

The US Particle Accelerator School Vacuum Testing and Leak Detection

Lou Bertolini Lawrence Livermore National Laboratory June 10-14, 2002



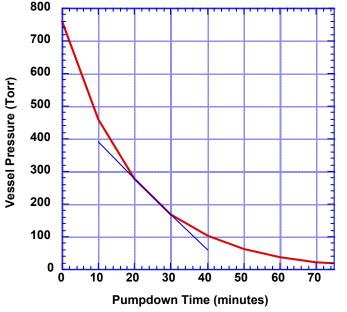
There are four common methods of measuring the speed of pumps:

- 1. Rate of Pumpdown method
- 2. Single Gauge Dome method
- 3. Three Gauge Dome method
- 4. Fischer Mommsen Dome method



- Measure the rate in which a pump evacuates a vessel
- This method is normally used to measure the speed of roughing pumps

$$Q = \frac{d(VP)}{dt} = P \frac{dV}{dt} = V \frac{dP(t)}{dt}$$
$$Q = V \frac{dP(t)}{dt}$$



Since
$$C_{t} \gg S$$
, $P \sim P_{pump}$:
 $Q_{v} \sim SP(t)$
 $S = S_{max} \left(1 - \frac{P_{B}}{P}\right)$
 $S_{max} \left(1 - \frac{P_{B}}{P}\right)P(t) = V \frac{dP(t)}{dt}$
Solving for P(t),

$$P(\mathbf{t}) = P_{B} + P_{o}e^{-\left(\frac{s}{V}\right)t}$$

Single Gauge Dome Method

- Used for many years to measure diffusion pump speeds
- Pump throughput is determined by measuring dP/ dt of a known volume

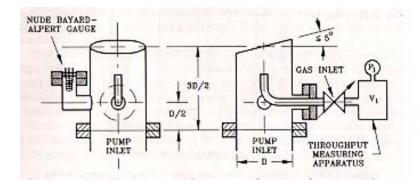
$$\mathbf{Q}_{\mathsf{d}} = \left(\frac{\mathbf{d}\mathbf{P}}{\mathbf{d}\mathbf{f}}\right)\mathbf{V}$$

• Pump speed is determined by assuming the chamber pressure is the same as the pump pressure

$$S = rac{V_d}{P_d} \times rac{dP}{dt}$$

 \cdot Requirements for this test method

-known volume





Three Gage Method

Pump throughput is determined by measuring the pressure difference along a tube of known conductance

$$Q_p = C_1 (P_1 - P_2)$$

We can either assume that the pressure of the pump is equal to P_3 or we can calculate the conductance between the pump and P_3

$$C_{3} = 12.1 \frac{(D_{2})^{3}}{L_{3}}$$
$$Q_{p} = C_{3}(P_{3} - P_{p}) = C_{1}(P_{1} - P_{2})$$
$$P_{p} = P_{3} - \frac{C_{1}}{C_{3}}(P_{1} - P_{2})$$

The pump speed is ultimately determine by the equation:

$$S_{p} = \frac{C_{3}C_{1}(P_{1} - P_{2})}{C_{3}P_{3} - C_{1}(P_{1} - P_{2})}$$

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- Calculated conductances introduce errors
- The three pressure gages must be "normalized" with respect to each other

Typically,

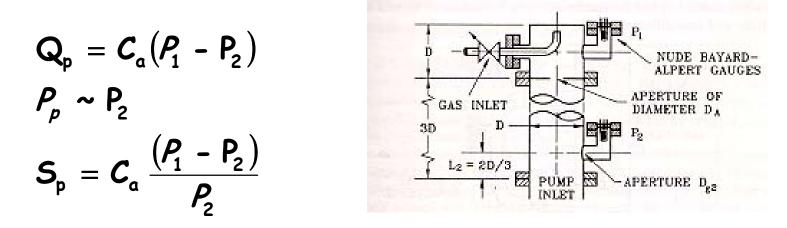
$$P_{abs} \neq P_{1i} + P_{2i} + P_{3i}$$

Normalized,

$$P_{1i} = k_2 P_{2i} + k_3 P_{3i}$$



- \cdot Also known as the CERN method
- The aperture diameter is sized to maintain a minimum pressure differential. This requires some knowledge of the pump speed.



Pressure gages need to be "normalized" to each other.

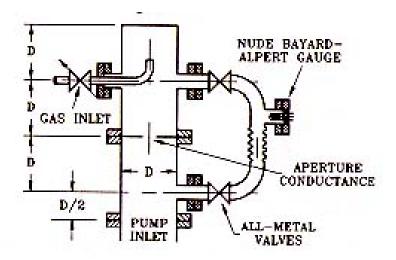
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Modified CERN Method



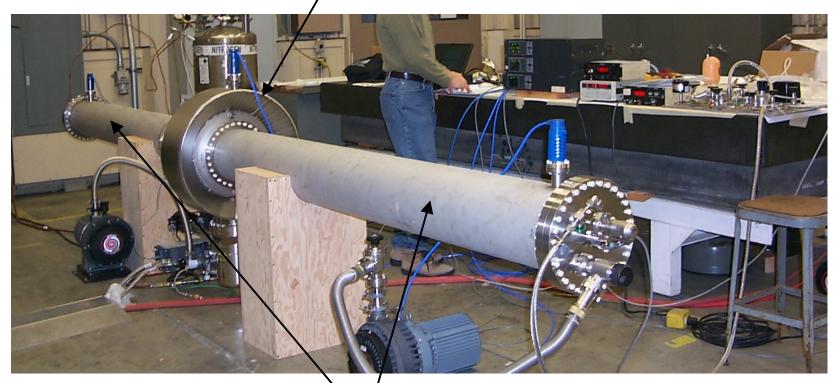
- Utilizes a single gauge.
- By opening and closing the isolation values, both P_1 and P_2 can be determined with the single gauge.
- Gauge normalization is eliminated (assuming gauge linearity with pressure).



DARHT II Accelerator Intercell Pump Speed Test



/Intercell Pump Station



Tubes mock-up the Accelerator Bore

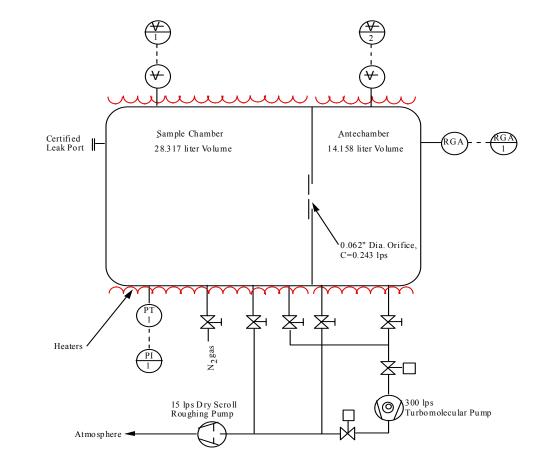


There are two approaches to conducting outgassing tests:

- 1. Measure rates of representative material samples within a test stand (such as an AVS dome).
- 2. Measure rates of actual vacuum system components (such as whole beam pipes, collimators, beam dumps, etc.).

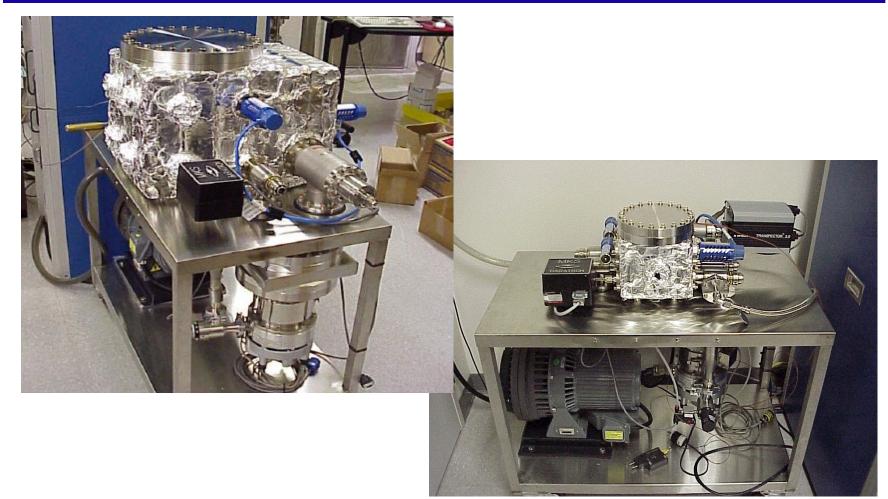


LLNL Outgassing Station Schematic



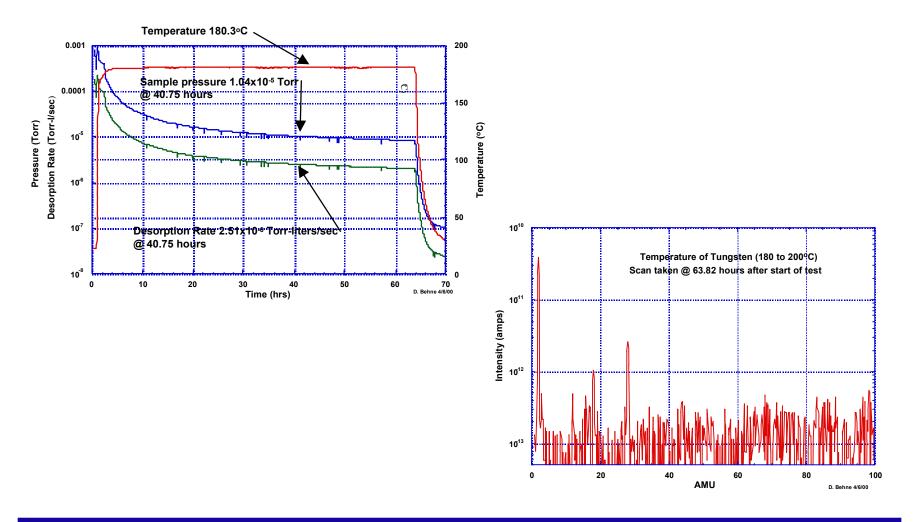


Photos of LLNL Outgassing Stations



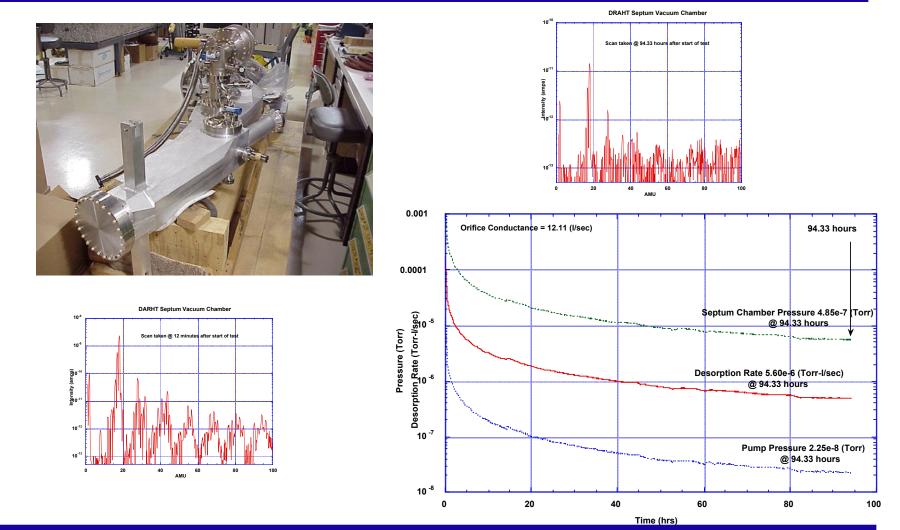


Sample Results from an Outgassing Test

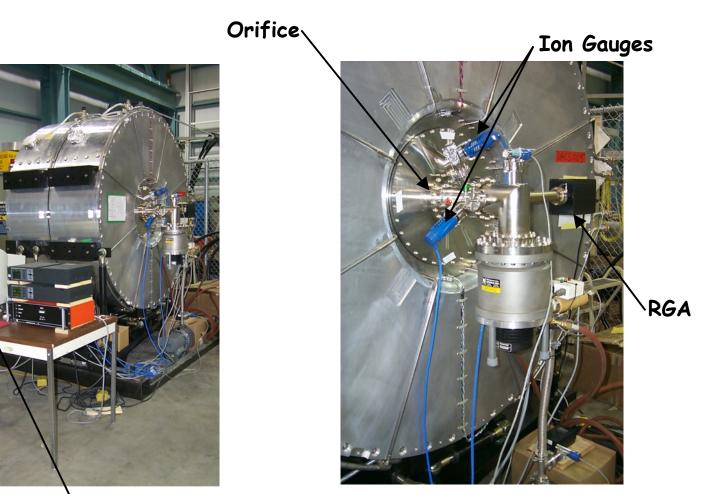




DARHT II Septum Chamber Test Results



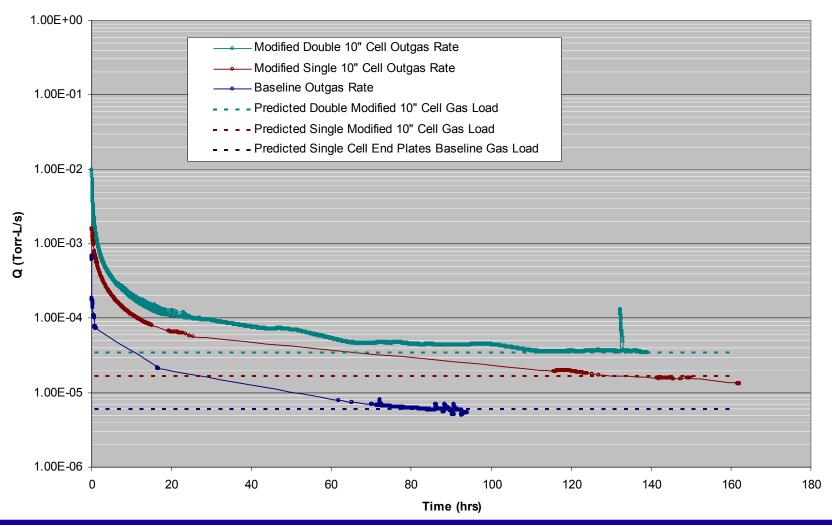
DARHT II Accelerator Cell Outgassing Tests



Data Acquisition

Data from DARHT II Accelerator Cell Outgassing Tests

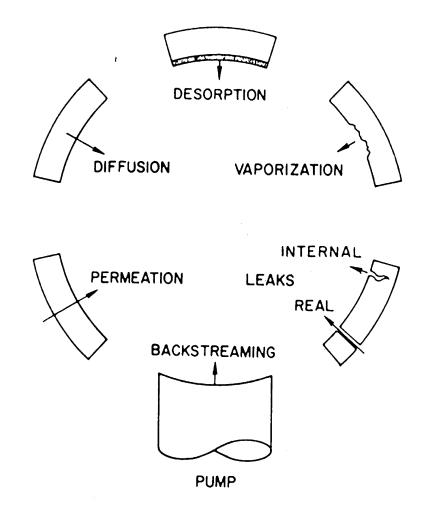




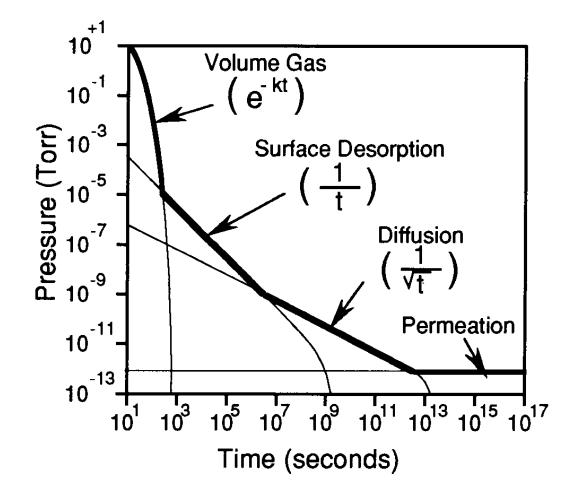
- The Mike Benapfl "motherhood" statement.
 "Leak Detection is an art. <u>Not everyone is an artist!"</u>
- Leak Detection should be performed in accordance with some ASTM Standard.
 - ASTM E493-94 ASTM E498-94
 - ASIM E498-94
 - ASTM E499-97
- \cdot Leak Detection should be performed in a series of logical steps.



Sources of Gases in a Vacuum System

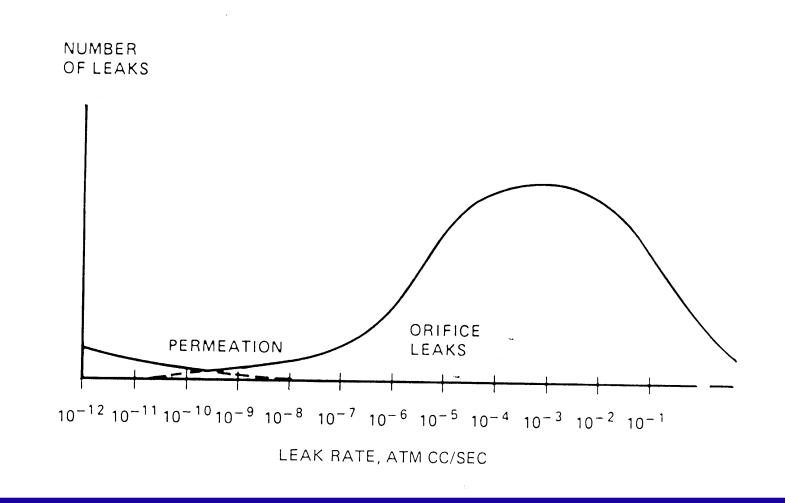








Distribution of Leaks



- Is a process being compromised by the current system performance? poor adhesion or bonding, short filament life reaction of process with gas (air)
- · Determine base and optimal system pressures.
- Choosing the appropriate method of leak detection. pressure gauges, mass spectrometers, *Snoop, acoustical*



Gases leaked into the vessel or system must be removed to maintain a defined pressure. In which case, the pumping speed for the in-leaking gas must be increased.

$$\mathbf{Q}_{t} = \mathbf{S}_{n} \times \mathbf{P}_{t}$$

Gases leaking into the vessel create a change in the vacuum environment. This may change in-chamber chemistry. The result could be a change in coating stoichiometry, adhesion, and possible increases in partial pressures. (Since most vacuum pumps pump gases somewhat selectively, a change in the partial pressures of the internal gases may occur).

Change in chemical composition within the vacuum vessel Change in the process Increase in pump speed required to maintain the desired pressure EXAMPLE:

A 1000-division leak will require a calculatable pumping speed to maintain a pressure (assuming the pump can handle the gas species).

Using the relationship $Q = S \times P$ and the calibration data, we can determine the speed required.

$$Q = 2 \times 10^{-10} \frac{\text{atm} - \text{cm}^3}{\text{sec} - \text{division}} \times 1000 \text{ divisions} = 2 \times 10^{-7} \frac{\text{Torr} - \text{liters}}{\text{sec}}$$
$$S = \frac{Q}{P} = \frac{2 \times 10^{-7}}{1 \times 10^{-7}} \frac{\text{Torr} - \text{liters}}{\text{Torr}}{} = 2 \frac{\text{liters}}{\text{sec}}$$

- Acoustical
- Bubble testing
- Dye penetrant
- Vacuum decay ("rate of rise" test)
- Pressure decay
- Thermocouple gauges
- \cdot Ion gauges and ion pumps
- Halogen leak detectors
- · Partial pressure analyzer (PPA)
- He Mass Spectrometer Leak Detector (HMSLD)



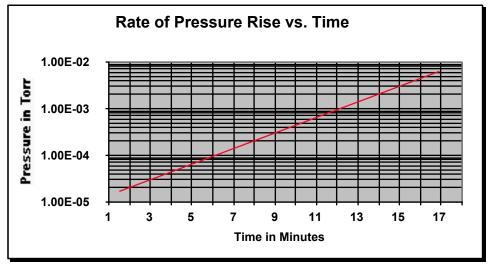
- This procedure integrates the accumulation of gases in the vessel from all sources; outgassing, permeation, inleakage, etc.
- The procedure is to evacuate the vessel to a pre-determined pressure, isolate it from the pump(s) and measure the rate of pressure increase.

$$\mathbf{Q} = \mathbf{V} \frac{\mathbf{P}_2 - \mathbf{P}_1}{\mathbf{t}_2 - \mathbf{t}_1}$$

- What is measured is "Q", gas load in Torr-liters/sec, assuming the vessel volume is known or approximated.
- The slope of the resulting curve can be used to determine the integrity of the vessel regarding leaks and surface cleanliness, and as a proof-test to verify that the vessel will achieve the desired pressure when placed in operation.



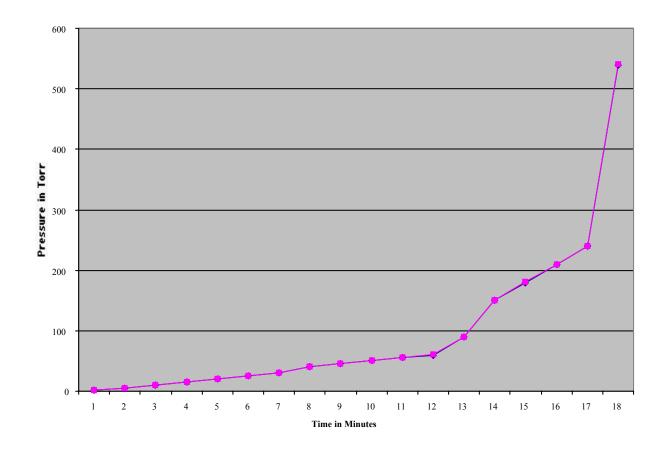
- The rate of pressure rise can be used to determine inleakage, permeation, and outgassing in a system or vessel.
- As an aid to the designing of new systems, rate of pressure rise data can be used to "model" the gas loads of existing systems or process.
- The pumping speed delivered to the vessel can be determined by using the rate of pressure rise data and the known pressure at the start of the test.





Rate of Pressure Rise--plotted data

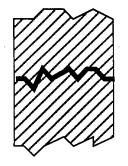
Rate of Pressure Rise vs. Time

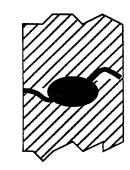


Types of Leaks--Real Leaks



Leaks through a vacuum vessel wall

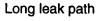




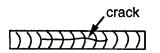
Intermediate volume

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Real leak – physical hole or crack in vessel wall allowing gas to enter the vessel



Leaks caused by stress cracks in welds

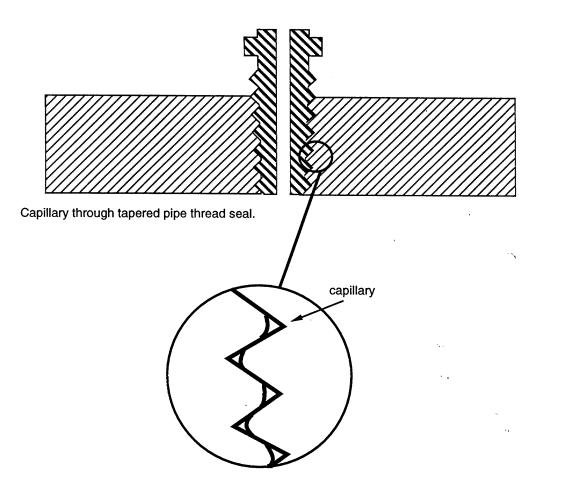


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Cross-section of weld seam

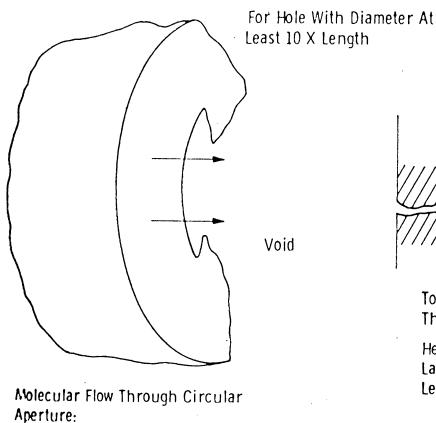


Types of Leaks--Real Leaks



Vacuum Leak Detection

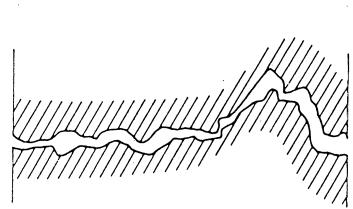




Helium Rate = 2.7 x Air Rate



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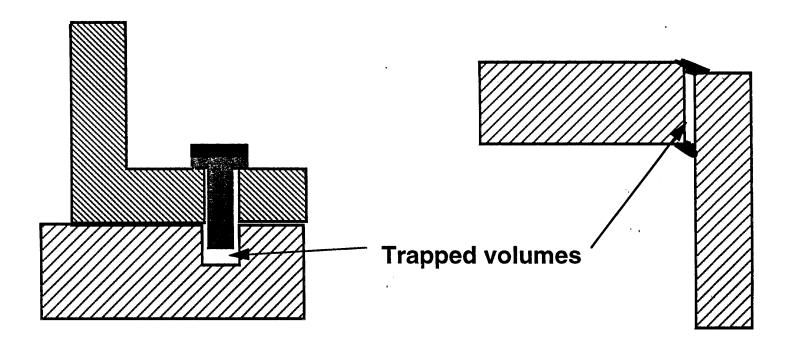
Tortuous Path Whose Length is Greater Than Cross-Section:

Helium Rate May be Equal to Air Rate for Large Leak or Many Times Larger for Small Leak

Most Leaks

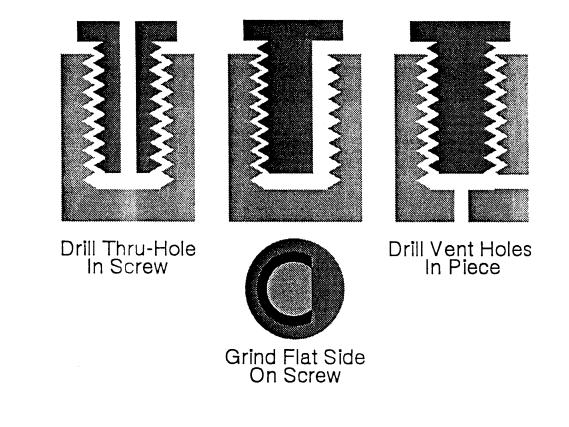


A virtual leak is a volume of trapped atmospheric gas that leaks into the vacuum vessel through holes or cracks that do not go all the way through the vessel wall.

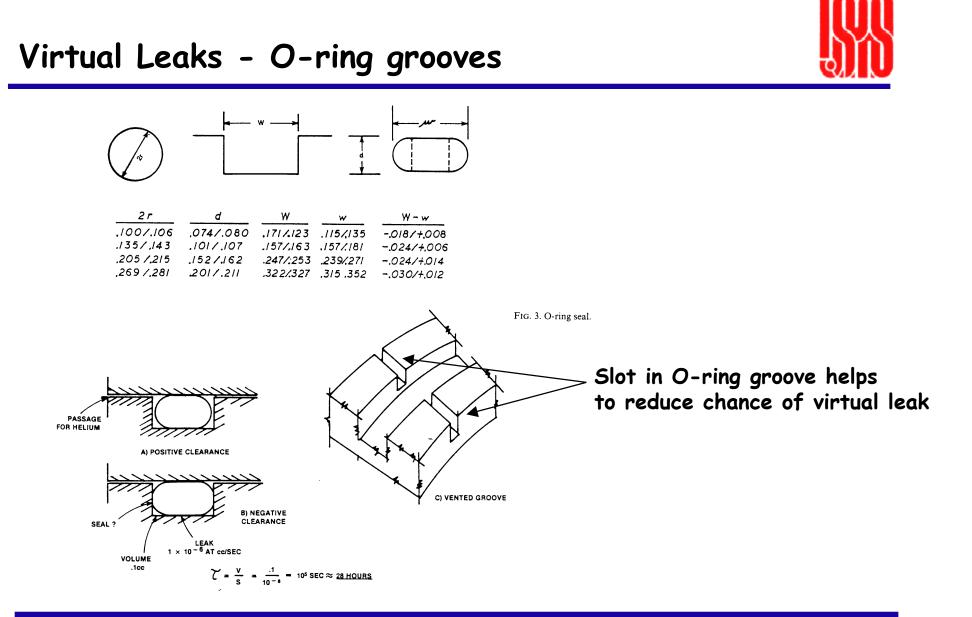




Virtual Leaks - blind tapped holes

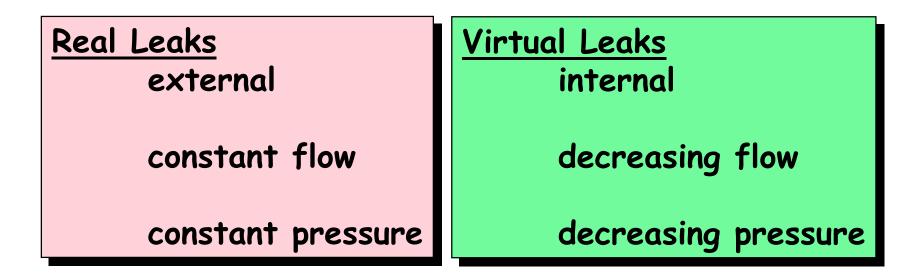


Thread and blind hole venting techniques





Real Leaks vs. Virtual Leaks





Types of Leakage

Gas passes through holes or cracks in the vessel wall. Flow is a function of the size of the flaw.

Permeative gas diffuses through a material having no holes large enough to allow passage of more than a few molecules of gas per unit time. (polymeric materials such as rubber gaskets, O-rings diaphragms, etc.)



Leakage and Outgassing

In general, unless leaks are large, the effects of outgassing will overwhelm the effects of the leaks.

Technique:

- 1. Pump from ~100 mtorr to 1 mTorr, and record time.
- 2. Isolate pumps, allow the pressure to rise to 100 mTorr.
- 3. Repeat step 1 and compare pumping times.

If $T_1 = T_2$, then a leak is suspected. If $T_2 < T_1$, then outgassing may be the culprit.

Average Composition of Dry Air

Gas	<u>Partial Pressure (Te</u>	orr) <u>Volume %</u>
Nitrogen	593	78.1
Oxygen	159	20.9
Argon	7.1	0.934
Carbon dioxide	0.25	0.033
Neon	1.4×10^{-2}	0.0018
Helium	4.0×10^{-3}	0.00053
Methane	1.5×10^{-3}	0.0002
Krypton	8.6 × 10 ⁻⁴	0.00013
Hydrogen	3.8 × 10 ⁻⁴	0.00005
Nitrous Oxide	3.8 × 10 ⁻⁴	0.00005
Xenon	6.6 × 10 ⁻⁵	0.000087

Gas molecules must "fit" through real leaks to be a problem. Molecules Are not discrete spherical particles, however....molecular diameter can be Calculated from gas viscosity.

Hydrogen	2.75 Angstroms
Helium	2.18
Argon	3.67
Oxygen	3.64
Nitrogen	3.64
"Air"	3.74

A 10⁻¹⁰ atm-cc/sec air leak @ 20° C in a 0.25" plate will have a diameter of 10⁻⁵ cm, or 10³ Angstroms, or about 300 times the size of an air molecule.



Helium is the most common gas used as a "tracer" in locating leaks

When compared to other gases, helium has certain advantages as a tracer:

Low molecular weight (4) High intrinsic velocity Small molecular size Chemically inert Non-flammable Readily available Inexpensive Low partial pressure in the atmosphere



Some disadvantages are:

Is not well pumped by ion or chemical combination pumps

Is not well pumped by cryogenic pumps



Molecules in the gas phase have a distribution of velocities, the average velocity (v):

$$v = 14,551 \left(\frac{T}{M}\right)^{\frac{1}{2}}$$
 cm/s

M = molecular weight

For N_2 at room temperature (20 °C) :

$$v = 14,551 \left(\frac{293}{28}\right)^{\frac{1}{2}} = 4.71 \times 10^4 = 1054 \text{ mph}$$

Note that v is independent of pressure



- It is a Helium-specific partial pressure analyzer
- It detects Helium applied as a tracer or probe gas

It consists of:

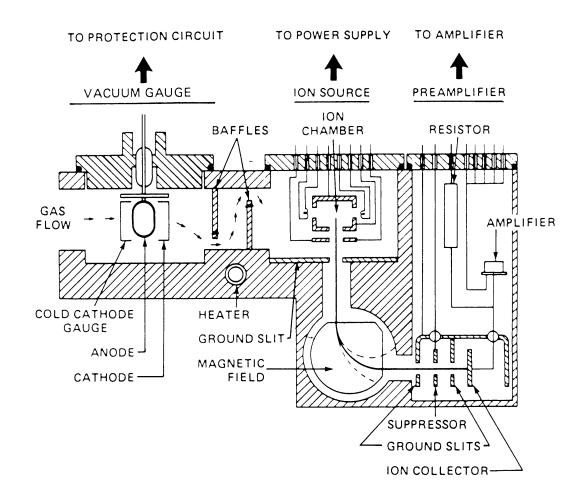
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the mass spectrometer tube it's own vacuum system capable of 10⁻⁵ Torr in the spectrometer tube a sensitive and stable amplifier valves, and auxiliary pumps for interfacing to vacuum system a display for monitoring leak rate normal-flow vs. counter-flow configurations



Cross-section of a Typical Spectrometer Tube





Calibration of a leak detector is accomplished by attaching the leak standard, allowing the leak to flow into the detector, and reading the output from the spectrometer tube on the leak rate meter. A straight forward calculation is made and the calibration of the meter is understood. It must be noted that variations in temperature, detector pumping speed, electronic "drift" and background noise can influence the stability of the calibration.

 $Calibration = \frac{Standard Leak Rate}{Change inLeak Rate Meter}$

Calibration = $\frac{\text{atm} - \text{cm}^3}{\text{sec} - \text{division}} = \frac{2 \times 10^{-7}}{1000} = 2 \times 10^{-10} \frac{\text{atm} - \text{cm}^3}{\text{sec} - \text{division}}$



Most common (and desirable method): The HMSLD is connected to the system, and a helium tracer gas is applied to the exterior of the system under test in a controlled manner. ASTM E-498-94

Connection configurations:

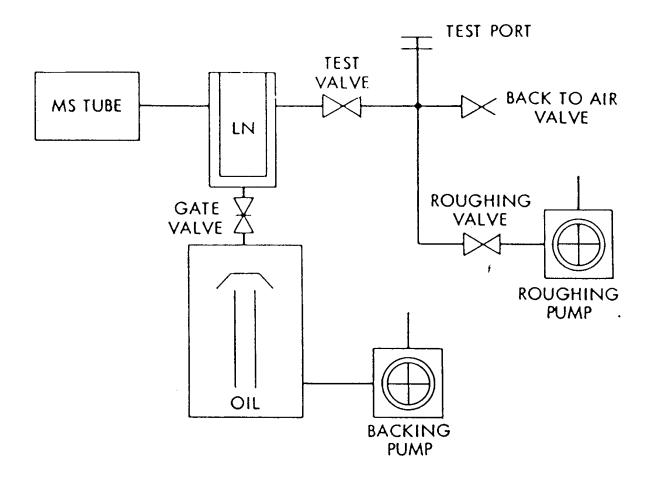
- 1. Directly to the component or system
- 2. In parallel with other pumps on the system
- 3. In series, backing another pump connected to the system



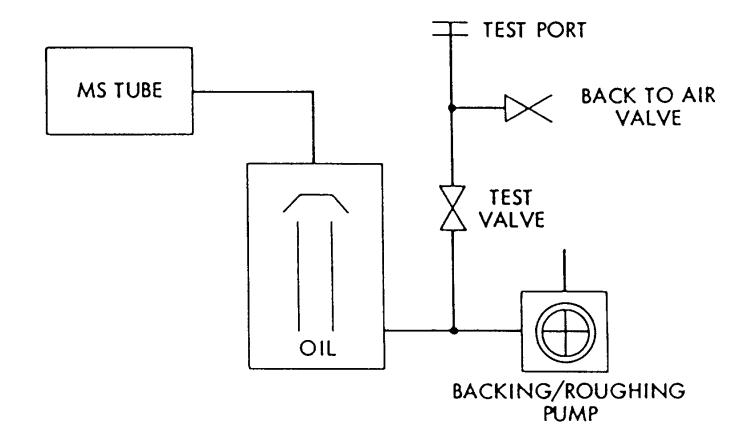
Most helium leak detectors have two test modes; normal-flow and contra-flow. You, as the operator, can decide which mode you should be operating in.

Either mode has distinct advantages and disadvantages!









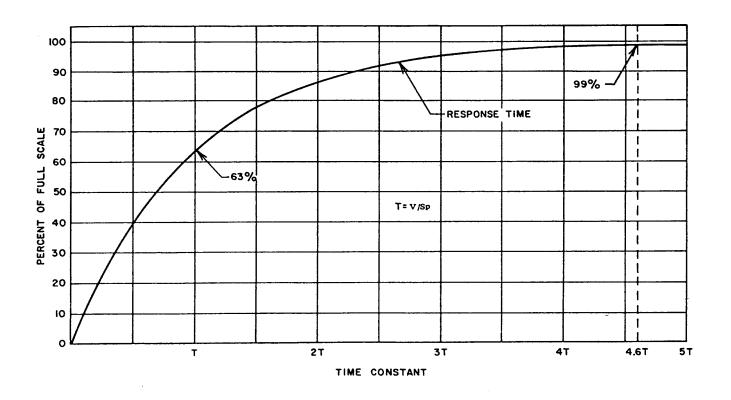
<u>Response time</u> is often defined as the time it takes a HMSLD to indicate a rise in signal (63%) after the application of a tracer gas.

- Sensitivity of the instrument
- Tracer gas leak rate
- Volume of the system under test
- Pumping speed for helium of the HMSLD
- Pumping speed of any additional pumps

Time Constant and Response Time



Ideal Response Time





Manifolding Cells Together for Leak Testing of Spatial Filter Vacuum Vessel (Ranor)

10" vacuum lines connect to 2000 l/s turbo pump



<u>S</u>S

Calibrated Leak Mounted to 100,000 liter Vessel

Leak was attached at the most remote port on the vessel.

Response time was incredibly rapid, about 8 seconds!



Spatial Filter Being "Bagged" for Total Integrated Helium Leak Test







- · Background (outgassing)
- · Large Volumes, slow pumping speed for Helium
- Helium permeation
- · Leak "plugging"
- Detector maintenance
- Operator training



Leak Detection, Tips & Tricks

- Pipe threads (and the use of *Teflon* tape)
- Use of "Accu-pucky", vacuum sealants and sprays
- Helium dissolves in most vacuum greases
- · Isolate O-rings to prevent permeative "masking" of real leaks
- · Always test the connecting lines first!
- When introducing helium, start at top and work down (Tracer probe)









- Mount a calibrated leak to the system under test (response time)
- Calibrate the HMSLD before, and after, each use
- Minimal use of the tracer gas (adjust in water or solvent)
- Operate diffusion-pumped systems properly, especially at start-up and shutdown
- Don't use your leak detector as a portable pumping station!

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Techniques for Detecting Small Leaks

- · Check the sensitivity of the leak detector
- · Calibrate the HMSLD using an external standard
- Flow all pumped gases through the HMSLD, if possible
- Use low-flow tracer probe technique
- Keep Helium away from permeable materials (elastomers)
- Make use of "bagging" and "taping" techniques



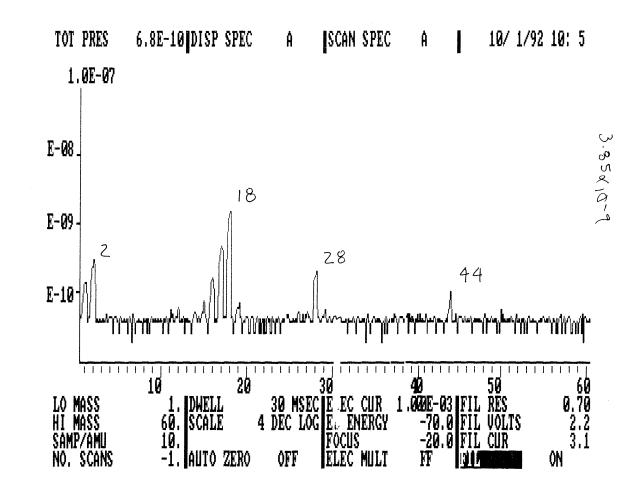




- When calibrated, they can provide quantitative as well as qualitative data regarding the vacuum environment.
- RGA's have the ability to measure real-time environment changes.
- Gas Analyzers can be calibrated for various gas species.
- Gas Analyzers are often mounted permanently on a vacuum vessel or process equipment, utilizing the equipment's own pumping system.

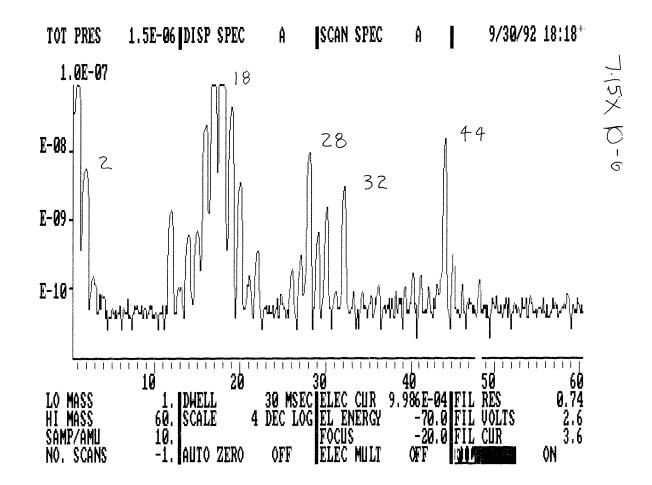


Clean System Spectrum



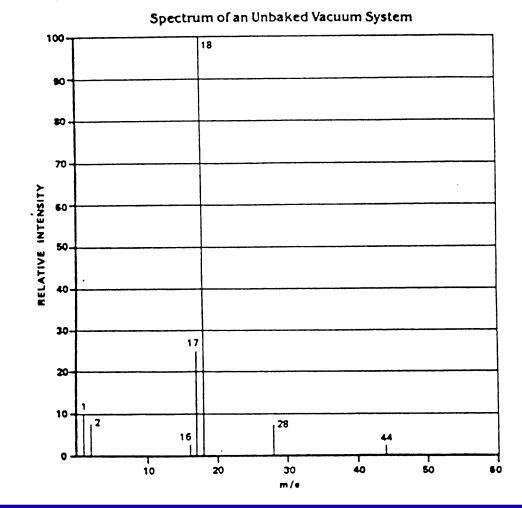
Less-than-clean System Spectrum





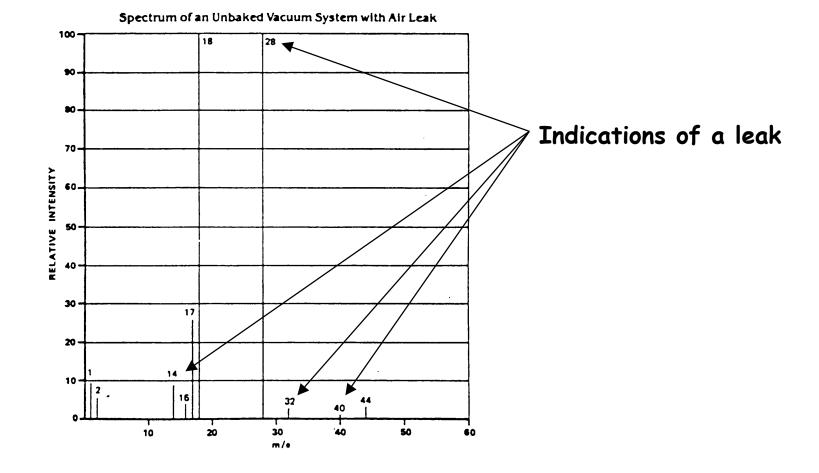


Spectrum of an Unbaked Vacuum System





Spectrum of a System With an Air Leak



Equipment-related Factors that Influence Residual Gases in the Vacuum Environment

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Backstreaming (system design)

Desorption

(surface condition of vessel walls)

Selection of pumping action

(capture vs. momentum transfer)

Vaporization of materials

(low vapor pressure materials)

Permeation

(elastomers)

Leakage (real leaks) (virtual leaks)

Operator-related Factors that Influence Residual Gases in the Vacuum Environment

Handling procedures grease, oil, salt

Cleaning procedures solvents (alcohol, acetone, MEK)

Fabrication Techniques machining coolants and lubricants voids and occlusions

Operation Procedures use of traps venting system to room air backstreaming



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- Specification can include:
 - · Maximum allowable leak size
 - · Total maximum leakage rate (infers bagging)
 - \cdot Component pressure during leak detection
 - \cdot Type and sensitivity of the leak detector (e.g. MSLD with a sensitivity of 2 x 10^{-10} atm-cc of He/s
 - Use of certified standard leak immediately before and after testing
- ASTM standards E432, E479, E493, E498, E499, and F97
- If application is critical, witness the testing, or do it yourself
- Avoid phrases like; leak tight, vacuum tight, good to 10⁻⁸
 Torr, good for ultrahigh vacuum, etc.