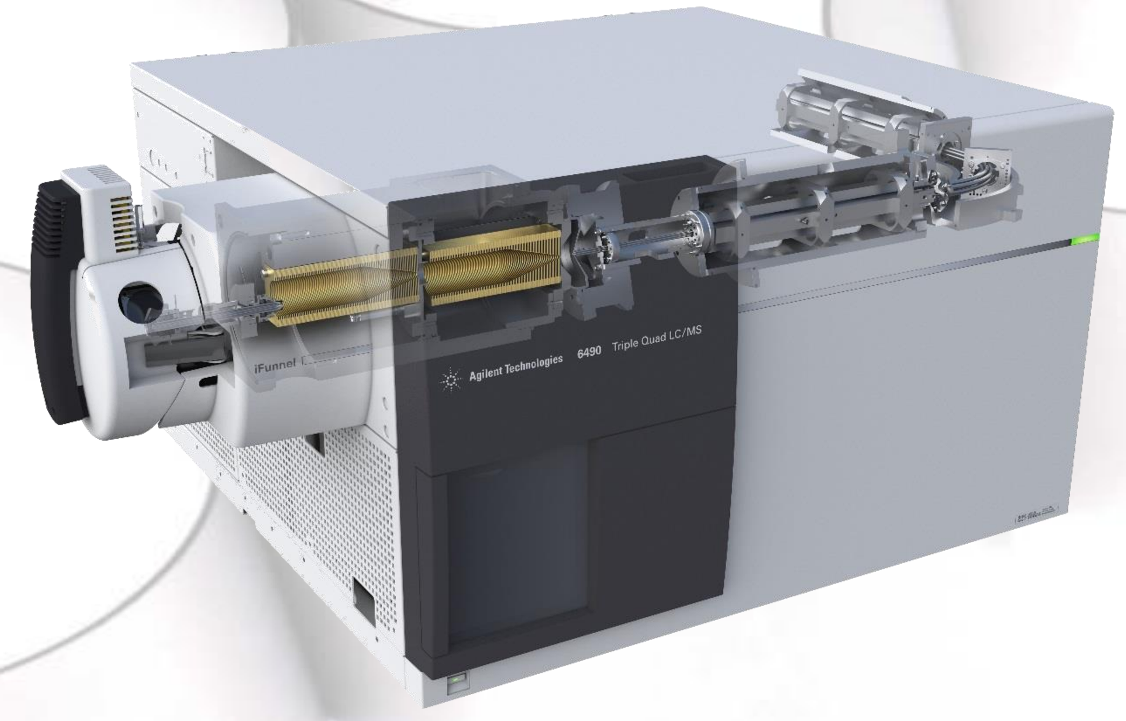
# Vacuum For Mass Spectrometry

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



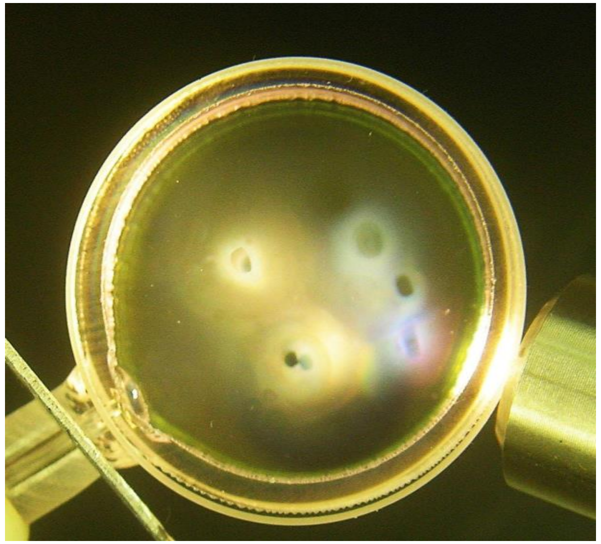
# Why We Need Vacuum for Mass Spectrometry

## 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

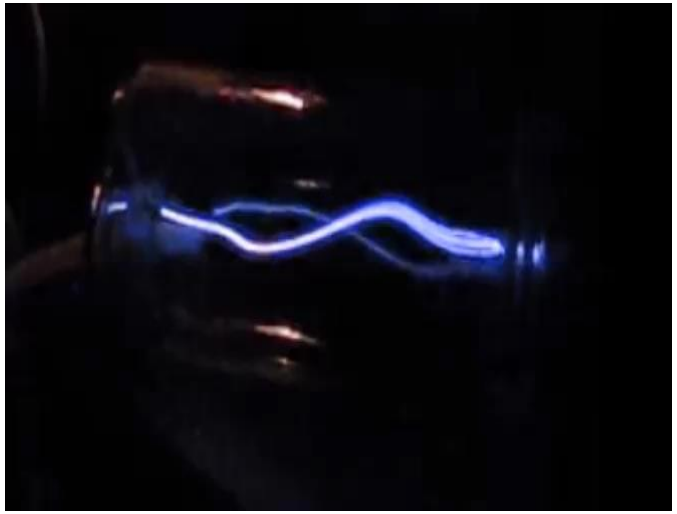
## Remove Chemical Contamination

- 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 ‘arcing’
  - ➢ Electrostatic Lenses (-250 ⟶ +250 DC)
  - ➢ Quadrupoles (-4K ⟶ +4K RF + DC)
  - ➢ Ion Detectors (-7K DC ⟶ +7K DC)



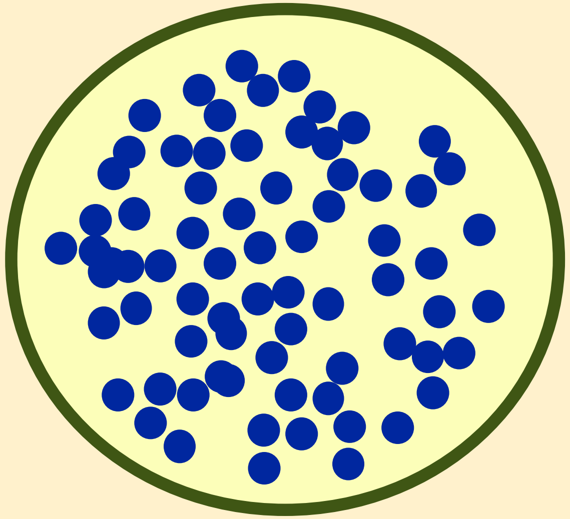
# Rough, High and Ultra-High Vacuum

**Ultra-High Vacuum**

**High Vacuum**

**Rough Vacuum**

**Molecular Flow**



**Viscous Flow**

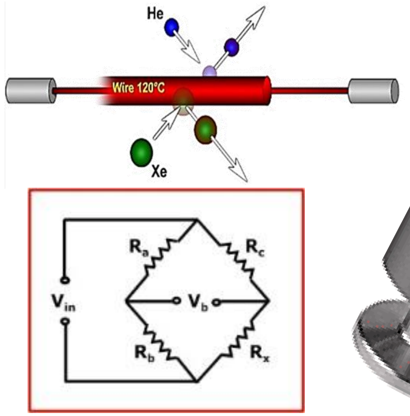
# MEASURING ROUGH VACUUM: Atm - $10^{-3}$ Torr

## Thermocouple Gauges

- **CONVECTION**: Maintain filament at constant T (above ambient)
  - Pressure ∝ Current
  - Atm → < 1 x 10-3 Torr
- **THERMOCOUPLE**: Maintain filament at constant CURRENT
  - P ∝ T
  - Slow response time; non linear above ≈ 2 Torr

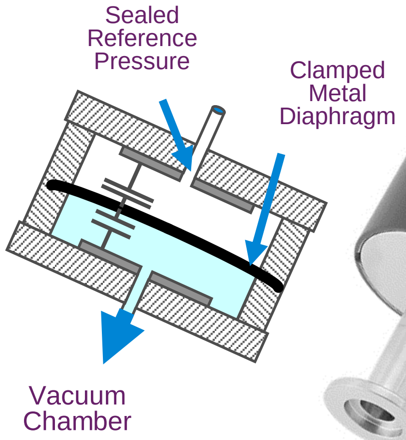
## Thermal (Pirani) Gauge

- **Energy (temperature) loss from a heated filament upsets the balance of a Wheatstone Bridge (typ) circuit**
  - ➢ Pressure ∝ 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 ∝ 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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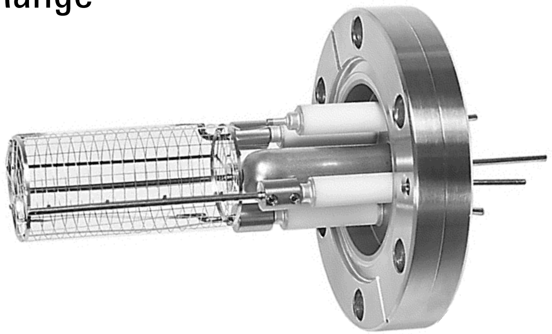
# MEASURING HIGH VACUUM: $10^{-3}$ – $10^{-8}$ Torr

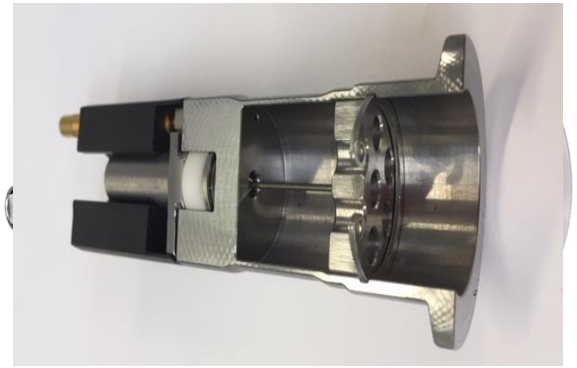
## Hot Ionization (BA) Gauge

- 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: ± 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: ± 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

*FRG-720*
*Full Range*

*PCG-750 Pirani/*
*Capacitance*

https://cds.cern.ch/record/455555/files/p75.pdf

# 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 $m^3$/hr to 30$m^3$/hr pumping speeds
  - Low millitorr ultimate pressure

## Oil Sealed Rotary Vane

- Trend towards Large Capacity (> 40 $m^3$/hr) Single Stage pumps to evacuate Interface region (few Torr) AND back Turbos

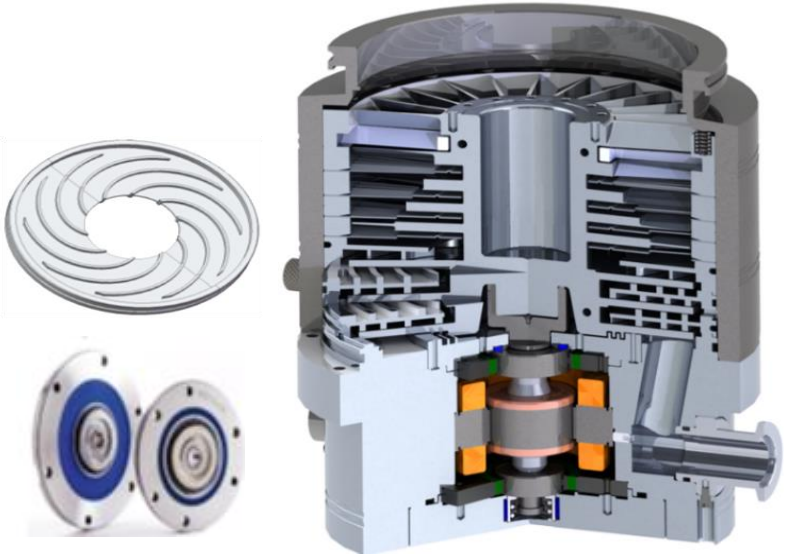
# CREATING VACUUM $10^{-3} – 10^{-8}$ Torr

## 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

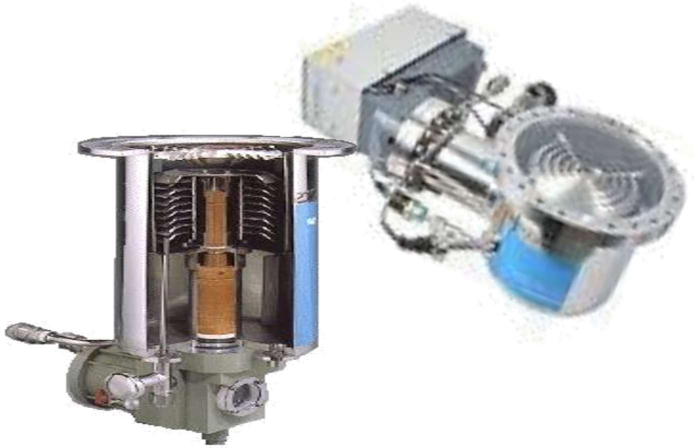
## Turbo-molecular Pumps

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



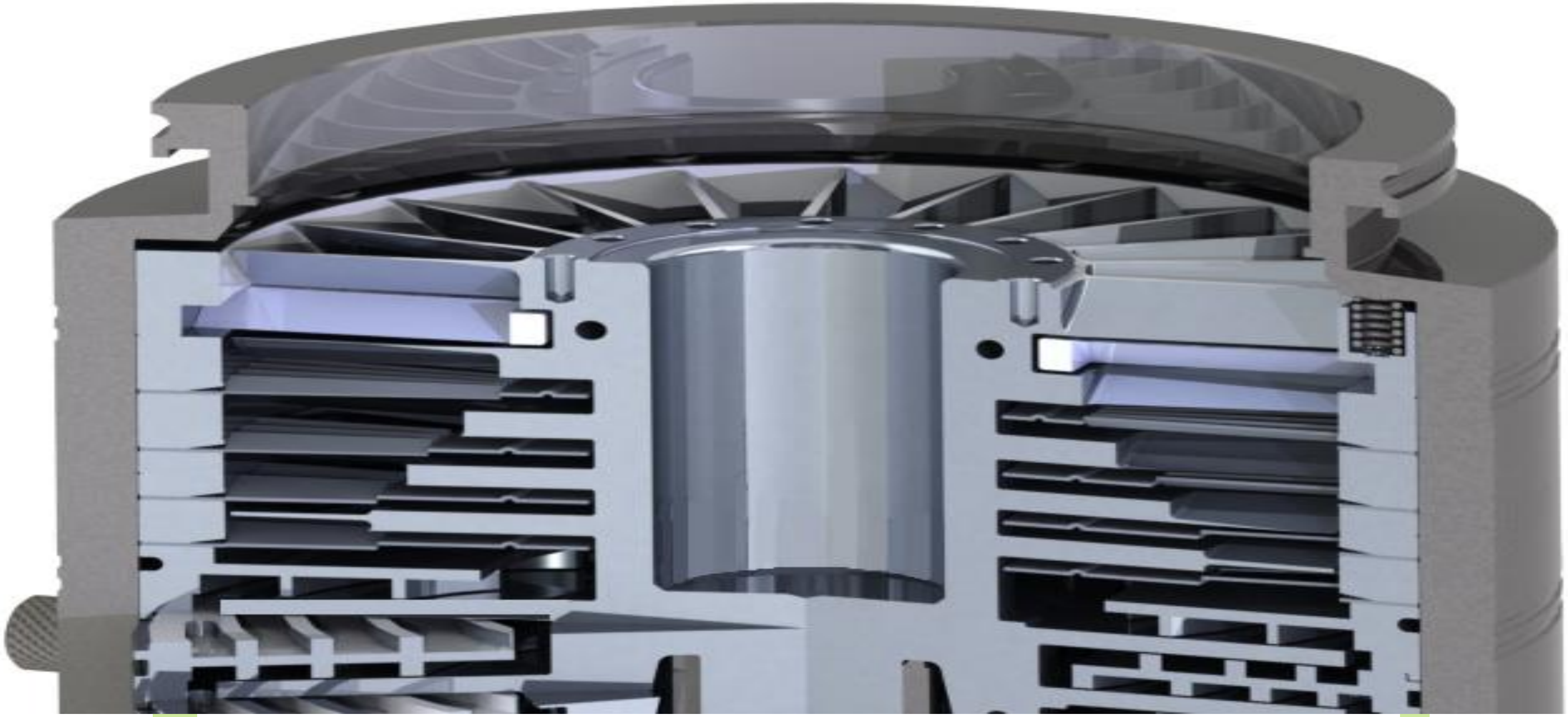
## Cryo-Pumps

- 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’



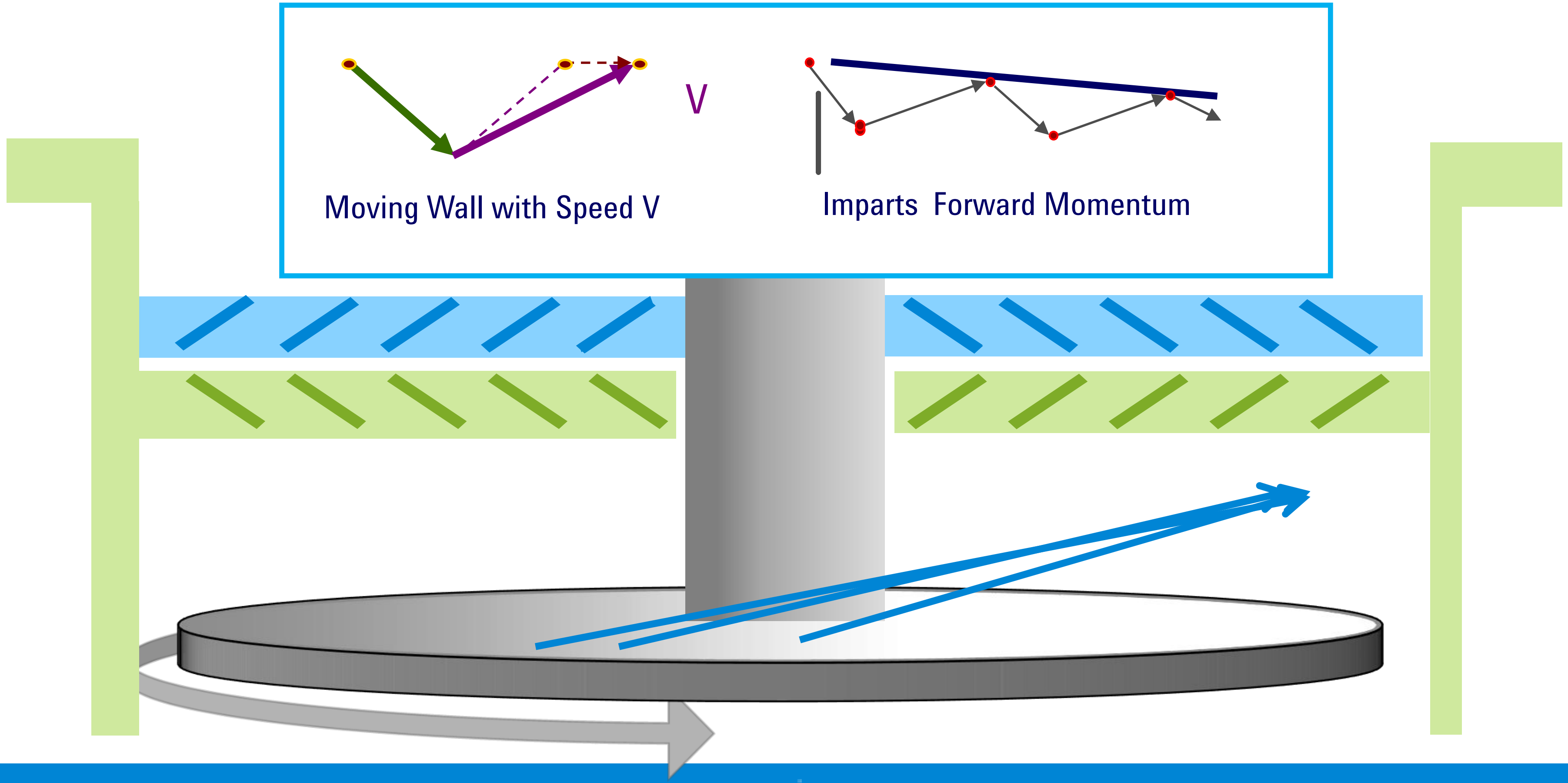
# Turbo-Molecular Pump: Blade Section

- 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

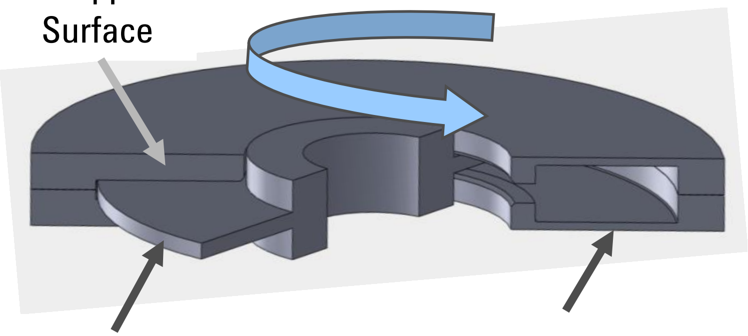
After particles exit the lowest blade stage (approx. $10^{-3}$ Torr) Spinning Disc transfers momentum to them during residence time on the disc



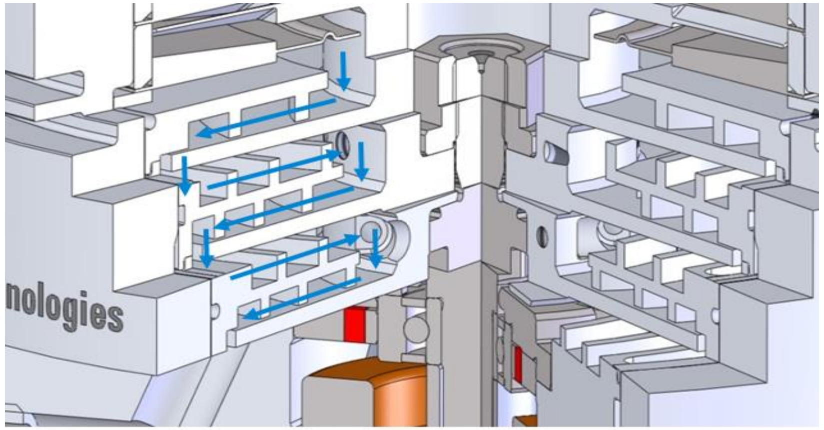
# Turbo-Molecular Pump: Molecular Drag Stage

- 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 ≈ $10^{-3}$ Torr (max) & has poor pumping for light gases

**MacroTorr (‘Gaede’)**

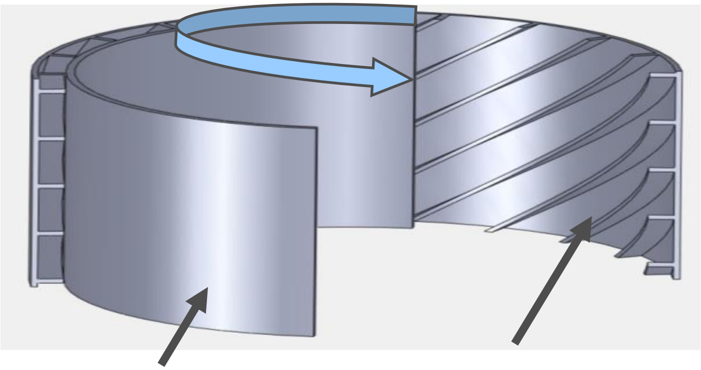


**TwisTorr (‘Siegbahn’)**



Gas flow in centripetal and centrifugal direction through TwisTorr channel

**Holweck**



# 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.

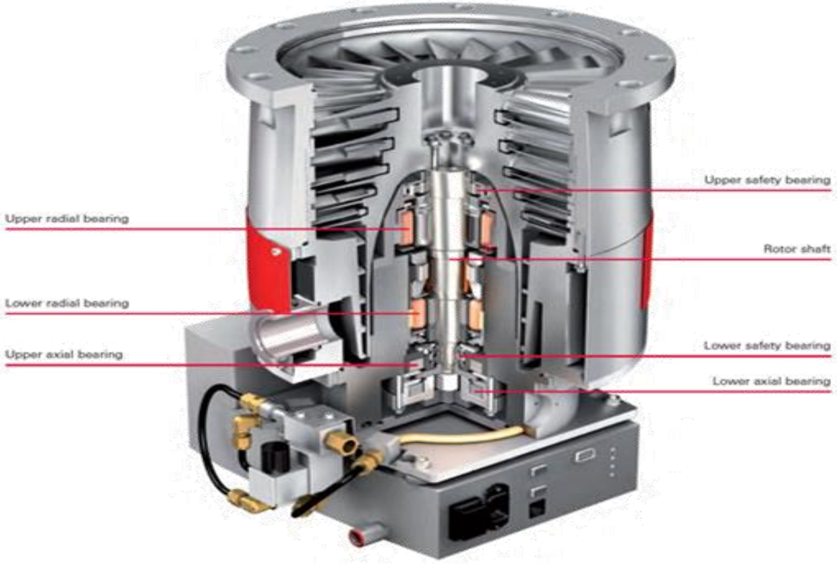
# Turbo-Molecular Pump:  Rotor Suspension

**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)

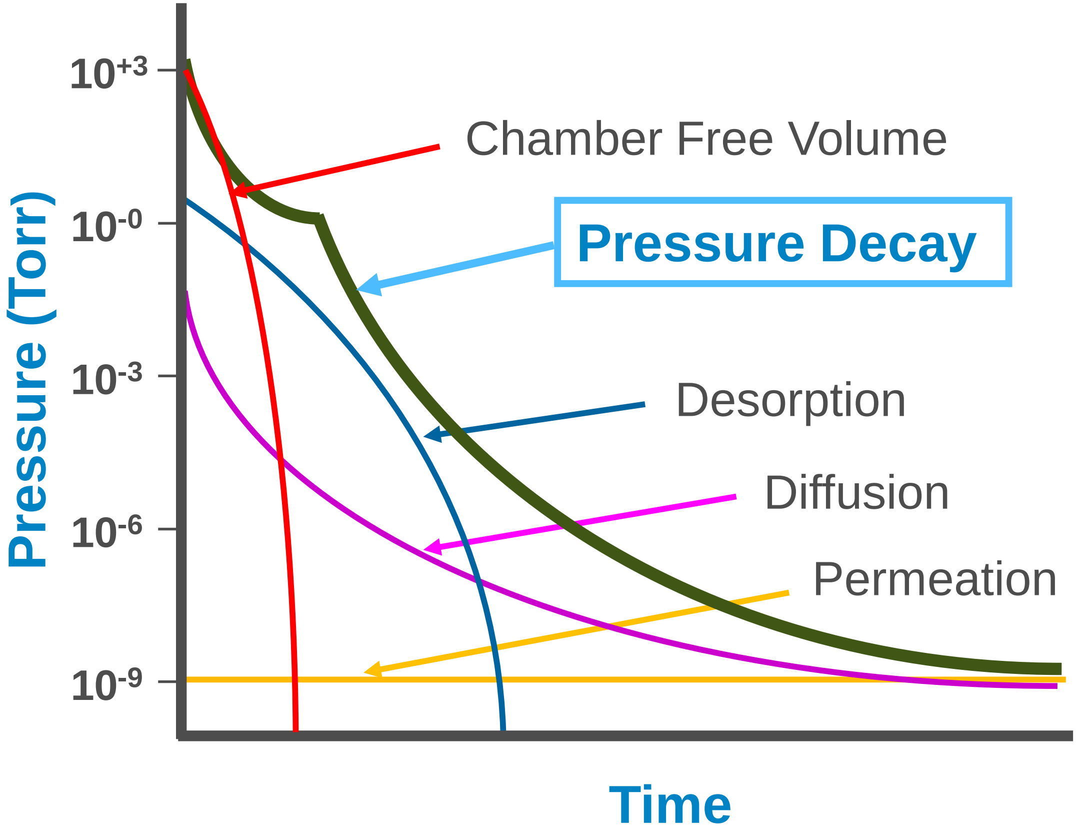
**Magnetic Levitation Turbos:**

- 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!

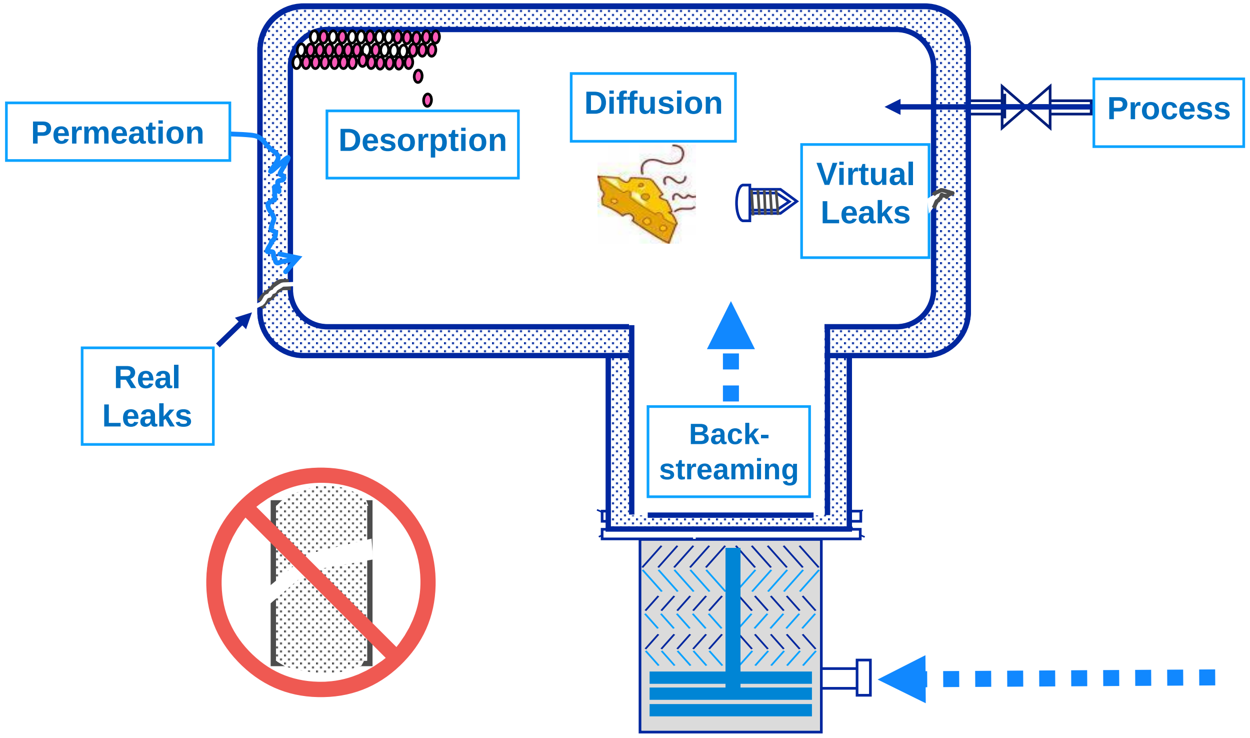


# Getting to near-UHV Pressures (Time-of-Flight Section)

## Consider the shape of the pumpdown curve



## Consider ALL potential gas sources



# 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**

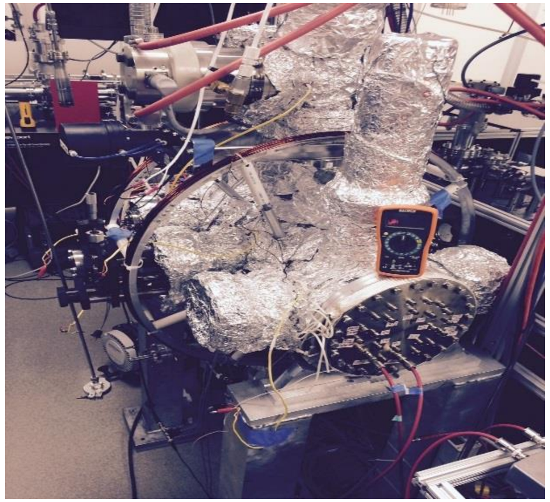
  **Electrical Properties**

- CERN 'Recipe' for cleaning parts for UHV:

https://pdfs.semanticscholar.org/fae5/6ae03f7c841f7aae5462a2631bbbbeed529f.pdf

## Outgassing & Diffusion

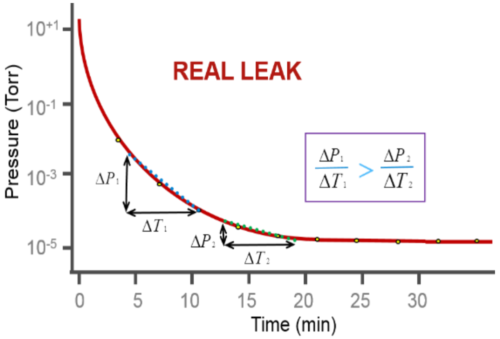
- TEMPERATURE is the single most effective way to increase the outgassing and diffusion rates of components inside the vacuum chamber, allowing our HV pumps to remove the gas (mostly water)
  - ➢ Heating (and Cooling!) must be done uniformly
  - ➢ Effectiveness is proportional to Time (linear) and Temperature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

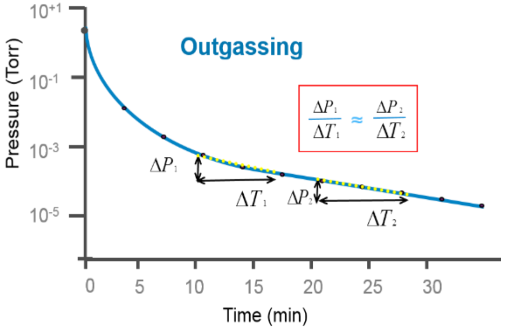


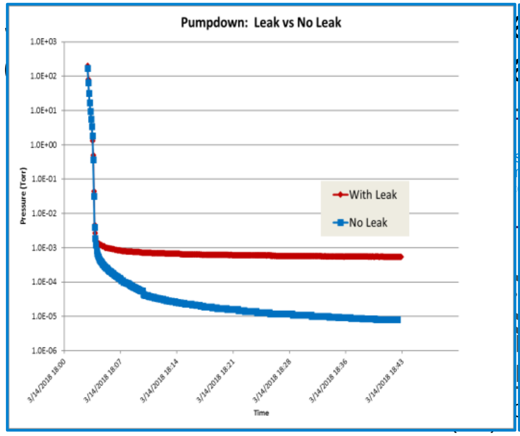
# Vacuum System Troubleshooting

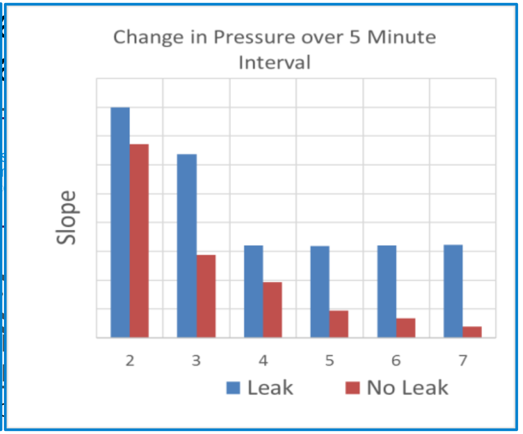
## Pumpdown Curve

- Slope (Δp/Δt) can help determine if vacuum leak is ‘**Real**’ (outside-in) or ‘**Virtual**’ (outgassing or desorption)





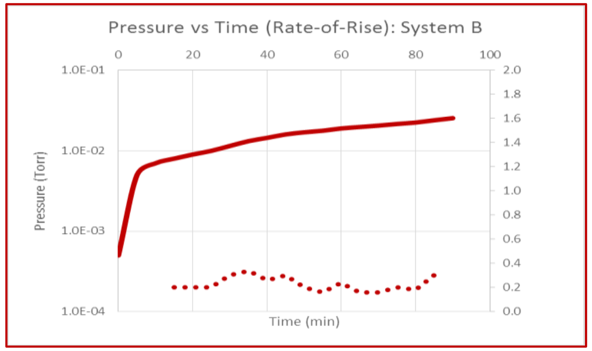


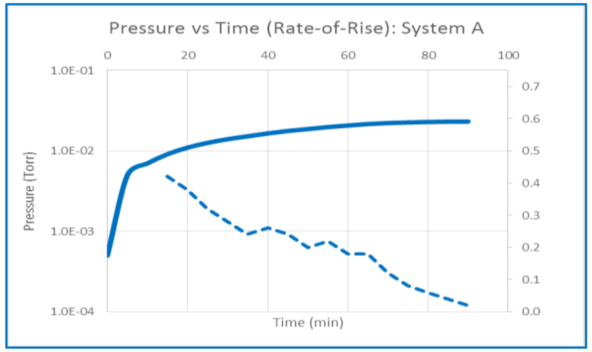


## Leak-Up Rate

- Slope (Δp/Δt can help determine if vacuum leak is ‘**Real**’ (outside-in) or ‘**Virtual**’ (outgassing or desorption)

| Time | Pressure (System A) | Rate of Rise | Pressure (System B) | Rate of Rise |
|---|---|---|---|---|
| 0 | 5.00E-04 | | 5.00E-04 | |
| 5 | 5.00E-03 | | 5.00E-03 | |
| 10 | 7.00E-03 | Virtual | 7.00E-03 | Real |
| 15 | 9.10E-03 | 0.42 | 8.00E-03 | 0.20 |
| 20 | 1.10E-02 | 0.38 | 9.00E-03 | 0.20 |
| 25 | 1.26E-02 | 0.32 | 1.00E-02 | 0.20 |
| 30 | 1.40E-02 | 0.28 | 1.15E-02 | 0.30 |
| 35 | 1.52E-02 | 0.24 | 1.32E-02 | 0.34 |
| 40 | 1.65E-02 | 0.26 | 1.45E-02 | 0.26 |
| 45 | 1.77E-02 | 0.24 | 1.60E-02 | 0.30 |
| 50 | 1.87E-02 | 0.20 | 1.70E-02 | 0.20 |
| 55 | 1.98E-02 | 0.22 | 1.78E-02 | 0.16 |
| 60 | 2.07E-02 | 0.18 | 1.90E-02 | 0.24 |
| 65 | 2.16E-02 | 0.18 | 1.98E-02 | 0.16 |
| 70 | 2.22E-02 | 0.12 | 2.06E-02 | 0.16 |
| 75 | 2.26E-02 | 0.08 | 2.16E-02 | 0.20 |
| 80 | 2.29E-02 | 0.06 | 2.25E-02 | 0.18 |
| 85 | 2.31E-02 | 0.04 | 2.40E-02 | 0.30 |
| 90 | 2.32E-02 | 0.02 | 2.55E-02 | 0.30 |





# Helium Leak Detector

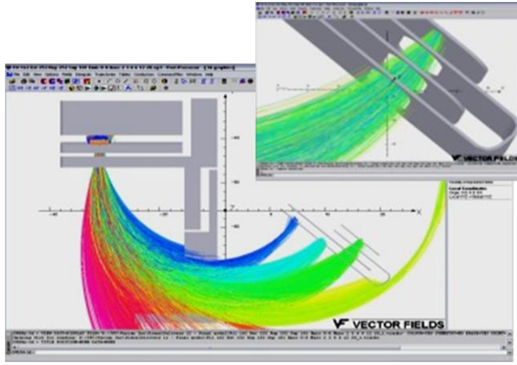
| Method | Min. Detectable Leak (atm. cc/sec) | Notes |
|---|---|---|
| Rate of Rise | $\geq 10^{-5}$ | • Qualitative: Real or Virtual<br>• Time Consuming |
| Radioisotope | $\geq 10^{-10}$ | • Good for Small Sealed Parts<br>• Special Equipment & RSO,<br>• High Cost |
| He Mass Spec | $\geq 10^{-12}$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

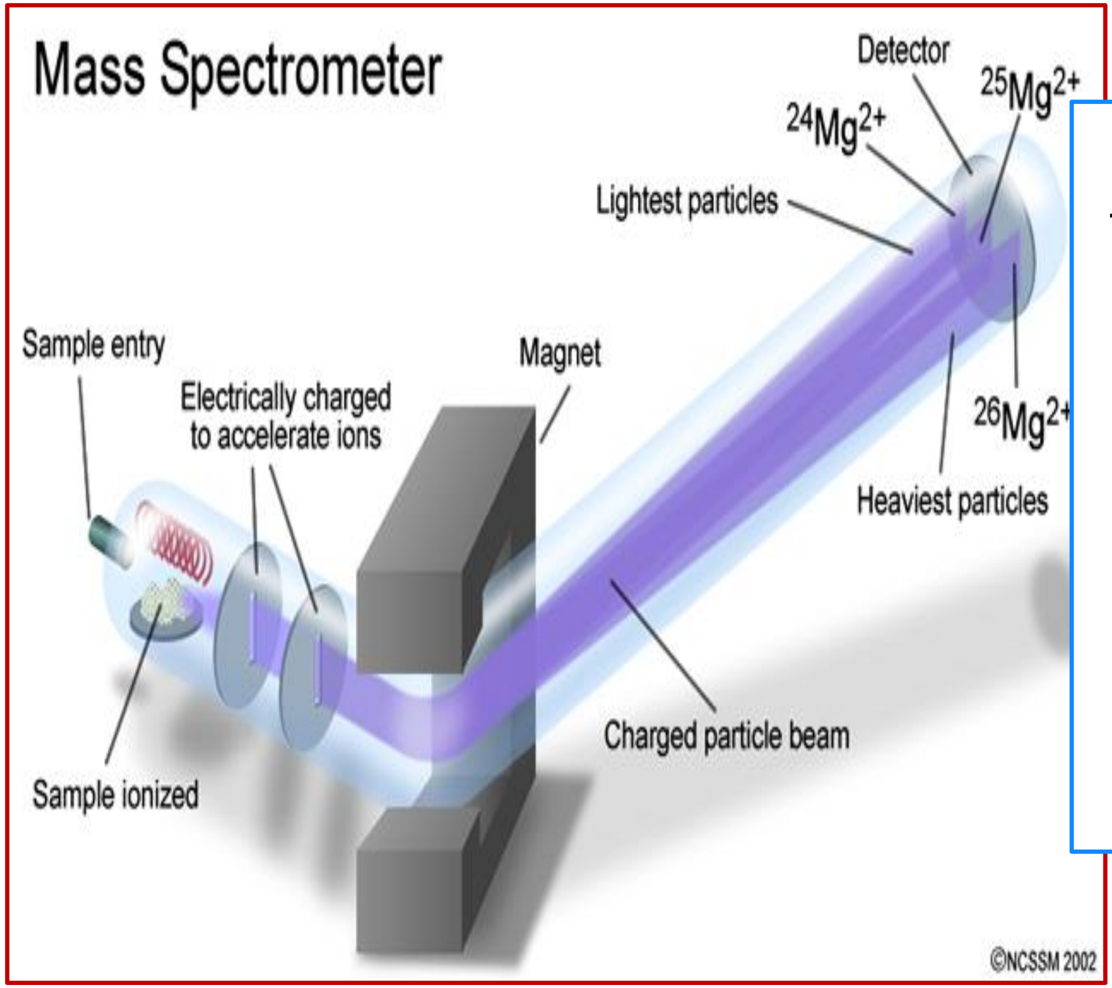
## Sniffing Application

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

# Simple Magnetic Sector Mass Spec Vacuum System

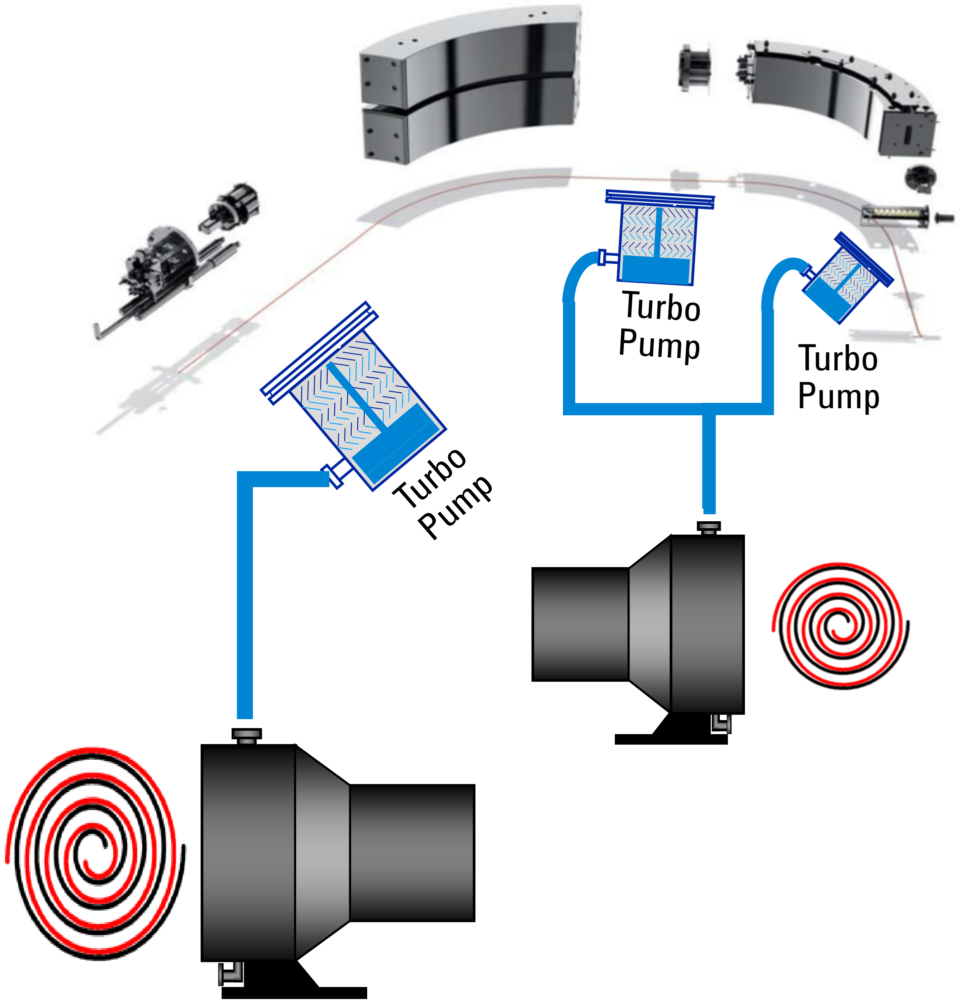




## System Design Features

- **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



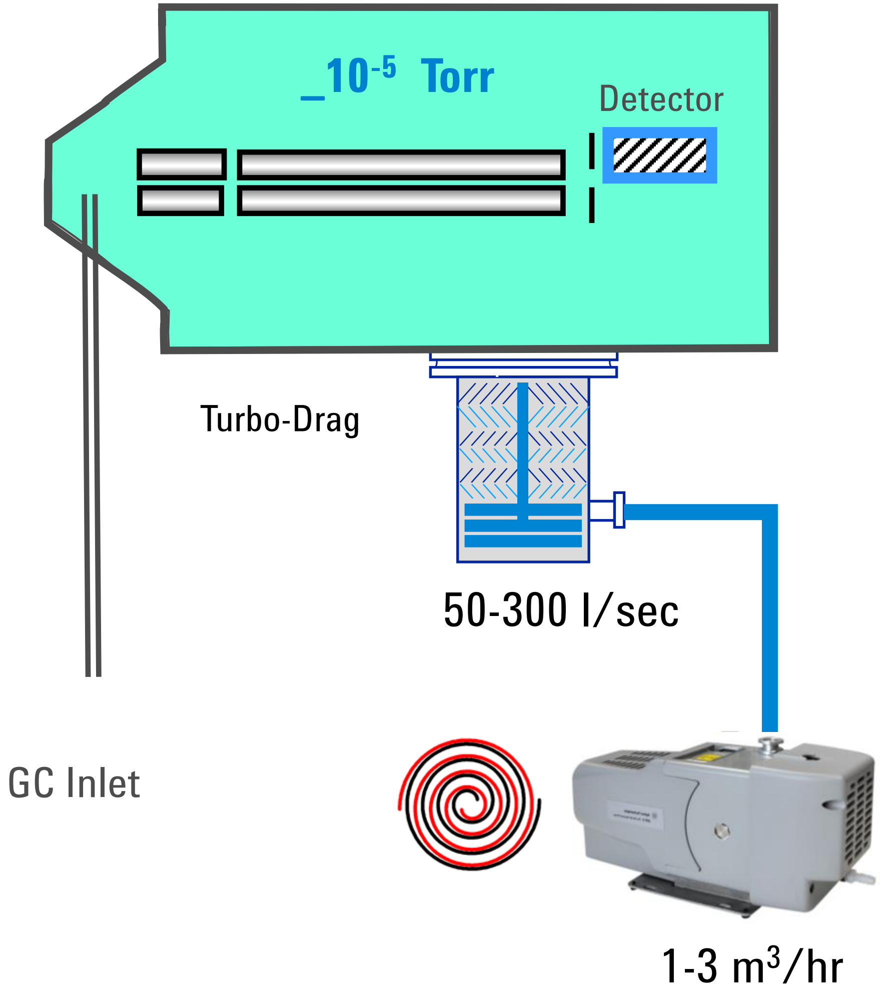
## System Design Features

- **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

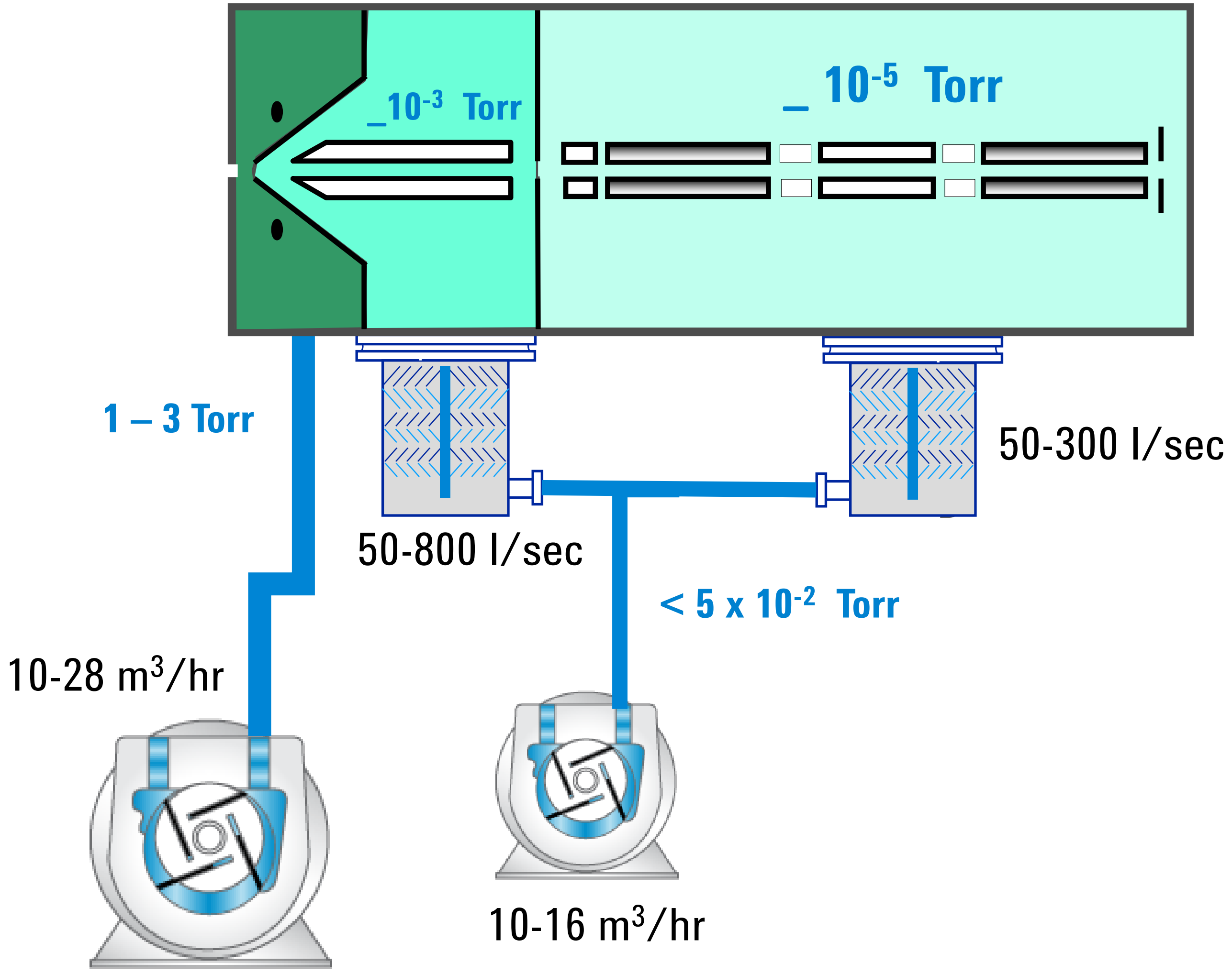
- Single inlet Turbo Pump (typ. With Drag Stage) backed by Oil-Sealed RVP or Multi-Stage Diaphragm Pump

## 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

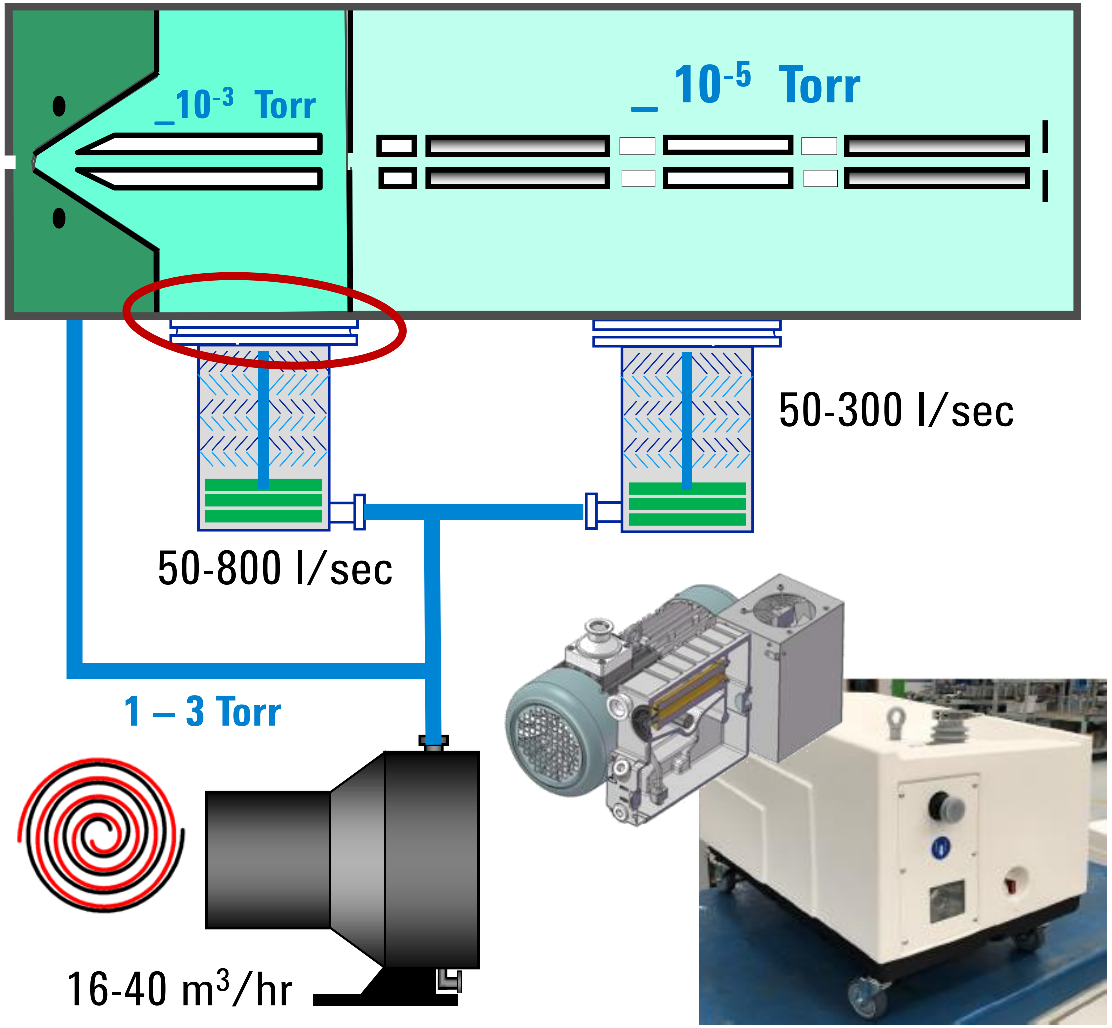
# Traditional Quadrupole LC-MS Vac System Design



## 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



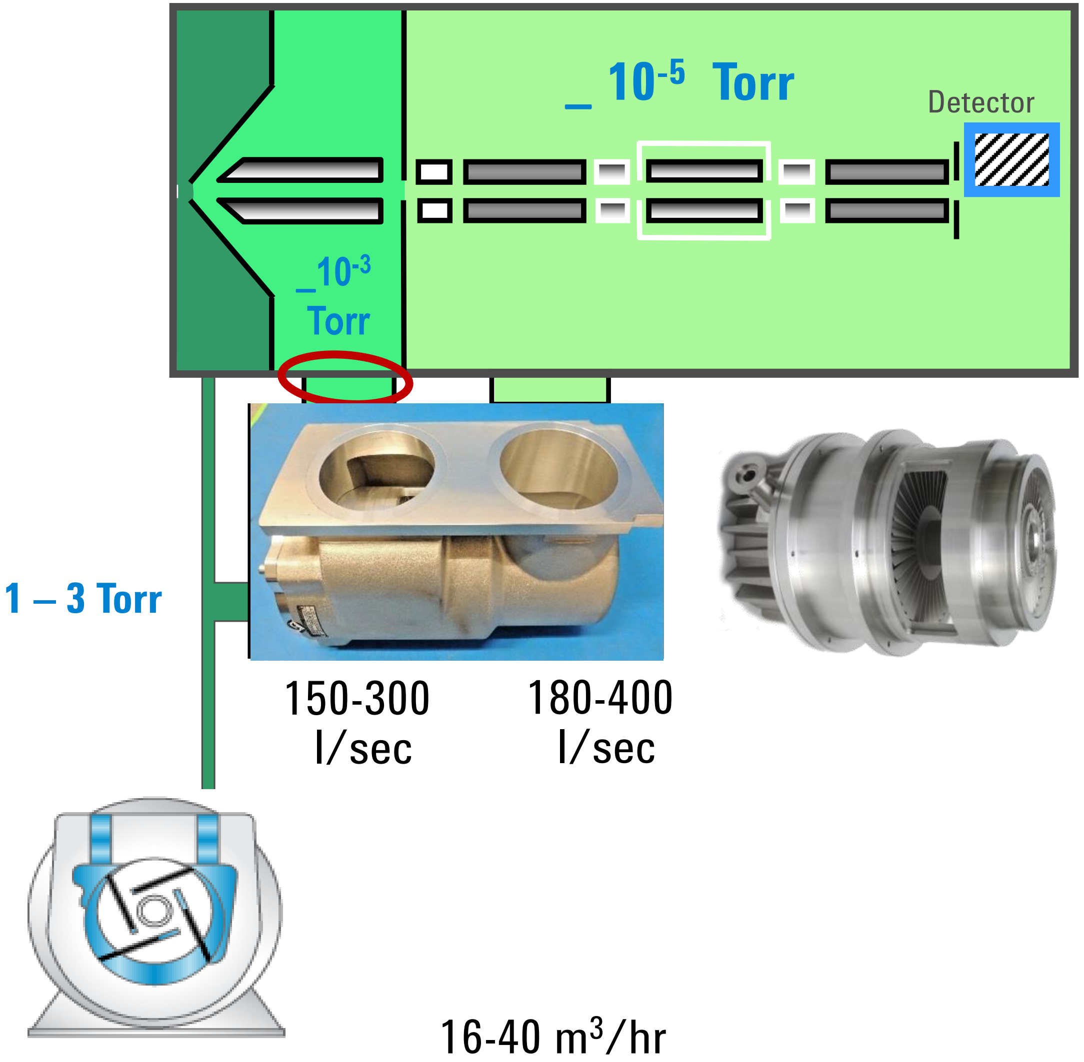
## System Design Features

- **Drag Stage Turbos** eliminate need for dedicated dual-stage RVPs
- **Inverter Driven RVPs** obliterate 28 $m^3$/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 $m^3$/hr!)
- Some movement to Oil-Free Pumps

# Optimized Quadrupole LC Mass Spec Vacuum System Design



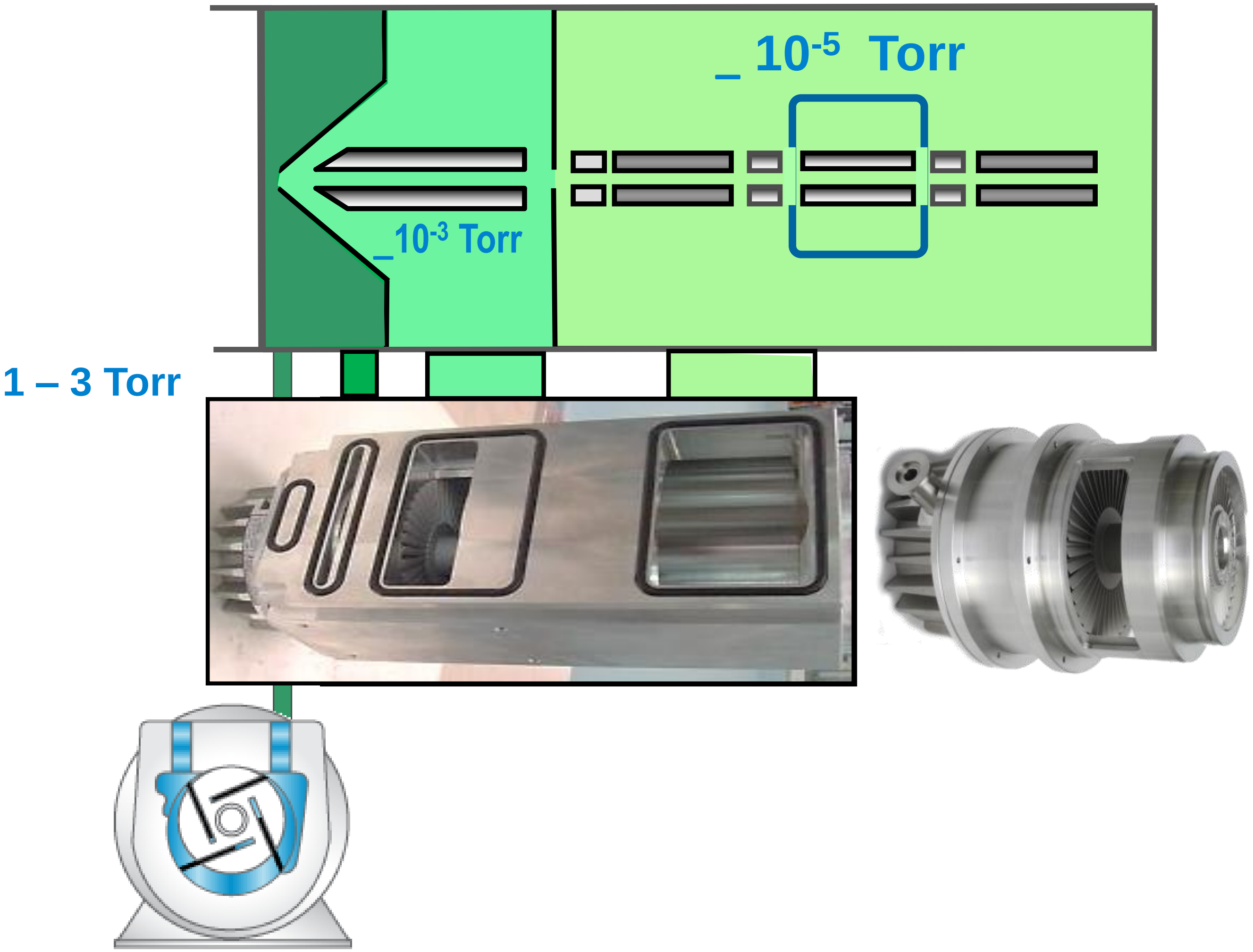
## System Design Features

- **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



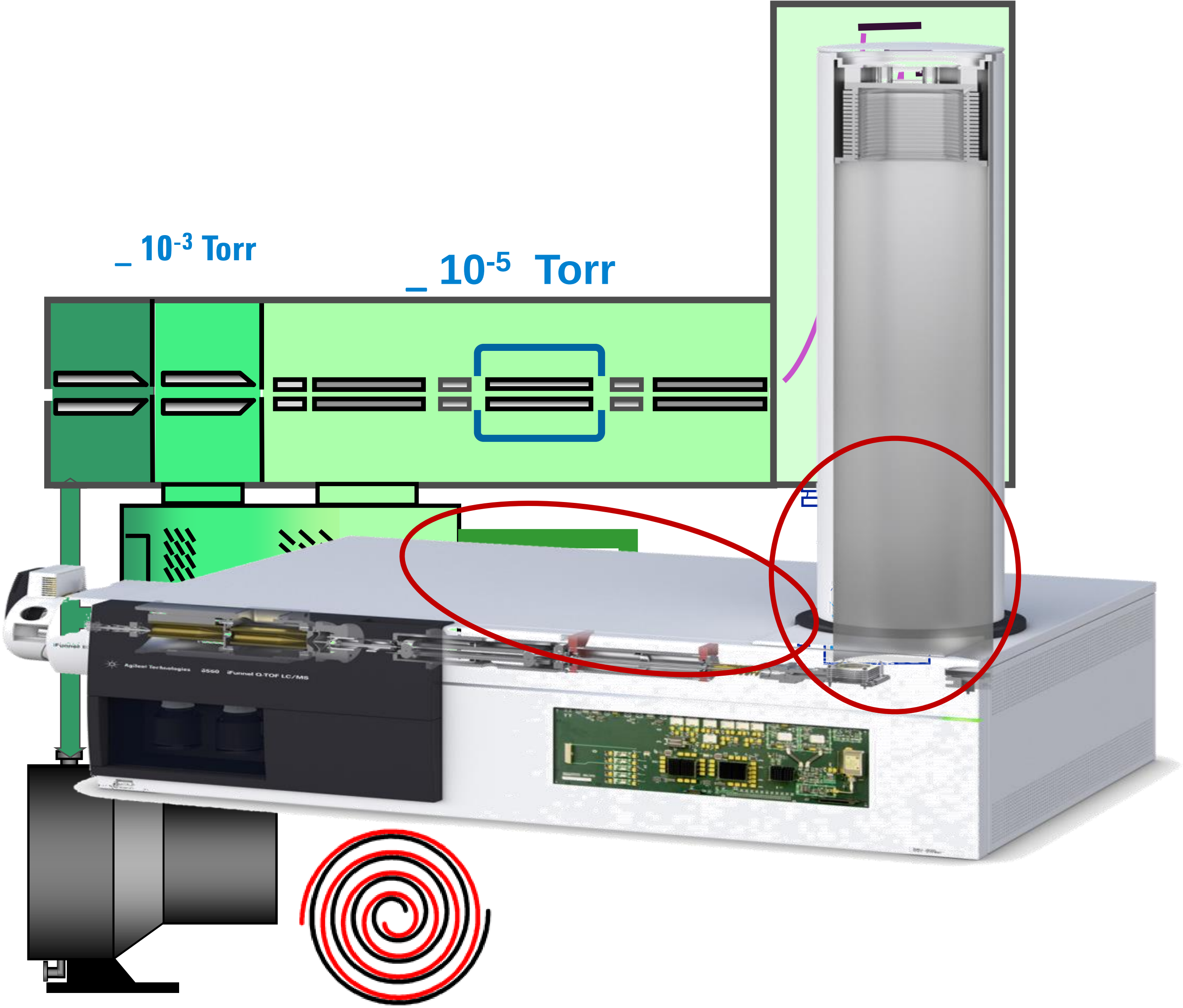
## System Design Features

- **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



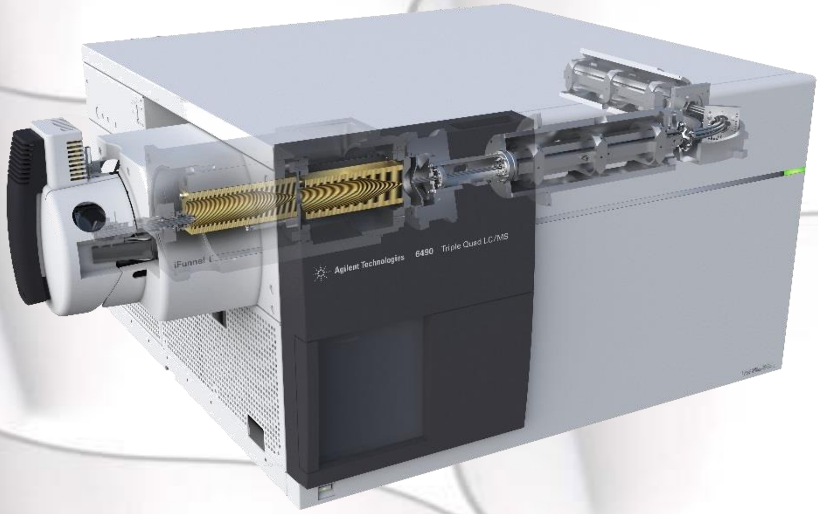
## System Design Features

- **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



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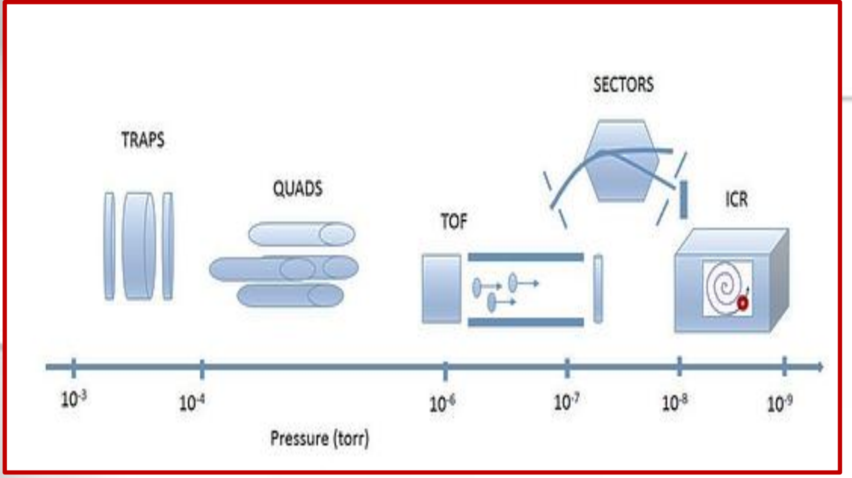

- **Vacuum Measurement**
  - Balance between system cost & precision required
  - Reducing #gauges impacts diagnostic capability
- **Pump Technology**
  - Single Stage RVPs and Compound Turbo Pumps dominate
  - Move to Oil-Free Scroll Pumps driven by sample integrity and maintenance simplicity
  - Lower 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.