Sources of and Solutions to Contamination Issues in Space Simulation (TVAC) Systems

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Renewed Life for Space Travel

Space – the Greatest of all Vacuums

Humans as a Multi-Planet Species

Living in Extreme Vacuum, Temperature and Radiation

Into Low Earth Orbit - Micro-Satellite

Into outer space Mars or Lunar Rover

Surface Conditions for Rovers

Parameter	Moon	Mars
Atmosphere	Vacuum, <10^{-7} Torr (1.33 10^{-5} Pa)	3 to 12 mTorr (0.16 to 1.6 Pa), mainly CO_2 with some Ar and H_2O
Equatorial Solar Illumination	1366 W/m^2	588.6 W/m^2
Gravity	0.165g	0.375g
Day/Night length	14 Earth Days	24 hours 37 minutes (Earth Time)
Lofted Dust	Yes	Yes
Incident electrons	Yes	Yes
Incident Protons	Yes	Yes
Daytime temperatures	> 393K	256-300K
Night time temperatures	<120K (<40K in permanent shadowed polar regions)	130K (winter polar caps) - 166K (Viking landing site)

Examples of T-Vac Systems

Range of Specifications for Thermal-Vacuum Systems

- Vacuum Specifications
  - 1 x 10^-7 Torr up to 1,000 Torr
  - 1 x 10^-2 Torr up to 760 Torr
- Thermal Specifications
  - Overall System = +150 C, to -180 C, Test Pieces – 50/+70C
  - Overall System = +70 C, to -50 C
- Size, Accessibility, special requirements

System Performance: Ramps Rates

- Temperature
  - 1.5 C/min from -180 C to + 150 C
  - 1.2 C/min from -45 to +110 C
- Pressure
  - 1 hour to 1 x 10^-4 Torr
  - 6 hours to 1 x 10^-6 Torr
  - +21 days at 1 x 10^-6 Torr

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System Design Basics

- Payload
  - 100 to 400 lbs.
- Thermal Cycle Duration
  - Up to 500 hours
- Pumping System
  - Cryopump, Turbo pumps, RV or Dry Scroll
- Controls System –
  - Multiple profiles, sensors on test piece, LabView

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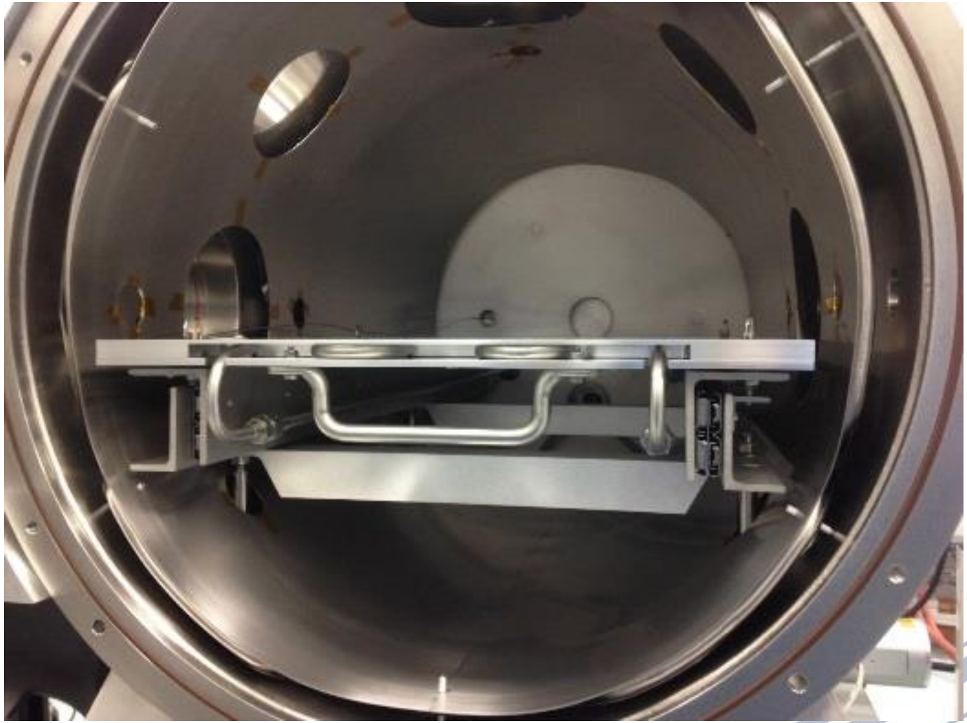
Teach Design to Avoid Contamination

- Vacuum Engineering
- Smart System Operation
- System Materials
- Manufacturing Expertise
- Vacuum Cleanliness

Temperature Controlled Shroud System

Thermal Control Skid Detail



Thruster Test Facility

Bogazici Univ Space Technology Lab

Contamination in Space Simulation Systems

- **Gas – Solid collisions from:**
  - **Vapors from the system**
  - **Vapors from the payload**
  - **Vapors from humans, other sources of microbials**
- ***Things that tend to stick***

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Gas Loads from Payload

- CubeSats
  - Circuit boards
  - Electronic components
  - Solders
  - Poor vacuum practice

- Reusable Rockets – like rental cars?

- Rovers
  - All of the above
  - Lubricants
  - Dusts

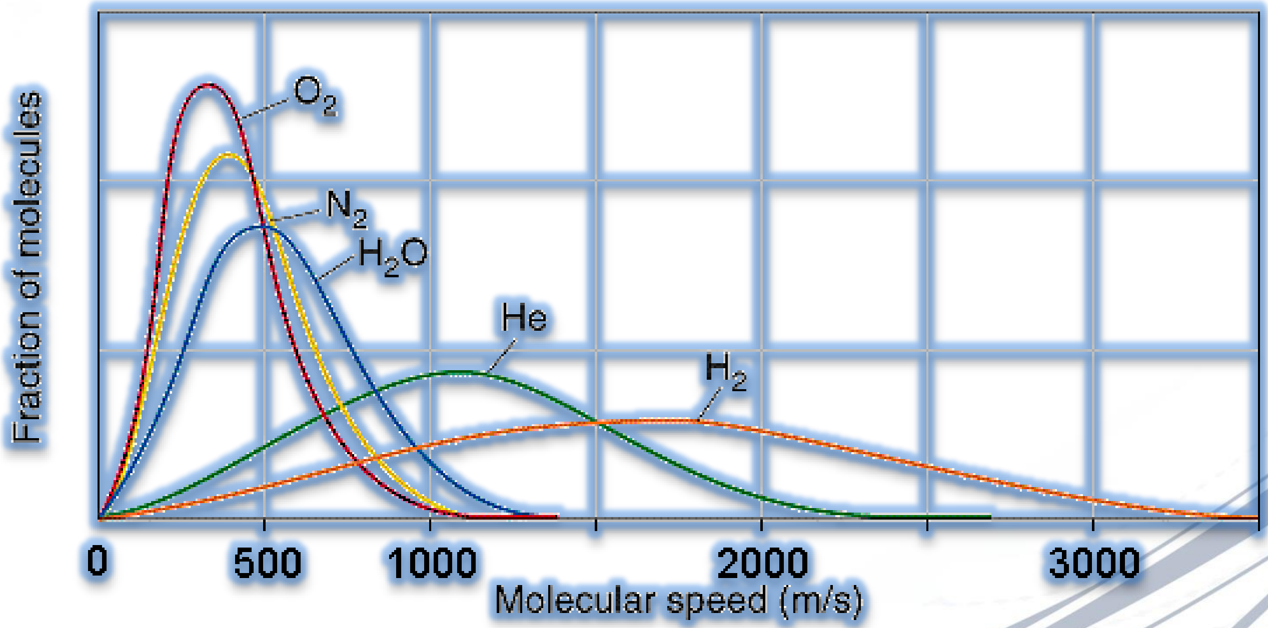
The Atmosphere

Constituent	Formula	Vol. Composition
Nitrogen	N_2	78.084%
Oxygen	O_2	20.948%
Argon	Ar	0.934%
Carbon dioxide	CO_2	0.0315%
Neon	Ne	0.001818%
Helium	He	0.000524%
Methane	CH_4	0.00015%
Hydrogen	H_2	0.00005%
Ozone	O_3	~0.00004%
Water vapor	H_2O	~0.5% to ~2.4%

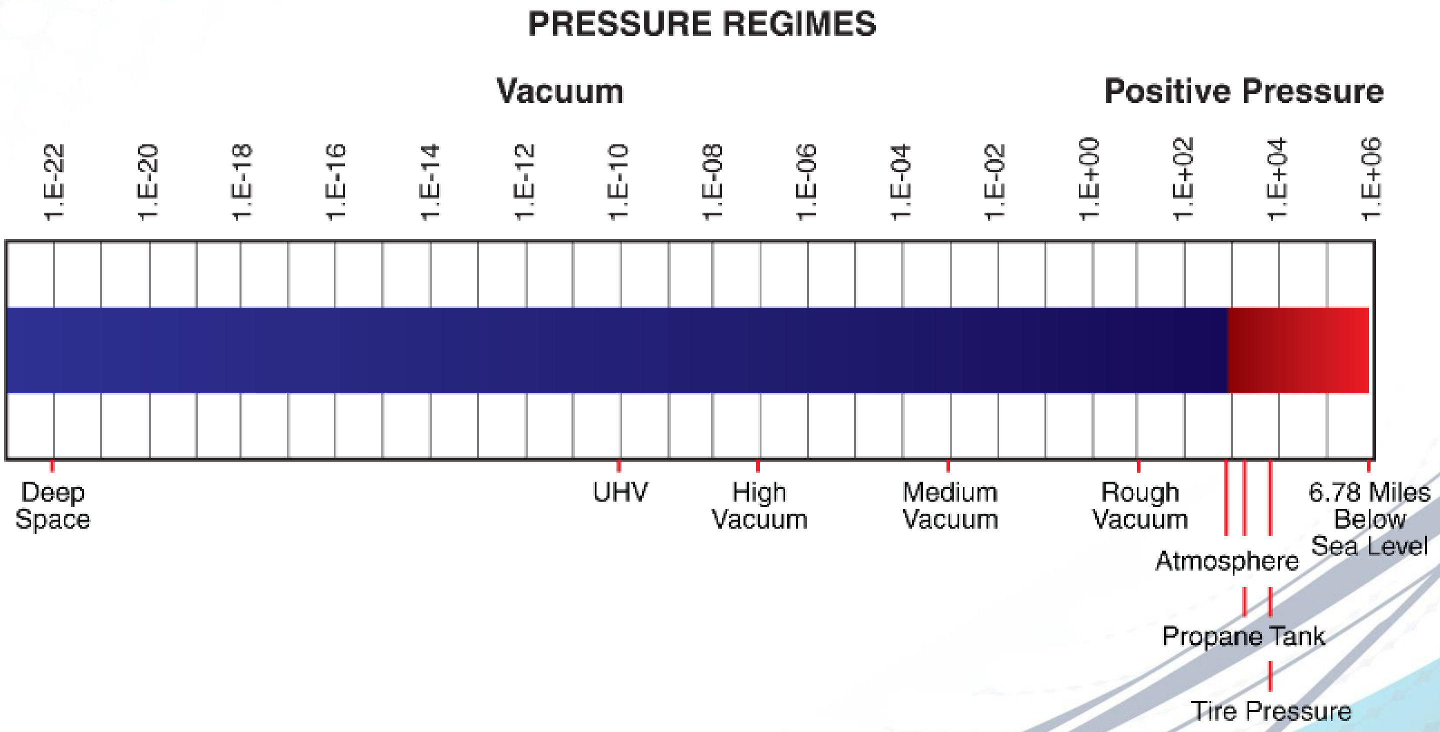
My Neighborhood

	Pulv. coal combustion flue gas	Waste incinerat. flue gas	Coal gasification fuel gas §	Coal-fired IGCC flue gas	Natural gas Groningen	Gas-fired CC flue gas
O_2 %-v	~ 6	7 - 14		~ 12		~14
N_2 %-v	~ 76	balance	~4 / ~1	~ 66	~14	~76
CO_2 %-v	~11	6 - 12	~4 / ~13	~ 7	~1	~ 3
H_2O %-v	~ 6	10 - 18	~4 / ~1	~ 14		~ 6
CO %-v		0.001-0.06	~58/~40			
H_2 %-v			~30/~29			
Ar %-v	~ 1	~ 1	~ 1	~ 1		~1
SO_2 ppmw		200 -1500		10 - 200		
H_2S ppmw			1000-4000			
NOx ppmw	500 - 800	200 - 500		10 - 100		10 - 300
NH_3 ppmw			300 - 800			
HCN ppmw			40 - 150			
HCl ppmw		400 - 3000	500 - 600			
HF ppmw		2 - 100	150 - 250			
dioxine ppb	<< 1	1 - 10				
CH_4 %-v					~ 81	
C_nH_m %-v		< 0.002			~ 4	
Hg ppmw	0.1 - 1	0.1 - 1	0.01 - 0.1			
Cd ppmw	0.01 - 1	0.1 - 0.5	0.01 - 0.2			
other heavy metal ppmw	0.5 - 2	1 - 5	~ 20			
dust g/m^3	5 - 20	0.2 - 15	~17 / ~8	<< 0.02		

Molecular Speed



Molecular Density in Vacuum



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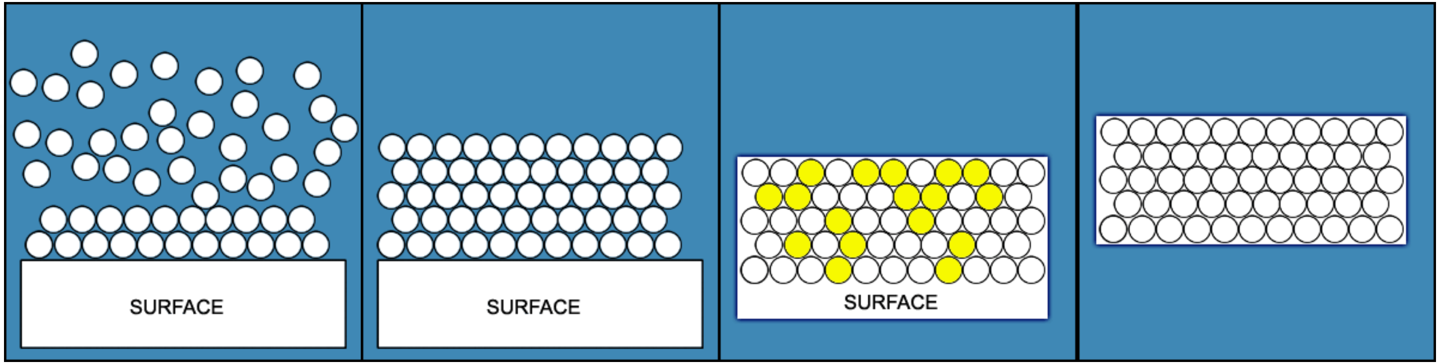
Chamber Atmosphere under Vacuum

Gas	Volume % in dry air	Volume % in ion pumped chamber at 2 x 10^-9 Torr	Molecules
N_2	78%	trace	
O_2	21%	trace	
Ar	0.93%	trace	
CO_2	0.03%	3%	975,000
CH_4	trace	3%	975,000
H_2O	trace	5%	1,625,000
CO	trace	6%	1,950,000
H_2	trace	78%	25,350,000

Gas/Solid Interactions – Basic Processes



Adsorption   Desorption   Diffusion   Permeation

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Gas Load – What is it?

The total mass (quantity/amount) of gas entering the vacuum volume from all sources in unit time

Mass flow measured in:

T.L/s, Pa.m³/s, mbar.cc/s, atm.cc/s, sccm

(any units of pressure x volume / time)

Gas Load - Sources

Permeation

Real leaks:

- *from air*
- *not from air*

Virtual leaks

Backstreaming

Diffusion

Sublimation/Evaporation

*Injected gas**

Outgassing

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Outgassing – Main Components

Water vapor

Oil/grease ('hydrocarbons')

Solvents

VOMs

H_2 and CO

'Other stuff' – used to make space-bound components

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Outgassing – Rate

Consider all gas phase and absorbed phase gas and vapor molecules inside a vacuum chamber

Outgassing Rate is Δ number of molecules:

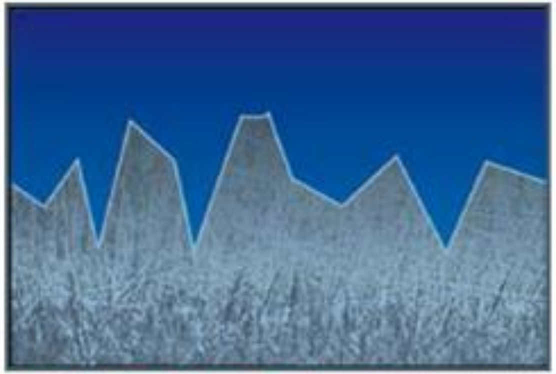
desorbing from the surface in time 't'

adsorbing on the surface in time 't'

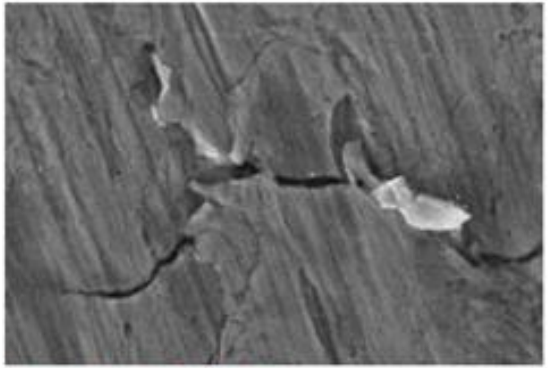
In a continuously pumped chamber :

Outgassing Rate decays exponentially

Surface Roughness of Stainless Steel

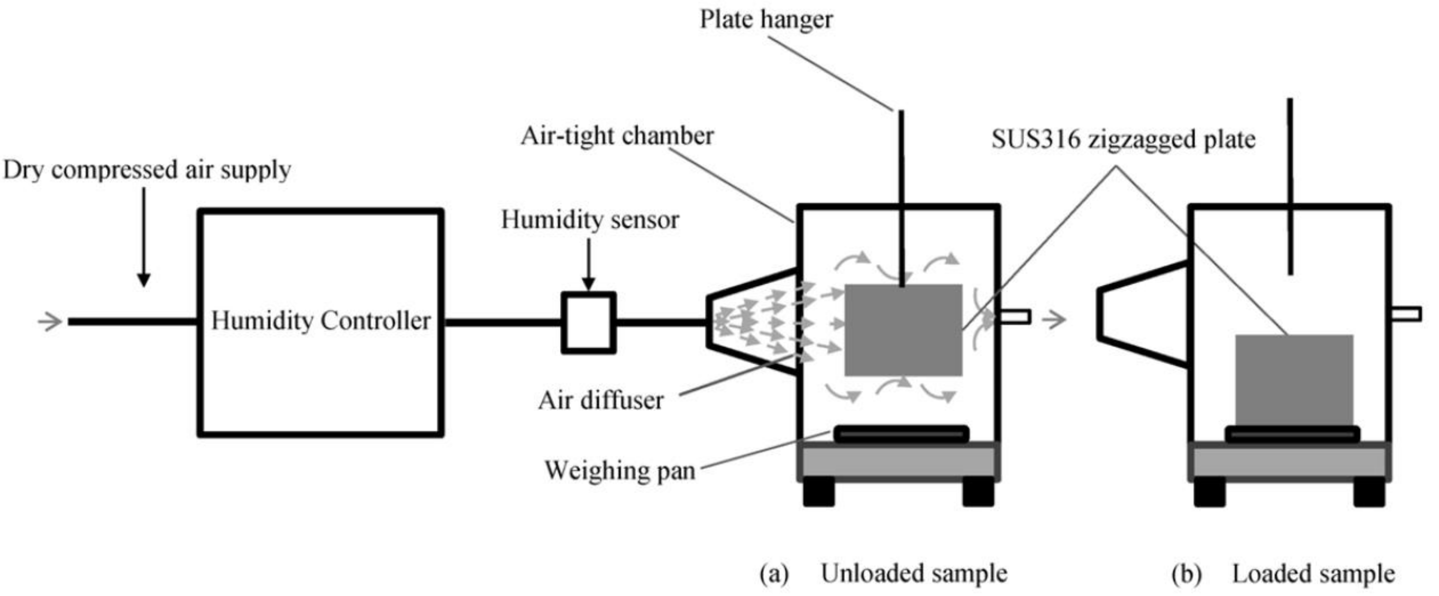


Surface Profile before Electropolishing



Original 'mill' finish (x5k)

Estimation of Adsorbed Water on SS



Number of layers of water at 90% RH

- One mono-layer of water contains 1 x 10^{15} molecules/cm^2
- Sample size of 30 x 10 cm, or 600 cm^2
- Nominally 333 monolayers, based on a weight gain of 0.006 gram

Why is Water so Sticky?

- Binding Energy (BE) between surface and adsorbed gas
  <15 kcals/mole, like Ar and N_2, will desorb in vacuum at RT

- If BE is >25 kcals/mole gas desorbs too slowly
  to cause gas load issue

- BE of water vapor is about 20 kcals/mole

Outgassing – Worst Sources

Open cellular material (large surface area)

Porous surfaces (ceramics or metals)

Plastics, elastomers, polymers

Previously back-streamed oil

Epoxy glues

Lubricating/sealing/heat transfer greases

And US!!!

(hair, skin cells, dust mites, spit, fingerprints, food)

Issues Caused by Humans

- Lack of vacuum cleanliness – the Human Touch
- Inexperience in vacuum system operation
- Gas flow patterns in the system (design issues)
- Response time, etc… (design issues)

Challenges of Hydrocarbons

- Pump oils – visible or not
- Fingerprints – about 10^19 molecules per print
- Skin cells – about 600,000 per hour
- Hair – about 100 to 125 hairs per day
- Speaking projects tiny spit balls

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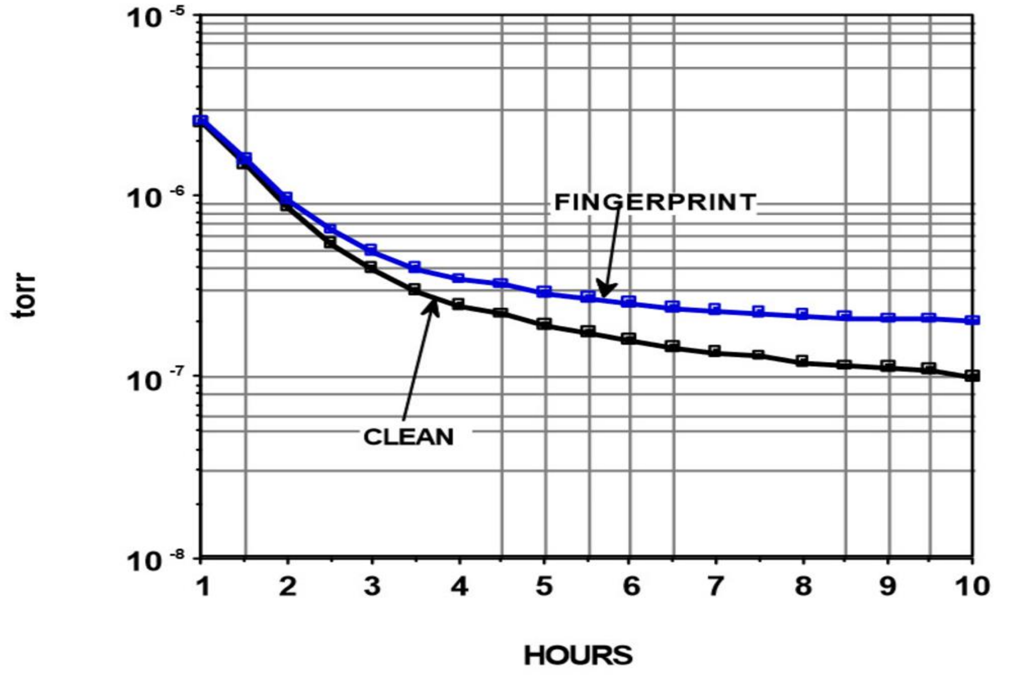
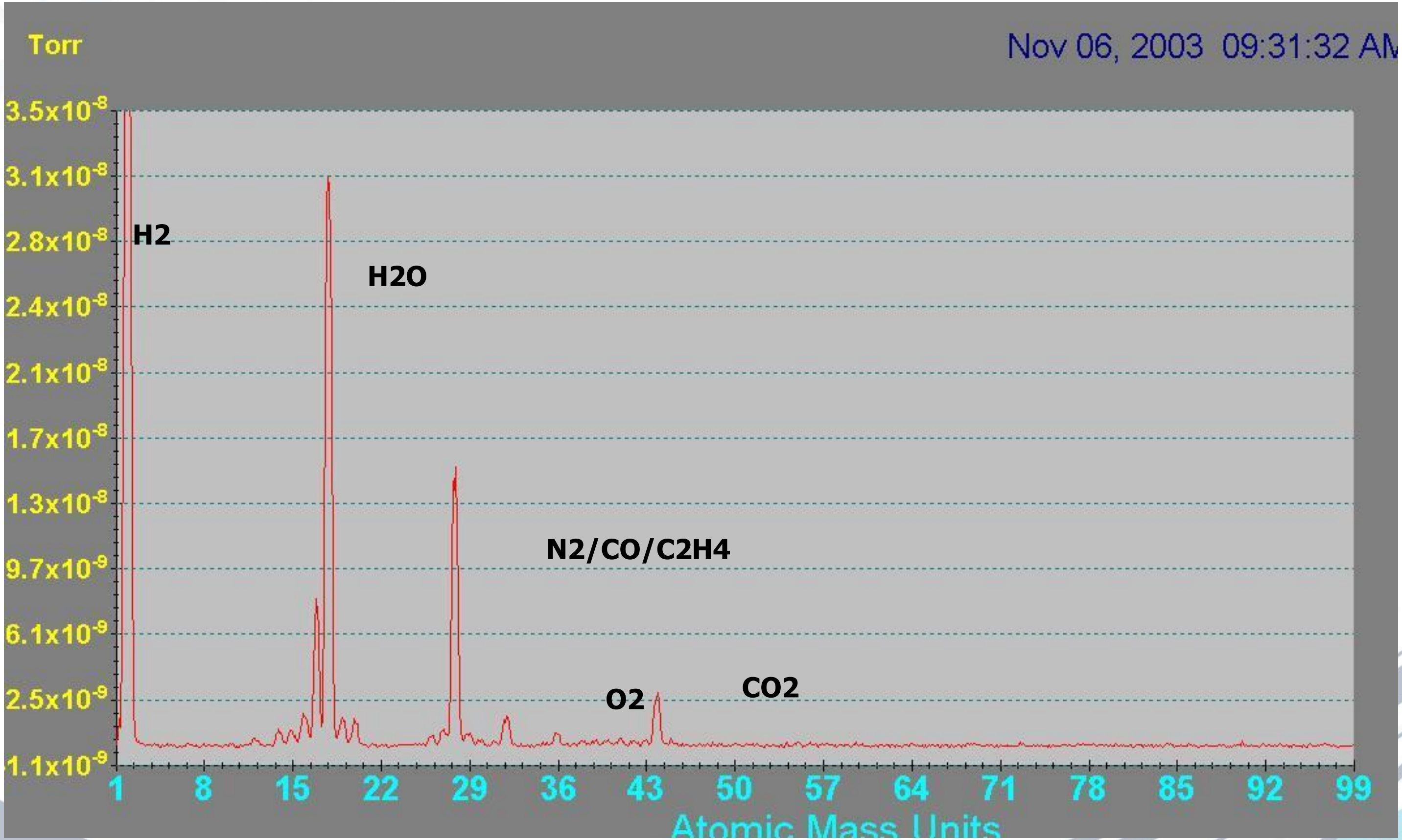
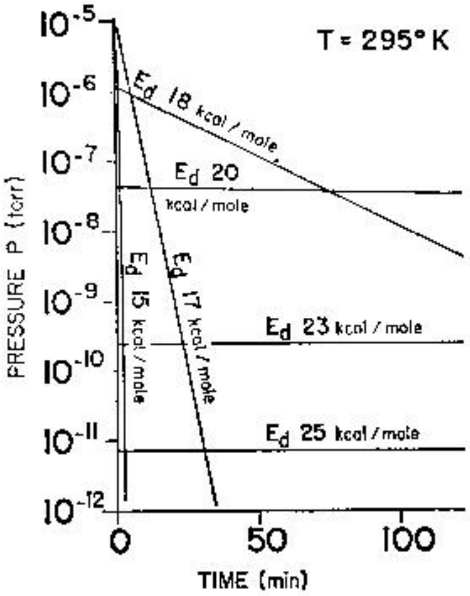
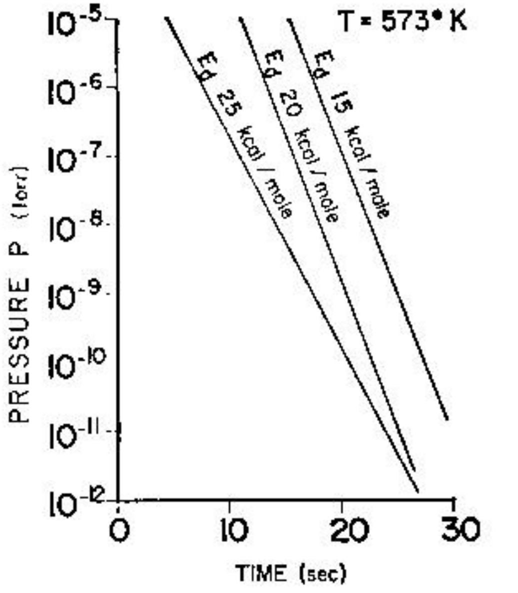
Impact of One Fingerprint




Torr
Nov 06, 2003 09:31:32 AM
3.5x10-8
3.1x10-8
2.8x10-8
2.4x10-8
2.1x10-8
1.7x10-8
1.3x10-8
9.7x10-9
6.1x10-9
2.5x10-9
1.1x10-9
H2
H2O
N2/CO/C2H4
O2
CO2
1
8
15
22
29
36
43
50
57
64
71
78
85
92
99
Atomic Mass Units

Outgassing Reduction – Baking





1L, 1L.s, 100cm2

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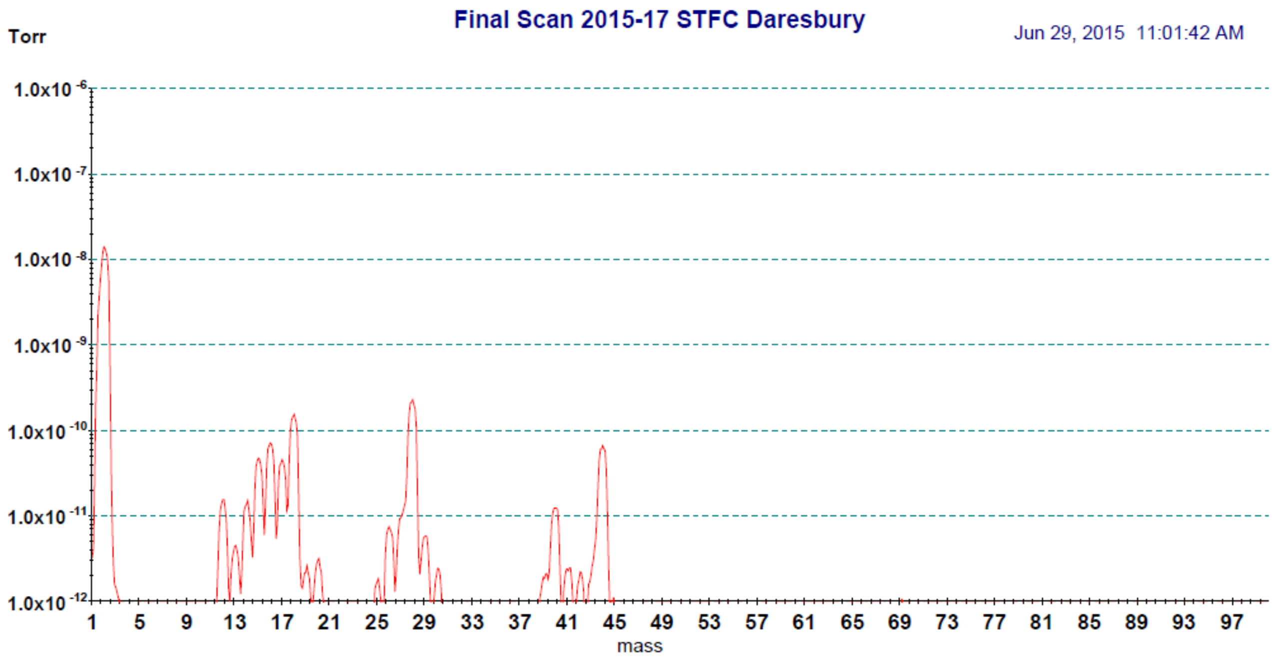
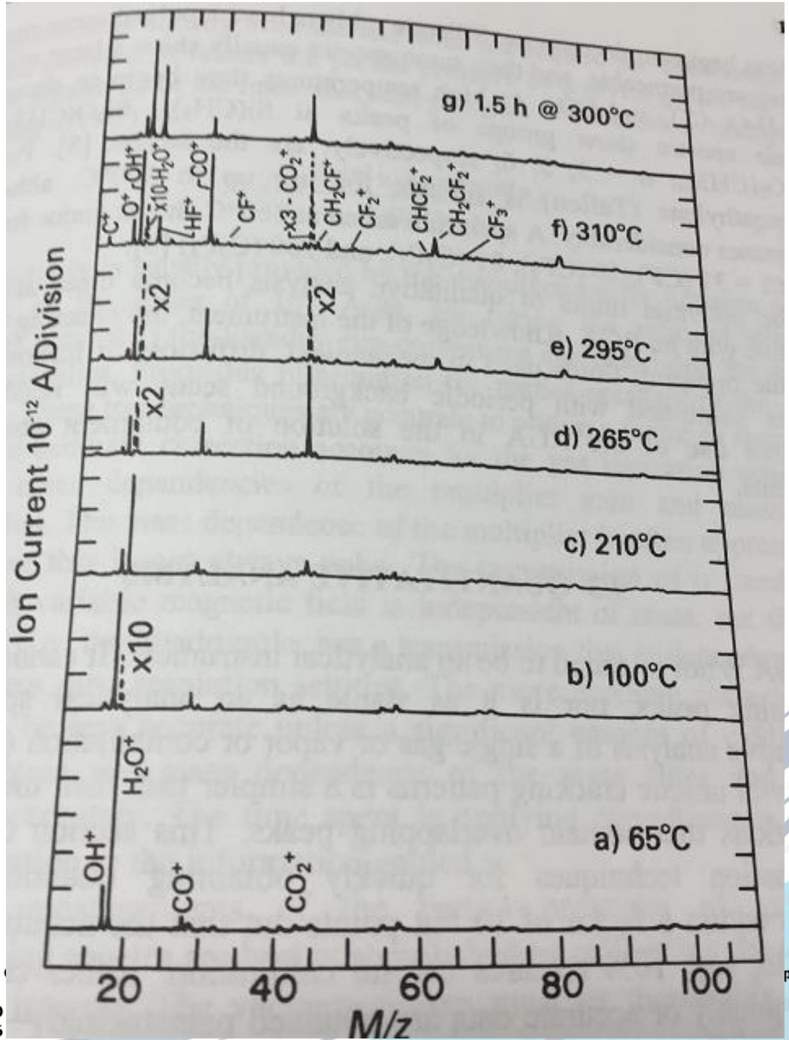
Component in Vacuum Bake out System with RGA

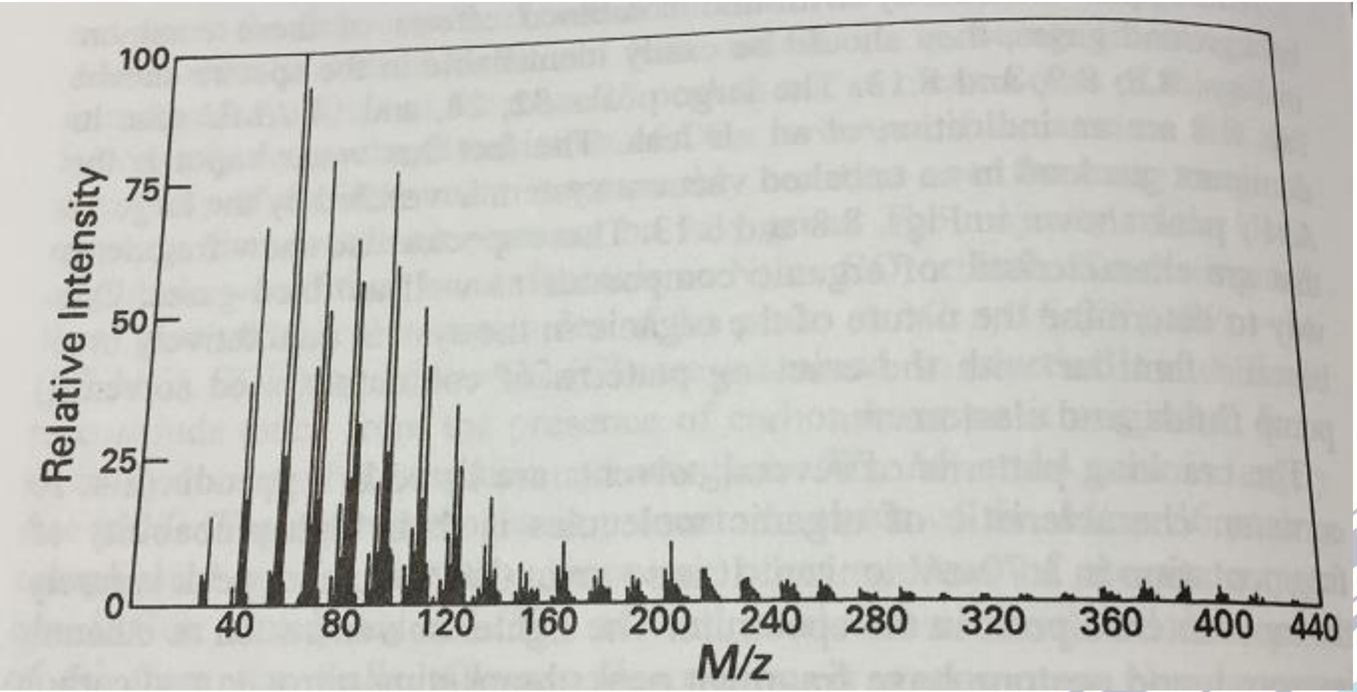
Raw RGA Data for New Vacuum Component bake-out, 250 C for 48 Hours



Decomposition of A Viton O-Ring at high temperature of 310 C

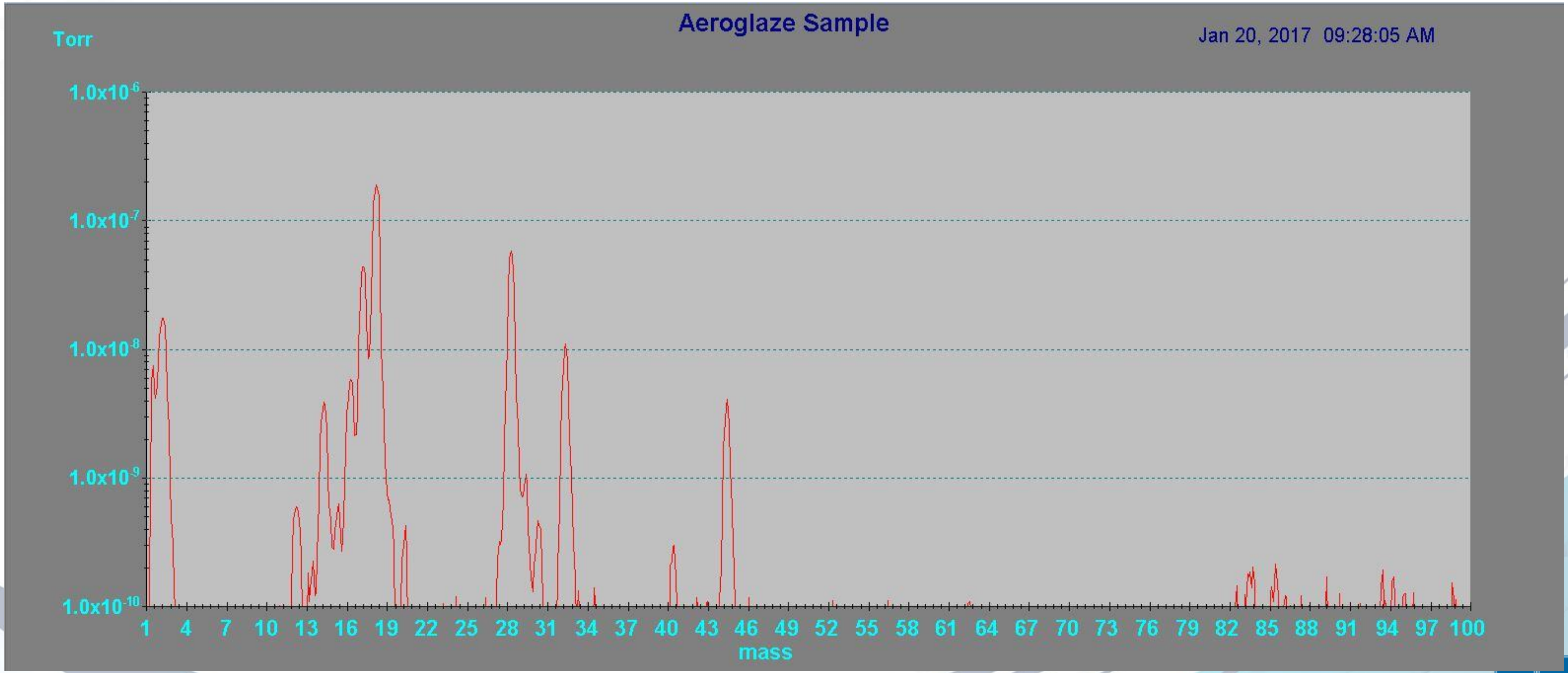


Apiezon Vacuum oil 'finger print'



Coatings to Enhance Heat Transfer

Outgassing of Emissivity Coatings



Bacteria Surviving in Space

Chemical Composition of Life

- Hydrogen, oxygen, nitrogen, carbon, sulfur, and phosphorus normally makeup more than 99% of the mass of living cells.
- Ninety-nine percent of the molecules inside living cells are water molecules.
- Cells normally contain more protein than DNA.
- Homogenous polymers are noninformational.
- All non-essential lipids can be generated from acetyl-CoA.
- Like certain amino acids and unsaturated fatty acids, various inorganic elements are dietarily "essential".
- Most all diseases in animals are manifestations of abnormalities in biomolecules, chemical reactions, or biochemical pathways.

Chemical Composition of Living Cells

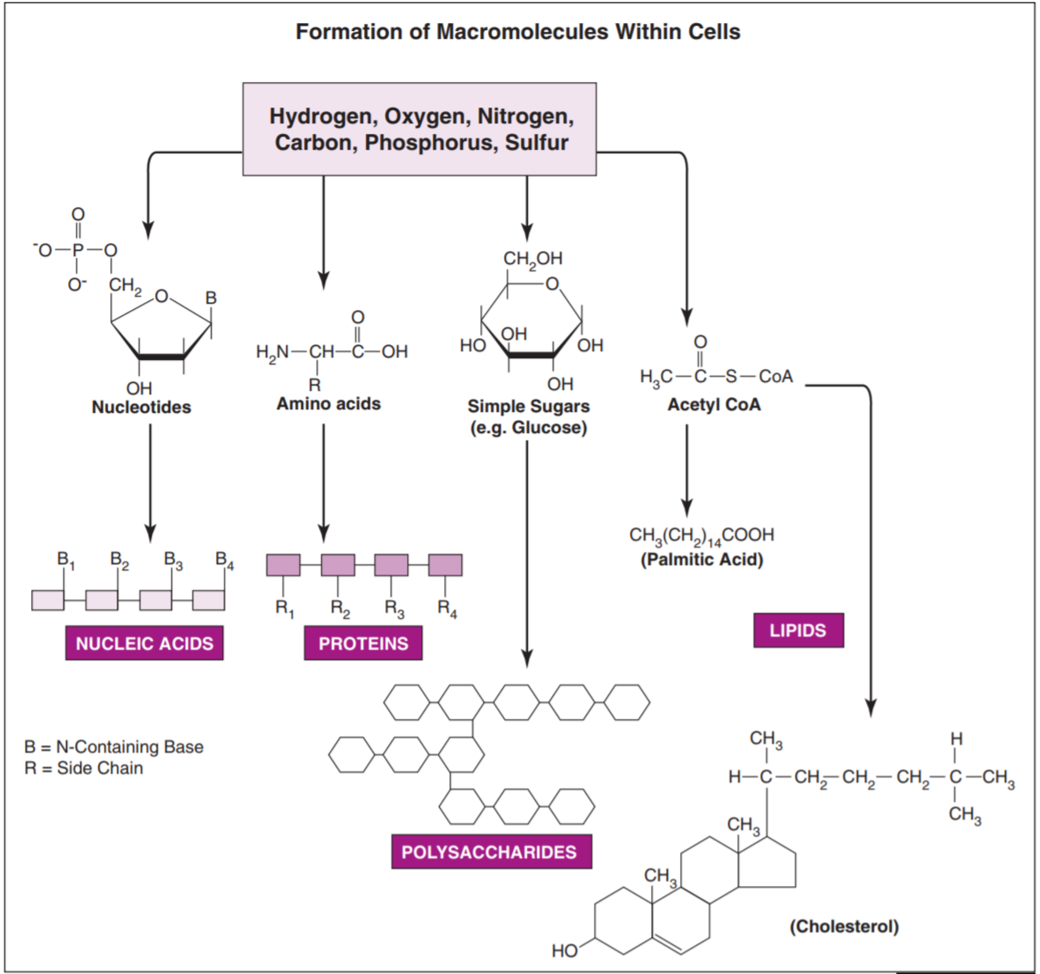


Figure 1-1

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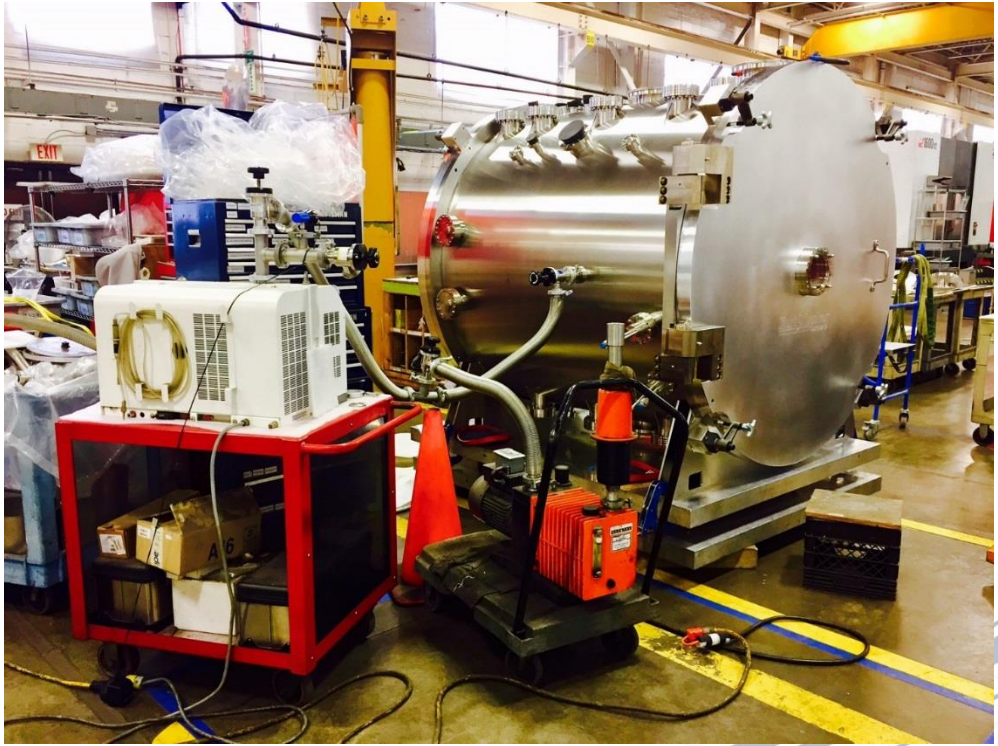
Atomic Mass Units of Usual Suspects

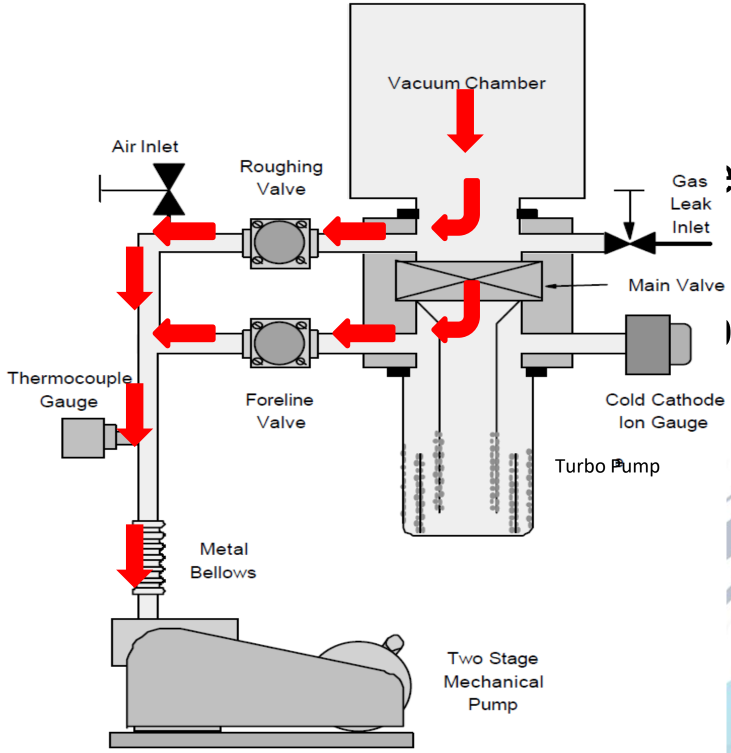
	Formula	AMU	Major Peaks	Secondary Peaks - Ions	Secondary Peaks - Ions	Secondary Peaks - Ions	Secondary Peaks - Ions	Secondary Peaks - Ions
Air	see below	29						
Nitrogen	N2	28	28	14				
Oxygen	O2	32	32	16				
Argon	Ar	40	40					
Carbon dioxide	CO2	44	44	32	16	12	28	
Neon	Ne	20	20					
Helium	He	4	4					
Methane	CH4	16	16	12	4	16	20	
Hydrogen	H2	2	2	1				
Carbon monoxide	CO2	44						
Ozone	O3	48	48	32	16			
Water vapor	H2O	18	18	4	16	8	20	
Acetone	C3H6O	58	58	12	36	1	6	16
Isopropal	C3H8O	60	60	12	36	1	8	16

Contamination from Pumping Systems

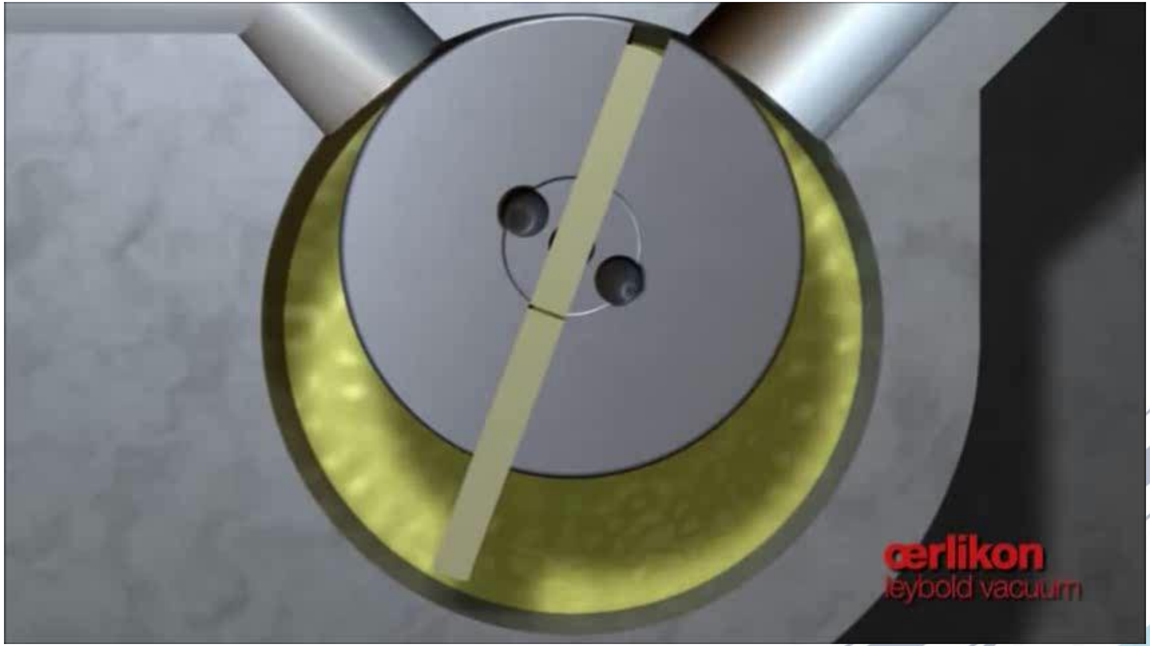


Roughing / Backing Pump

- Roughing Pump
  - Prior to starting Turbo fir[...]r from atm
- Backing Pump
  - Once able to start turbo [...]ports Turb



Rotary Vane Pump
760 – 10^{-4} Torr
1-1200 cfm
Gas Transfer Pump
Oil Sealed
1-Stage Pumps (Coarse Vacuum)
2-Stage Pumps (Rough Vacuum)
Vents to atmosphere

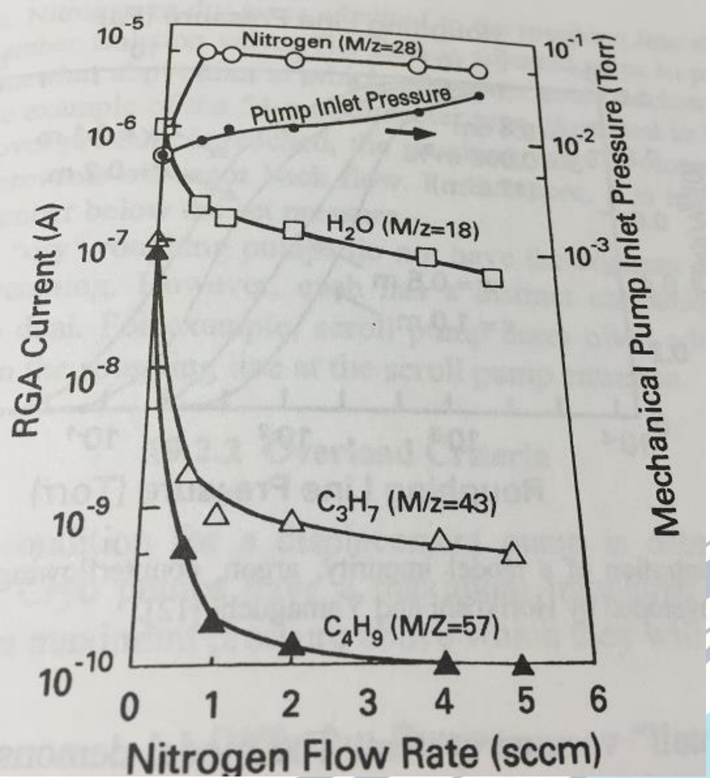


Backstreaming of Pump Oils

Well Studied (surprised?!!)

<u>Small</u> ~ 10^{-4} mg/min
at high pressure due to flushing action of atmosphere

<u>70 times larger</u>, ~ 7 x 10^{-3} mg/min,
at lower pressure of 10^{-2} Torr



Outgassing Reduction – Pump Time

	after 1 hour of pumping		after 10 hours of pumping	
Aluminum (fresh)	6.E-05	Torr-liters/sec	6.E-06	Torr-liters/sec
Copper (fresh)	4.E-04	Torr-liters/sec	4.E-05	Torr-liters/sec
Stainless steel	1.E-04	Torr-liters/sec	1.E-05	Torr-liters/sec

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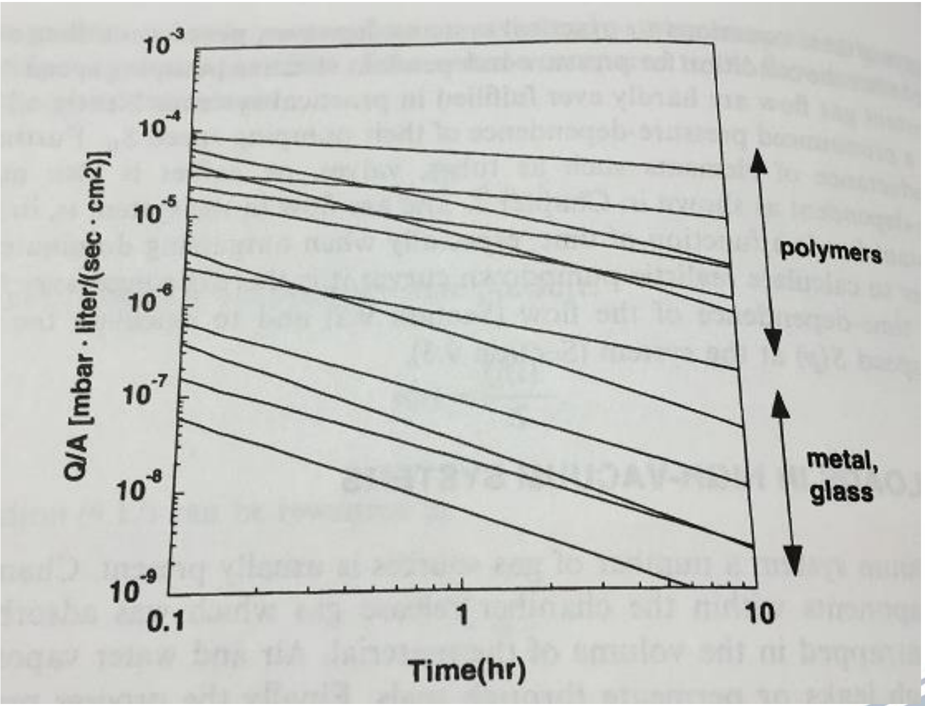
Outgassing flow rates



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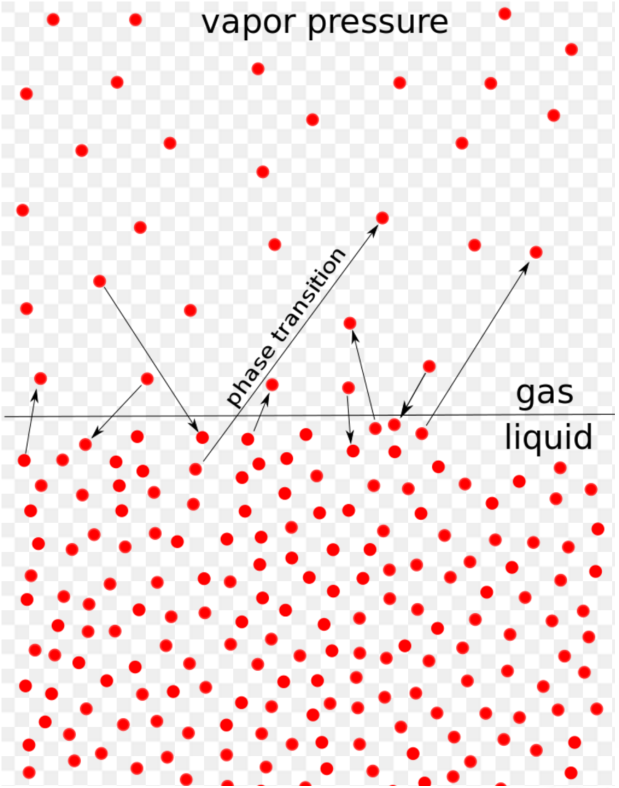
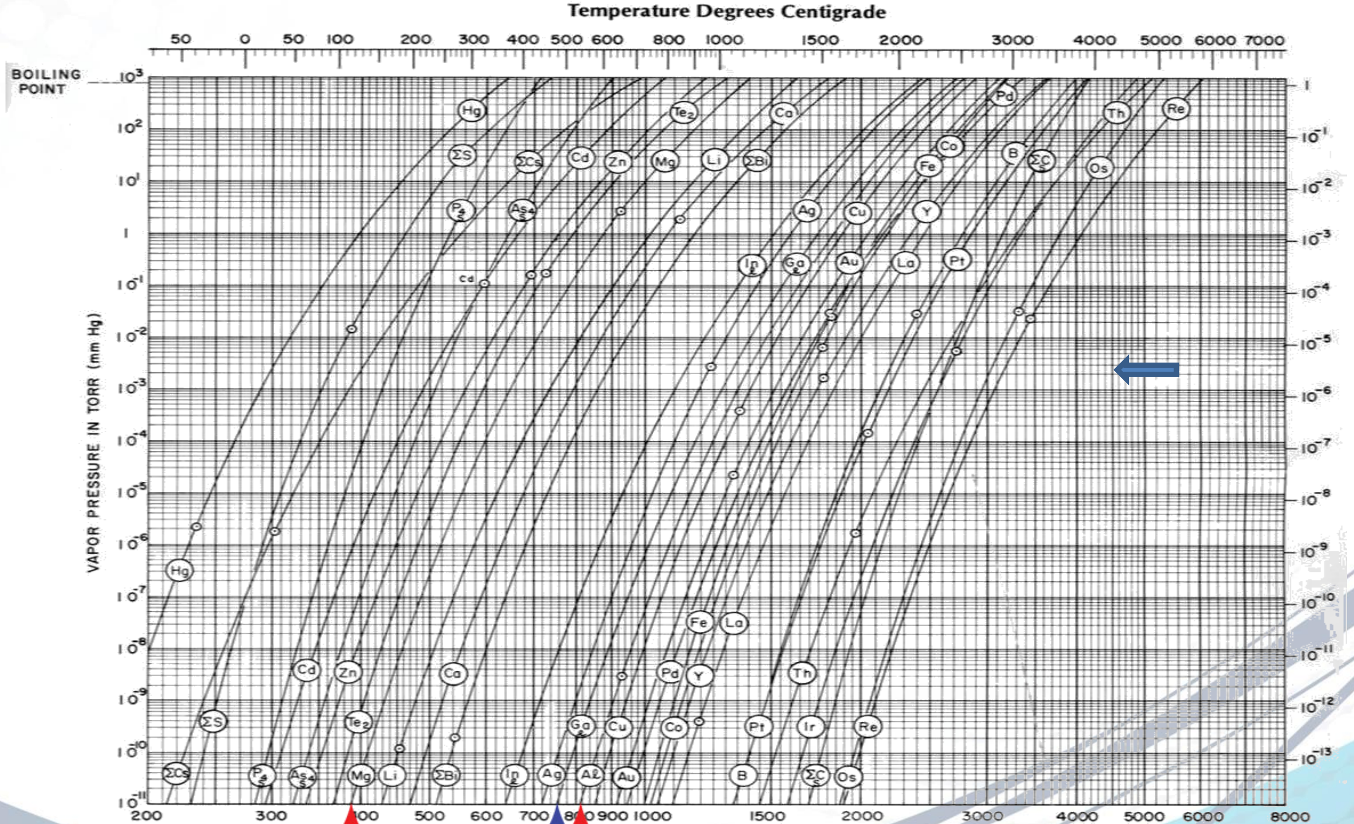
MATERIALS NOT FOR USE IN UHV

- Cadmium (Typically in the form of plating)
- Zinc (may be present in other materials such as brass)
- Magnesium
- PVC and most plastics
- Lead (which is in most solders)
- Anything wet such as oils or lubricants
- Paints
- Any high-outgassing, low melting point materials
- Circuit Boards
- Insulated multi-stranded wires

Solid/Gas interfaces based on Pressure and Temperture



Honig's Equilibrium Vapor Pressure Curves



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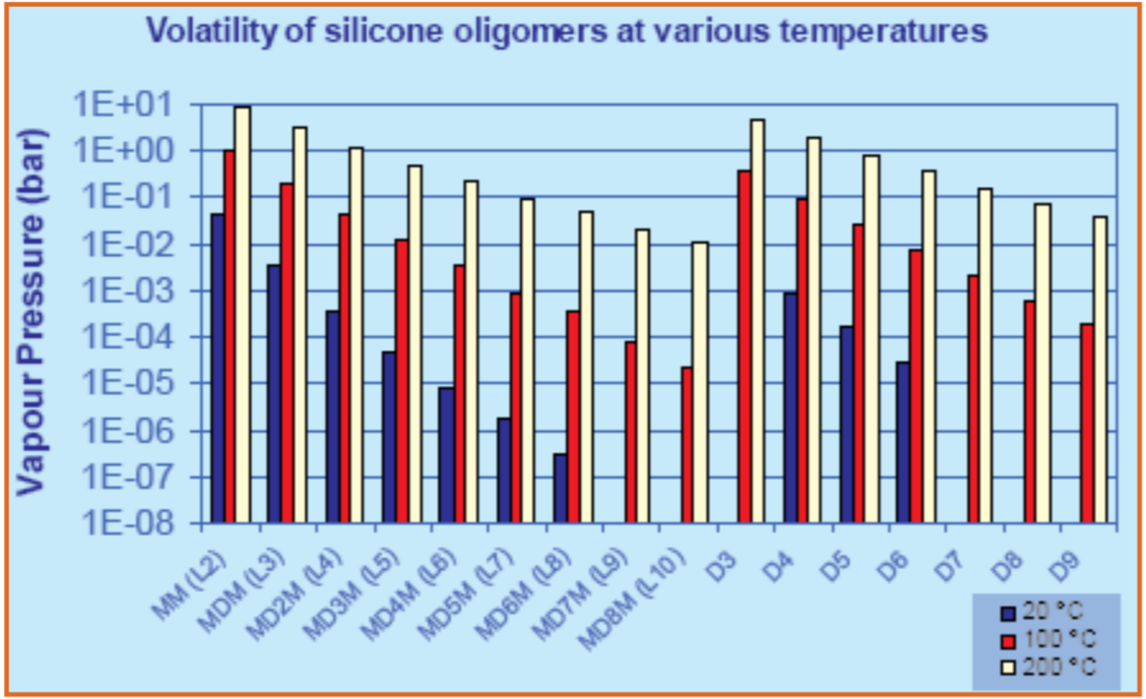
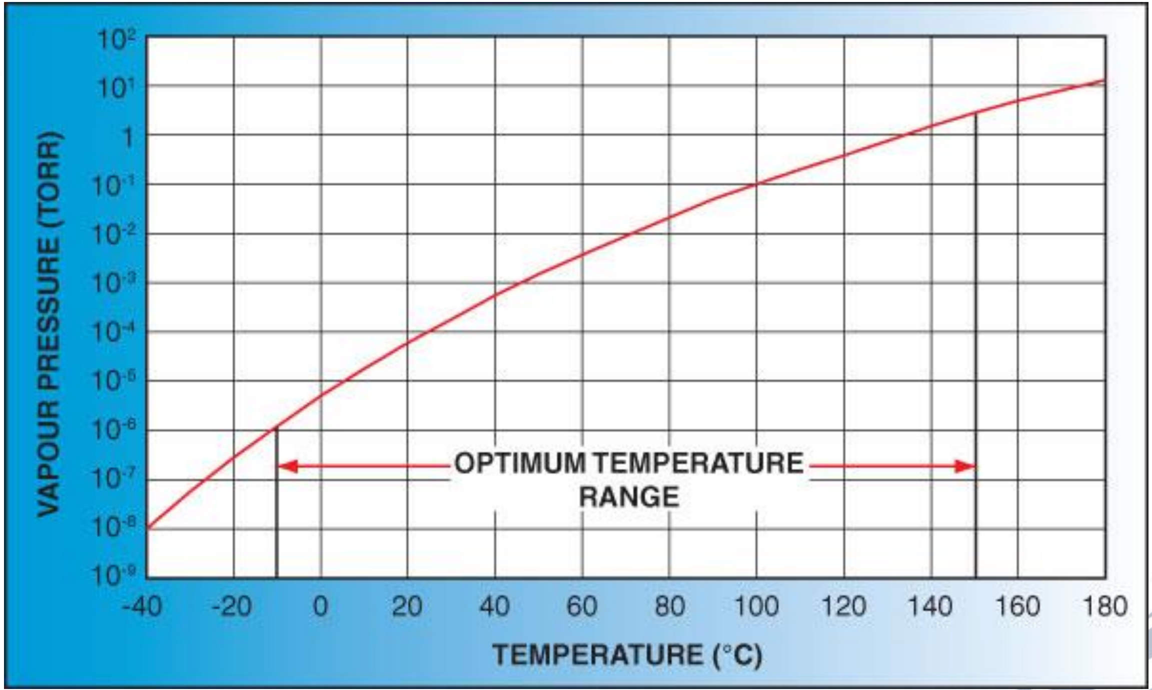
Vapor Pressure of Silicones



Vapor Pressure of Vacuum Lubricants



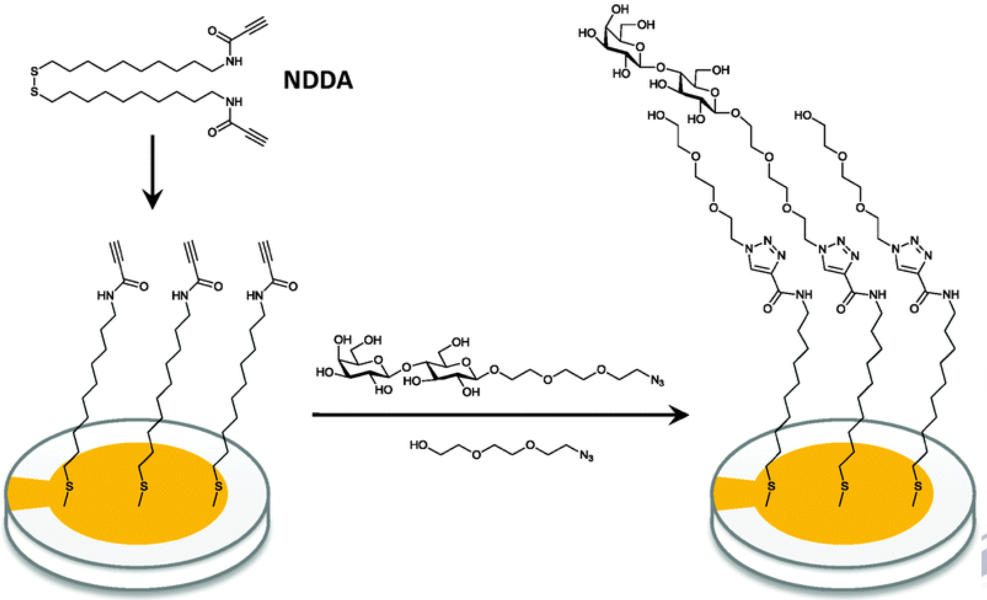
Solids to Gas

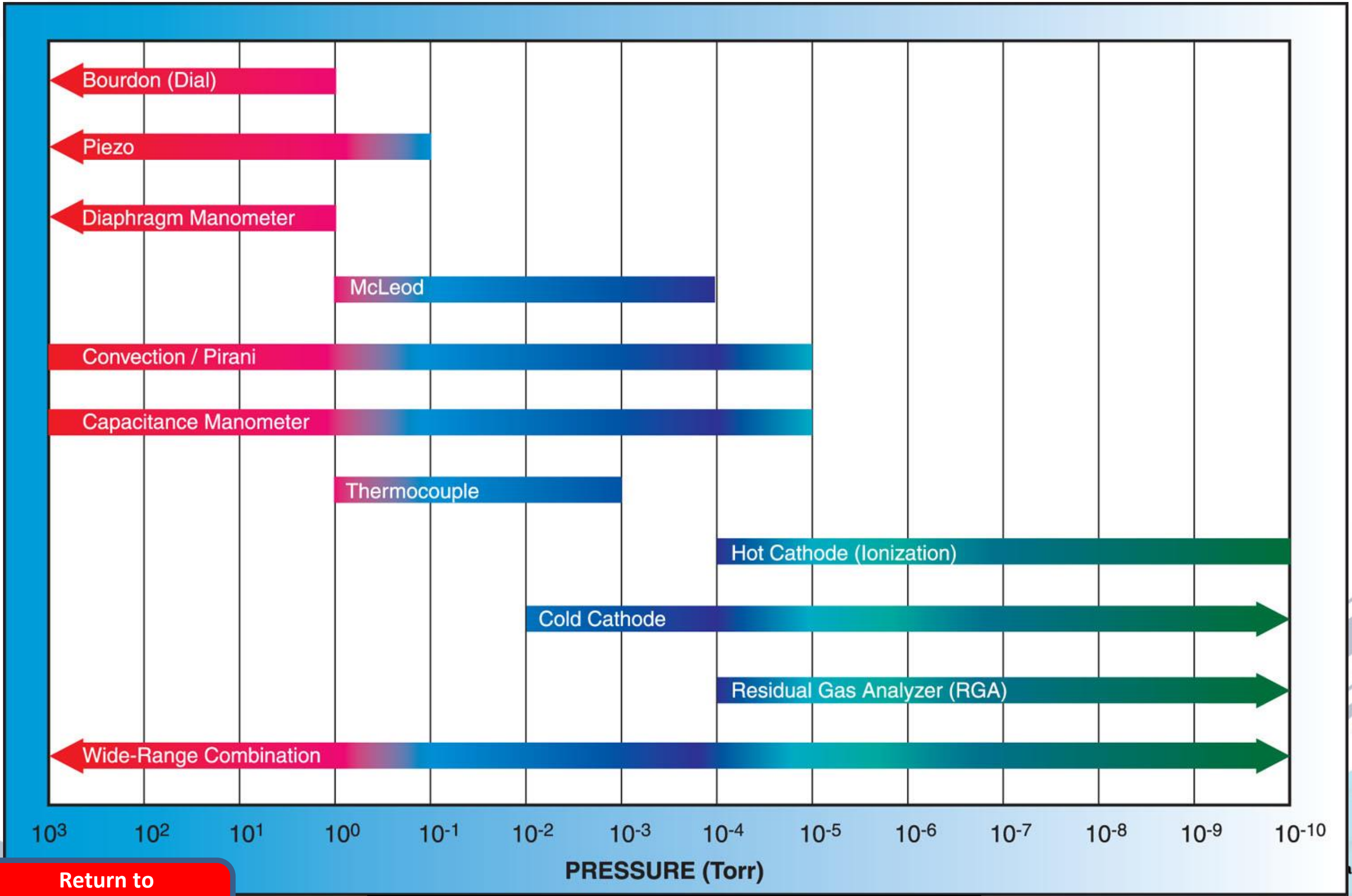
Chemical Name or Trade Name	Melting Point °C (°F)	Vapor Pressure at 20°C (70°F) mbar (mm Hg)	Remarks
Glyptal, red	[a]	$< 5 \times 10^{-6}$	Used for temporary thread and joint seals.
Grease, Apiezon N	43 (109.4)	10^{-3} at 200°C (392°F)	Maximum Temperature 30°C (86°F)
High Vacuum Grease (Dow Corning)	[a]	$< 10^{-6}$	Common vacuum sealing component used on "O"-rings and similar devices.
Nylon (external)	[a]	$\approx 10^{-5}$	Insulators
Oil, Apieson J	[a]	10^{-3} at 250°C (482°F)	Moderate viscosity
Rubber, natural	[a]	$\approx 10^{-5}$	Gasket material
Rubber, synthetic	[a]	$\approx 10^{-5}$	Gasket material
Teflon	[a]	$< 10^{-6}$	Insulators and gaskets
Vacuseal	50 – 60 (122 – 140)	10^{-5}	
Viton	[a]	10^{-8}	Gasket material
Wax, Apiezon W	85 (185)	10^{-3} at 180°C (356°F)	Permanent joints; maximum temperature 80°C (176°F)

Table 3[1] – Characteristics of Selected Solids in Vacuum

Notes: [a] Data not available.

Application of Quartz Crystal Monitor (thickness monitor)





Return to Application Issues Issues

Chart is on Page 7-2 of Catalogue

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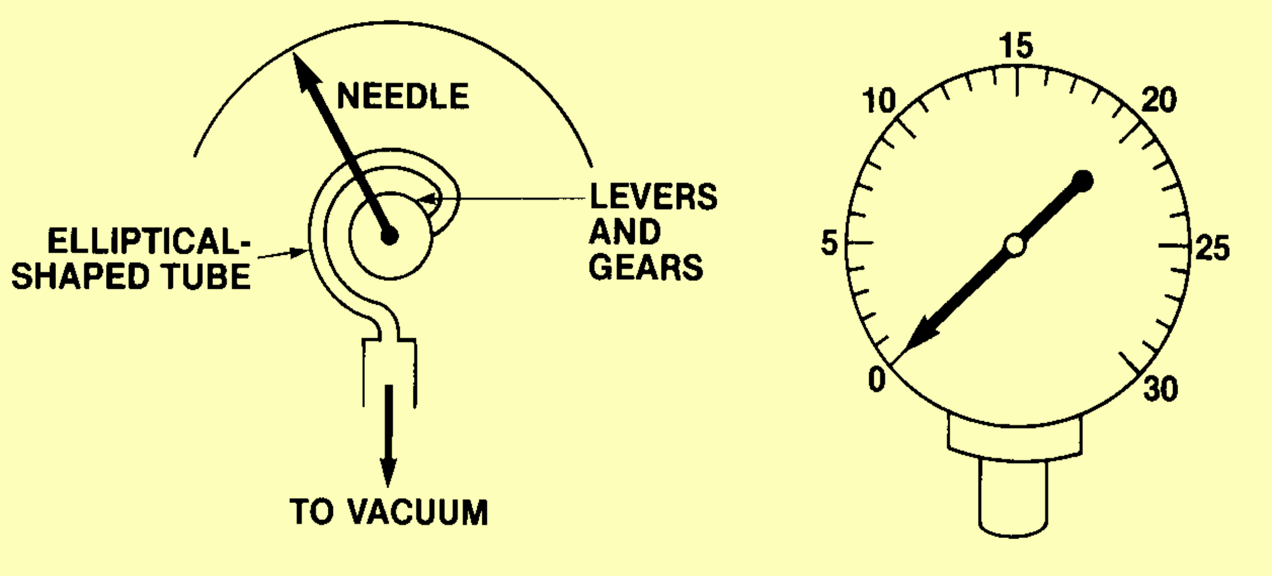
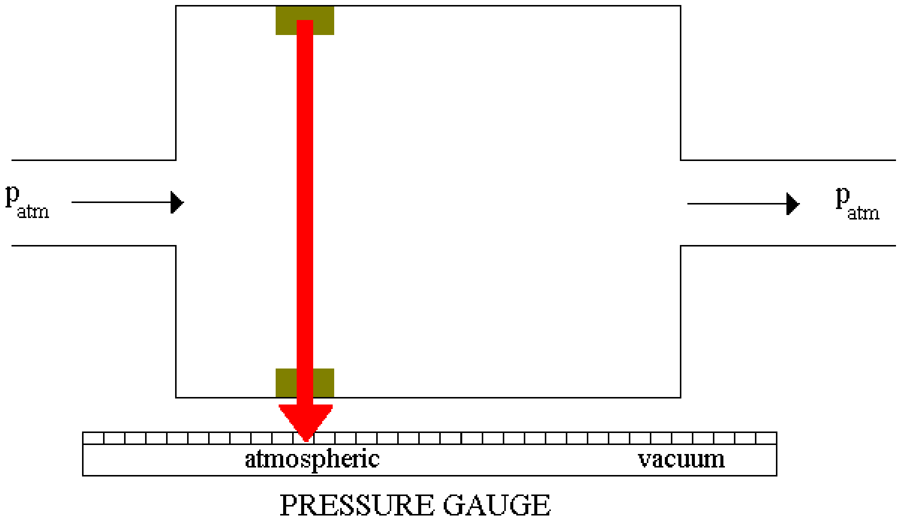
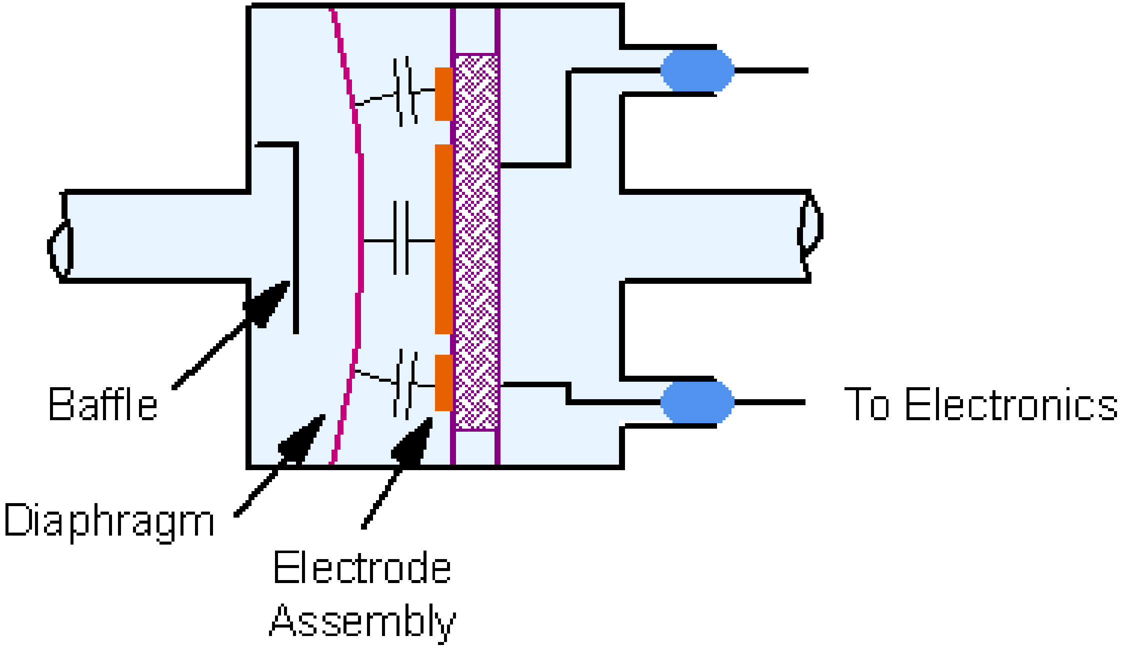
Mechanical phenomena How the gauge works



Mechanical phenomena Pressure Gauge

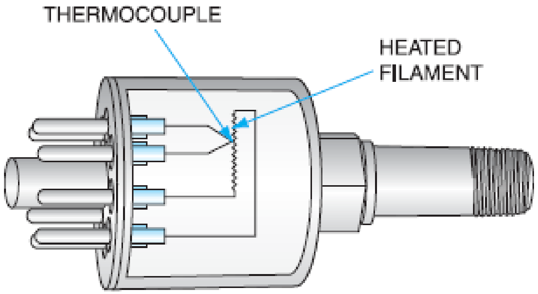


"Cap-Man" internals



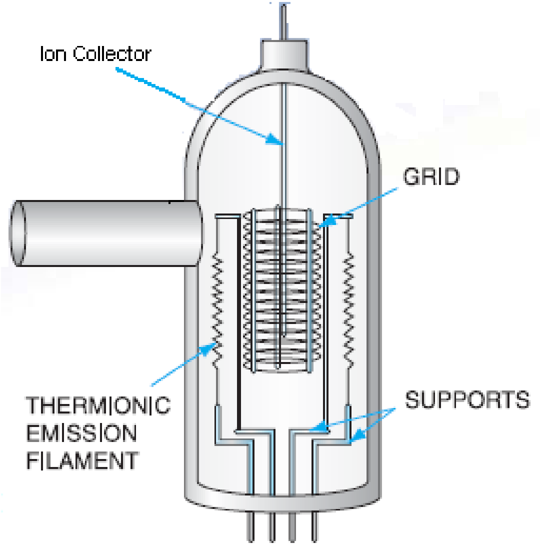
Thermocouple

- Range
  - 760 – 1x10^{-3} Torr range
- Accuracy
  - Range dependant, good to poor (explained more later)
- Operating Principle
  - Heat loss from filament wire (measured by thermocouple)
- Notes
  - Affected by gas composition; poor precision, not bakeable
  - Widely used in industrial and production applications



Hot Ionization

- Range
  - $5x10^{-2}$ to $1x10^{-9}$ Torr range
- Accuracy
  - poor – plus or minus 20% to 50% of reading
- Operating Principle
  - Electrons from filament ionize gas - ion current measured
- Notes
  - Low cost, “best game in town”
  - Affected by gas composition; glass; hot filament sensitive to gases; calibration not stable



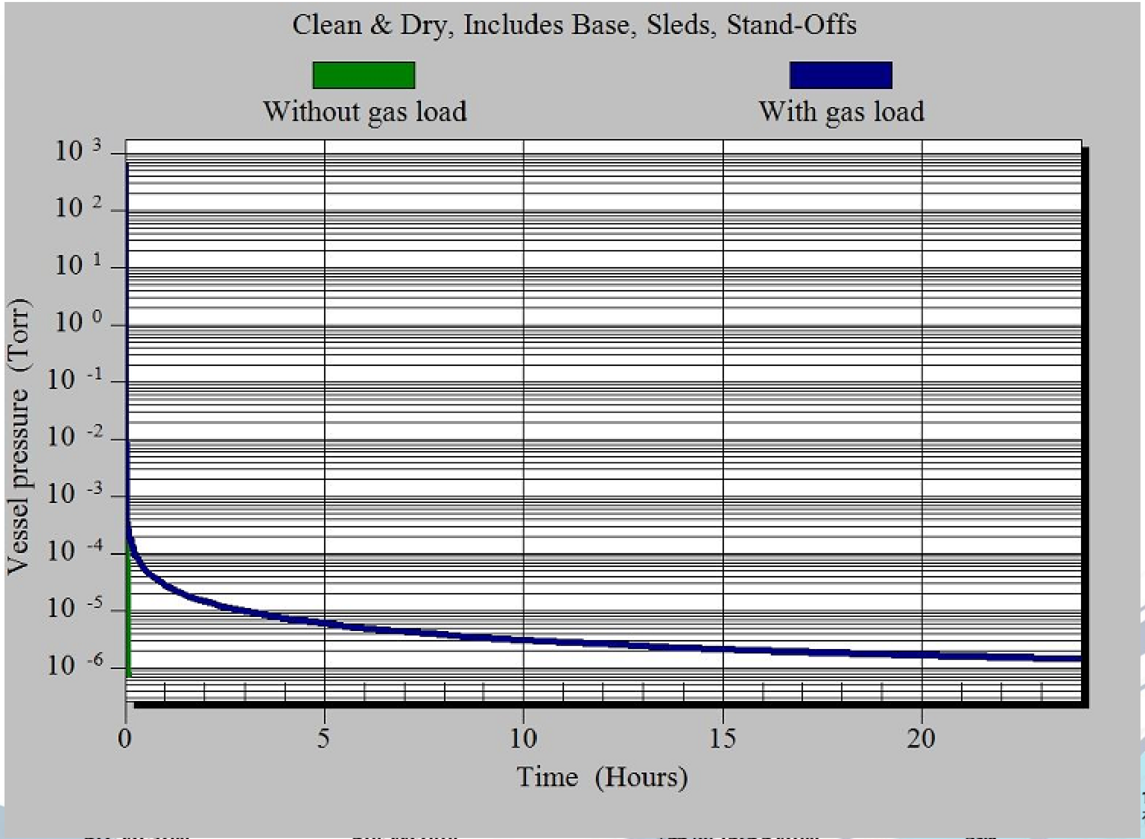
Real World Case on Contamination issues in a Space Simulation System for CubeSats

System Features Effecting Vacuum Performance

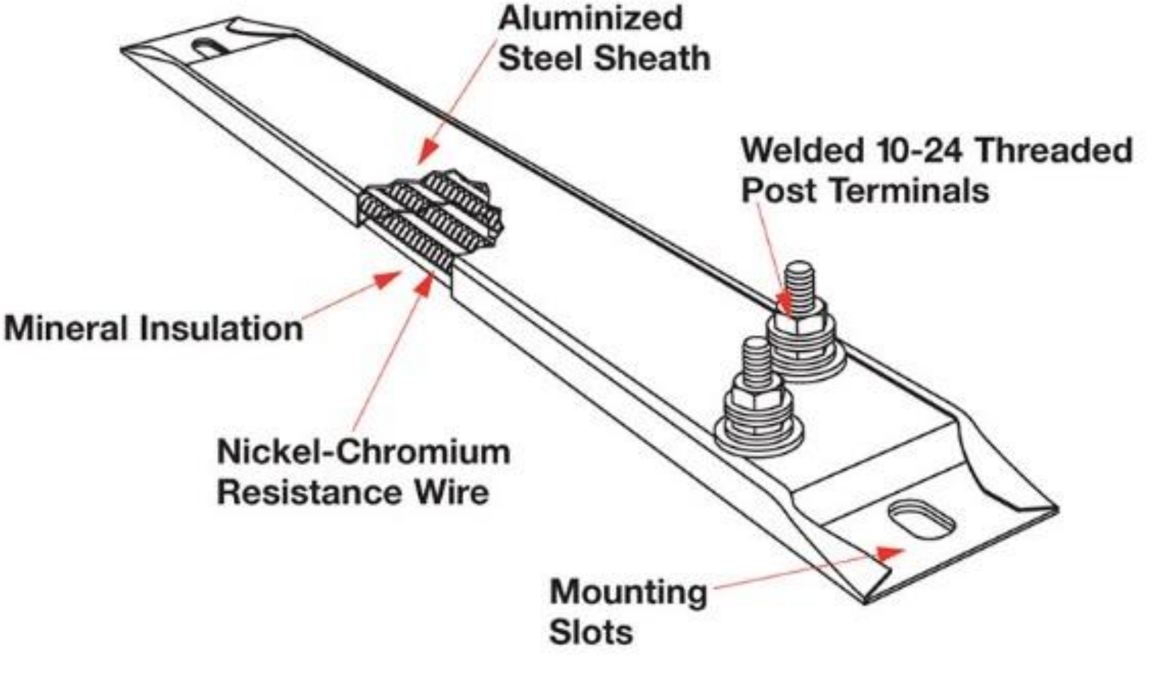
- Aluminum base plates 22 x 22 x 0.5”
- Four aluminum sleds at 4 x 2 x 12”
- 20 feet of PTFE insulated wire
- Four PTFE stand-offs 1” diam x 0.75” thick
- Four strip heaters – 1.5 x 14” - see image

Suspect Satellite

Modeling a "Clean & Dry" system

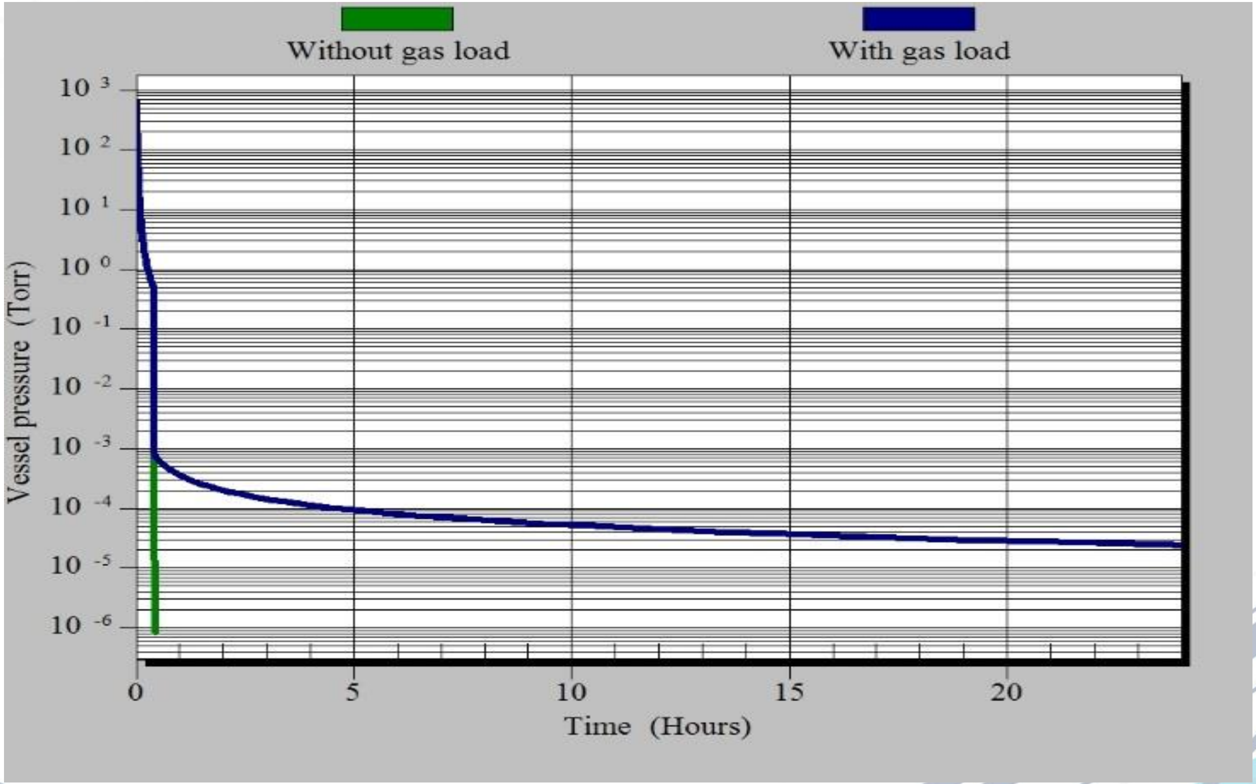


Strip heaters in vacuum space

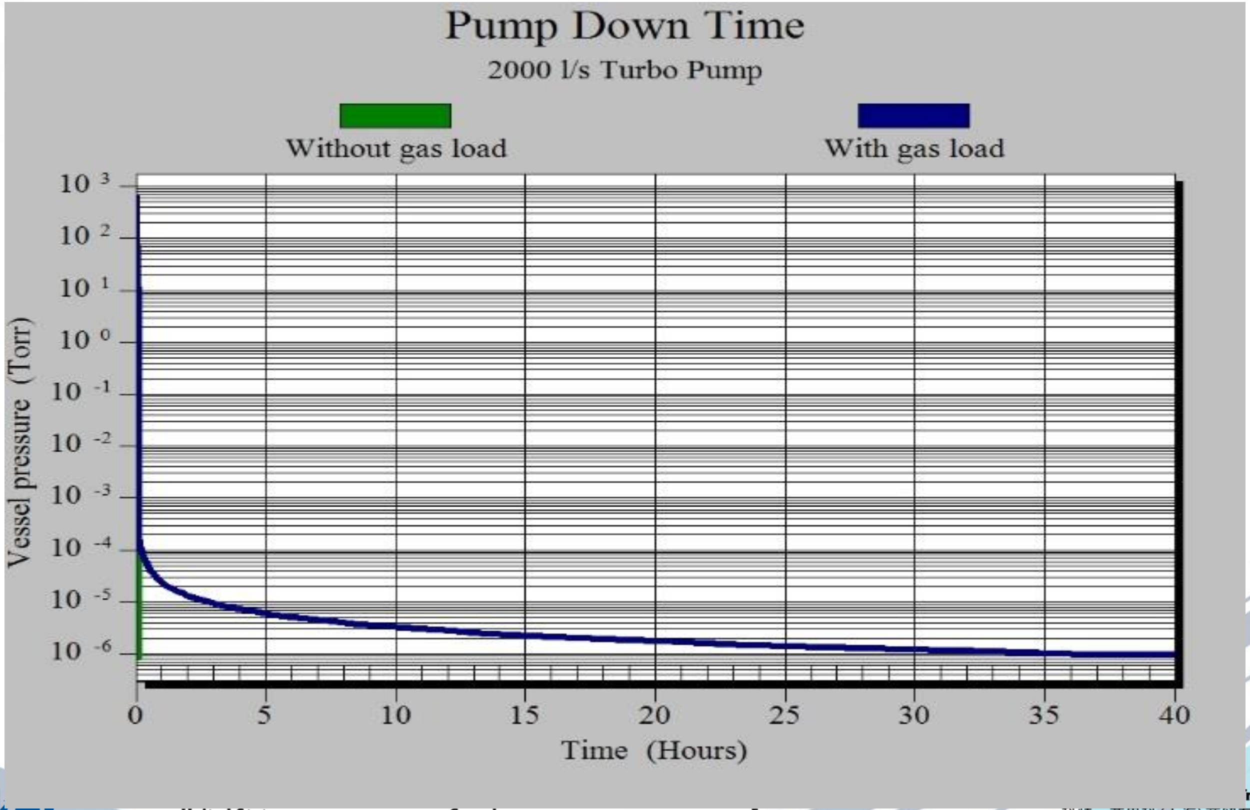


Assembling by Hand!

Actual performance with 150 L/s pumping stack



Simulation with 2,000 L/s pump stack



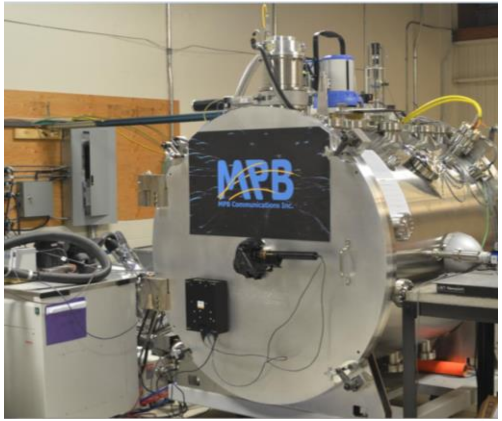
Other Interesting (dust) Samples

	JSC-1 (mean of 3)				Lunar Soil 14163*	
Oxide	Conc.		Std. Dev.		Conc.	
		Wt %		Wt %		Wt %
SiO2		47.71		0.10		47.3
TiO2		1.59		0.01		1.6
Al2O3	15.02		0.04		17.8	
Fe2O3		3.44		0.03		0.0
FeO		7.35		0.05		10.5
MgO		9.01		0.09		9.6
CaO		10.42		0.03		11.4
Na2O	2.70		0.03		0.7	
K2O		0.82		0.02		0.6
MnO		0.18		0.00		0.1
Cr2O3		0.04		0.00		0.2
P2O5		0.66		0.01		---
LOI		0.71		0.05		---
Total		**99.65**				**99.8**

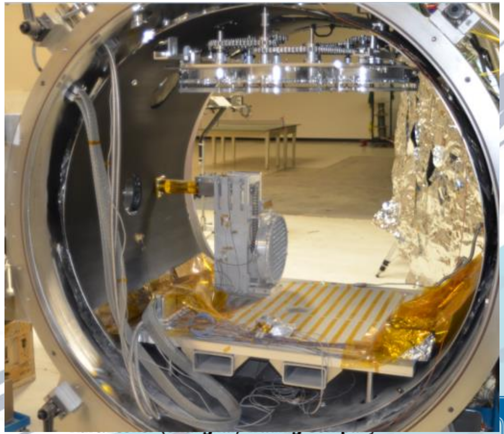
Martian Regolith
(Viking Lander Data)

- Average Density
  1.4 g/cm^3
- Chemical Composition
  58.2% SiO_2
  23.7% Fe_2O_3
  10.8% MgO
  7.3% CaO

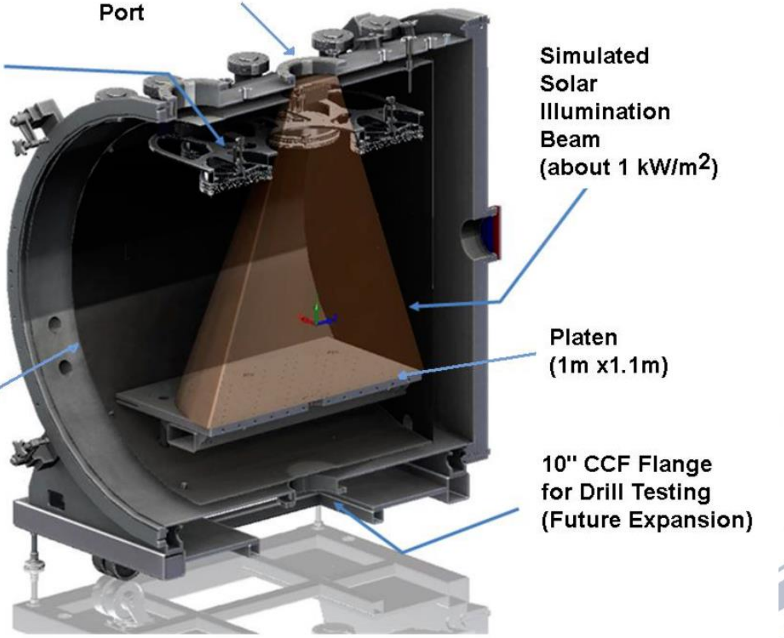
Dusty Thermal Vacuum Chamber (DTVAC) Overview



- DTVAC offers the simulation of lunar/Mars atmospheric pressures, temperatures, solar illumination and charged dust
- Vacuum Pressures:
  - < 10^{-7} Torr (1.33 10^{-5} Pa) without dust
  - < 10^{-5} Torr (1.33 10^{-3} Pa) with dust dispensing
- Recirculating chiller/heater with interchangeable coolants (Thermal C2 or C5):
  - Shroud and Platen heating to above $+60^{o}$C ($+120^{o}$C with C5 coolant)
  - Shroud and Platen cooling to below -60^{o}C (1.6 kW capacity)
  - Planned Upgrade for Liquid Nitrogen (80K)

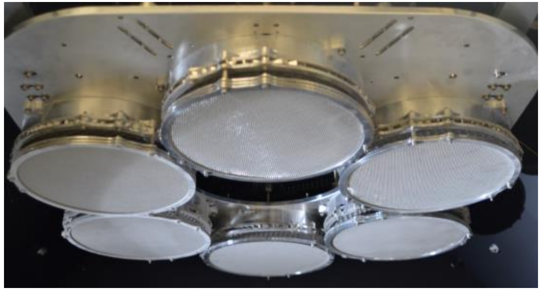


It’s Raining Mars Dust



DTVAC Unique Features

- Dust Dispersion with various lunar or Mars simulants (variable dispersion rate)
- Solar Illumination (variable intensity)
- Most of the vacuum ports protected against dust by internal baffles
- 25 Type-K Thermocouples
- Unit Under Test (UUT) accommodation up to 0.9 m^3 in volume (platen surface 1.1 m x 1.0 m)
- Rotary, Surface and Optical UUT testing capability
- Platen coverage by regolith simulant of up to 500 kg



Real Challenges

- Lots of surface area and in 3 dimensions
- Water and all it carries, stick to everything and are also very sticky
- Delicate (and highly pricey) components
- Limited approaches for Particulate, Hydrocarbon and Microbial elimination

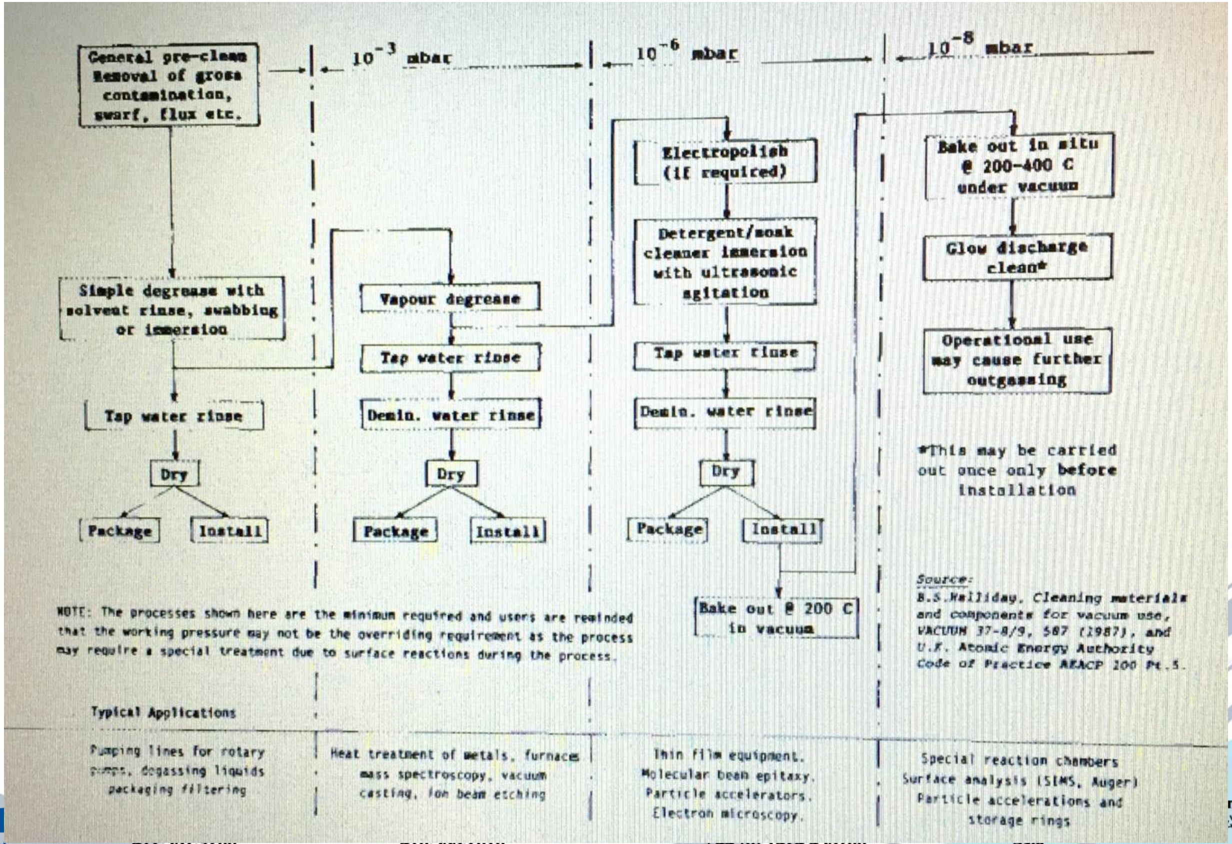
Sources of Gross Contamination

- –Finger prints
- –Oils
- –Cutting fluids
- –Greases
- –Solvents
- –Gunk from Payloads

•May Require Complete Dis-Assembly

Vacuum Layout can be Complex

Chamber Cleaning

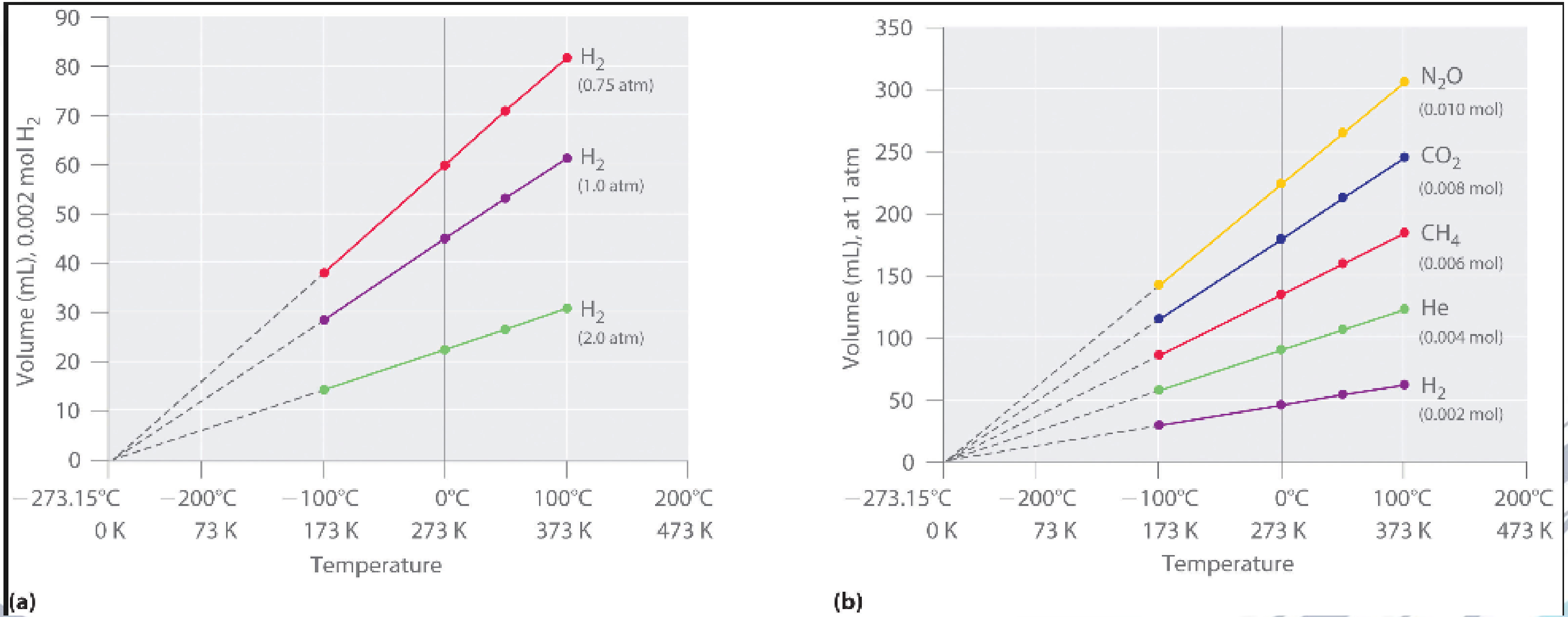


Multi-Step Chamber Cleaning

Outgassing Reduction by Chamber Cleaning

- Wear appropriate protective gear
- Mechanical abrasion – like bead blasting or hand rubbed with alumina-impregnated Scotch Brite
- Hot water wash
- Vapor bath with alkaline solvent
- Second hot water wash
- Blow dry or air dry at +100 C

Temperature and Pressure



Bacteria Surviving in Space

Detecting Microbial Contamination

- RGA – Atomic Mass Units?
- Black light
- Microbial detection sensors ?

Killing Microbial Contaminants in 3 dimensions

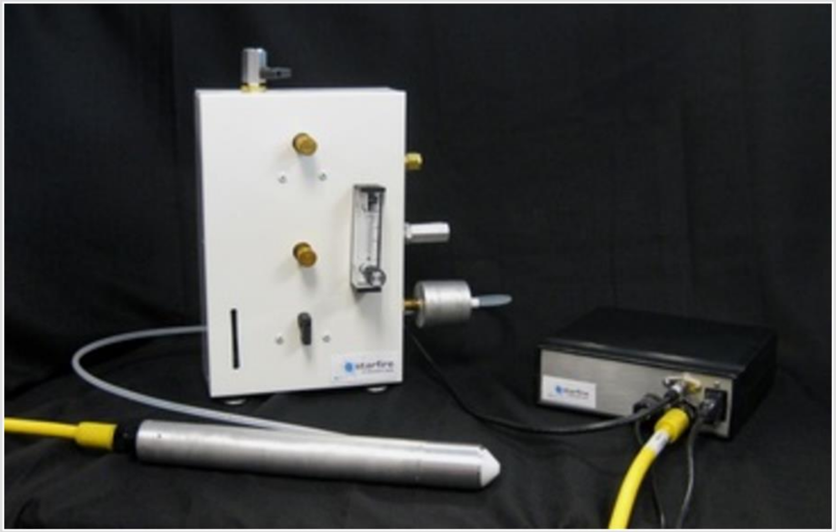
- **Autoclave - +121 C, several minutes**
- **UV Light – quick, line of sight**
- **Atmospheric plasma – quick, line of sight**
- **E-Beam or Vacuum Plasma – quick, special conditions**
- ***Low Temperature Hydrogen Peroxide - +30 C, minutes***

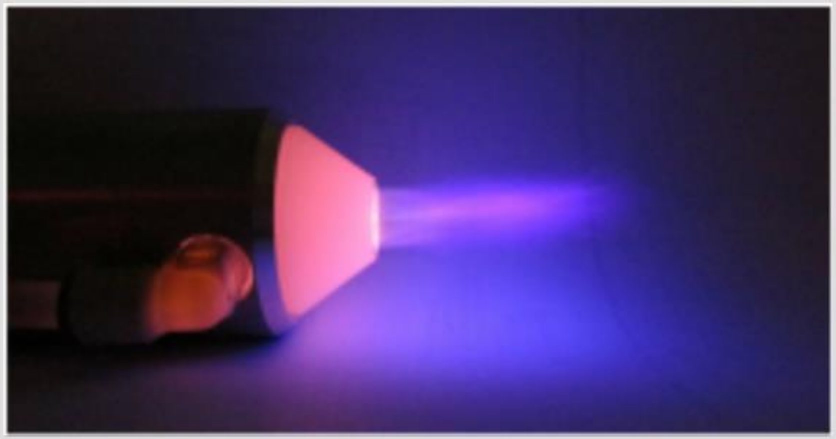
UV Light to Kill Microbials

5.1 Remote Inductively Coupled Plasma (ICP)

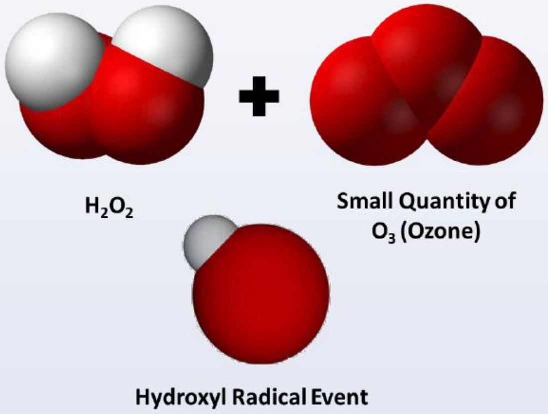
- ✓ 500 W Ar/O_2 plasma
- ✓ Cylindrical quartz plasma tube w/ helical inductive coil geometry
- ✓ 13.56 MHz frequency

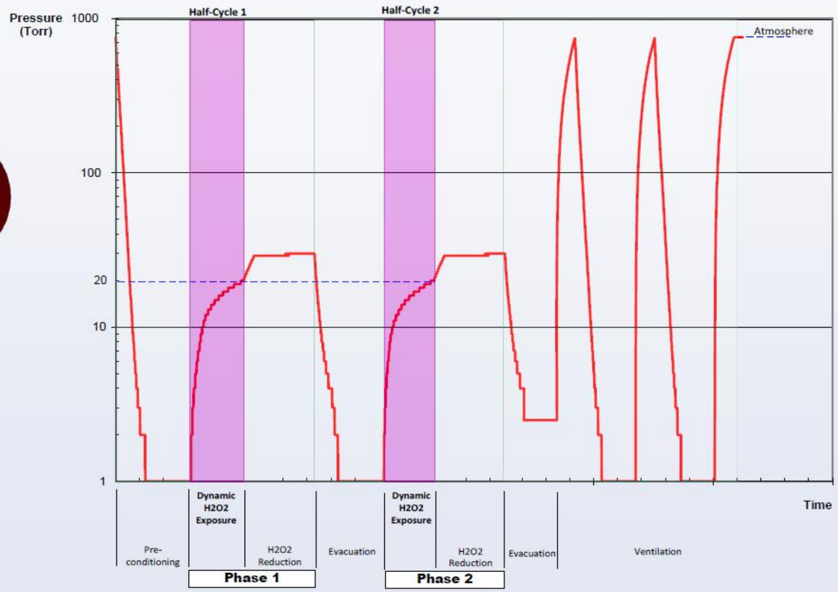
Atmospheric Plasma for Cleaning





Low Temp Vacuum-Enabled Sterilization Process





Commercial Systems for Components

- terminal sterilization of heat- and moisture-sensitive medical devices.
- This sterilizer is intended for the reprocessing of general instruments, rigid channel instruments, single/multi-channel rigid endoscopes, as well as short and long single/multi-channel flexible endoscopes.
- It utilizes a small vacuum chamber, pumping systems, valve and pressure control as well as liquid/vapor injection

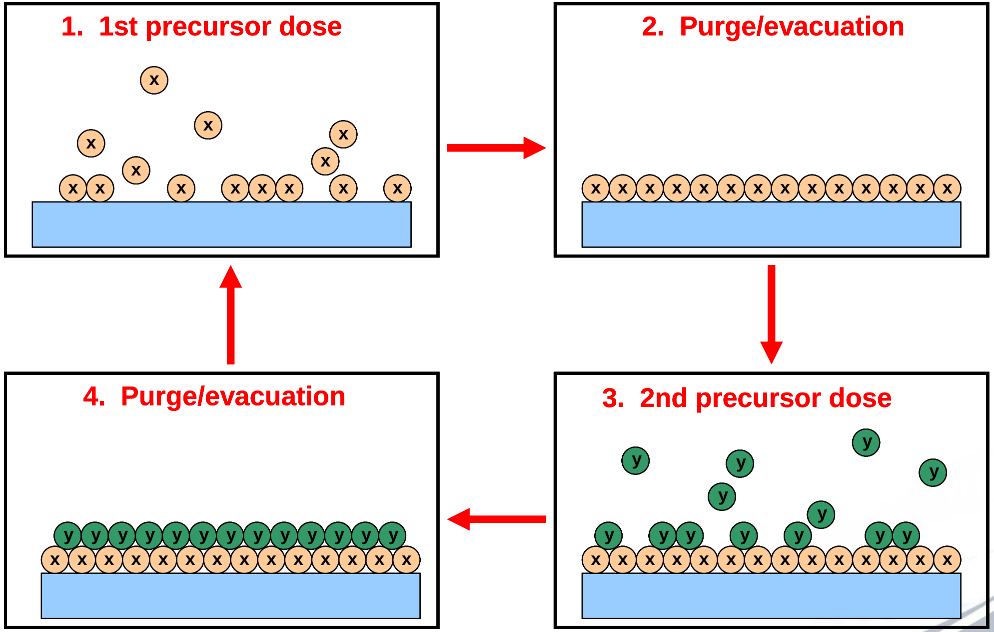
Containment/Encapsulation by Coatings

- Effective encapsulation of consumer electronics demonstrated with cell phones

- Surface driven, chemically reactive, 3-Dimensional coating approaches look good

- Examples of Atomic Layer Deposition on electronic components

Atomic Layer Deposition (ALD) for a binary material
Like alumina (Al_2O_3)



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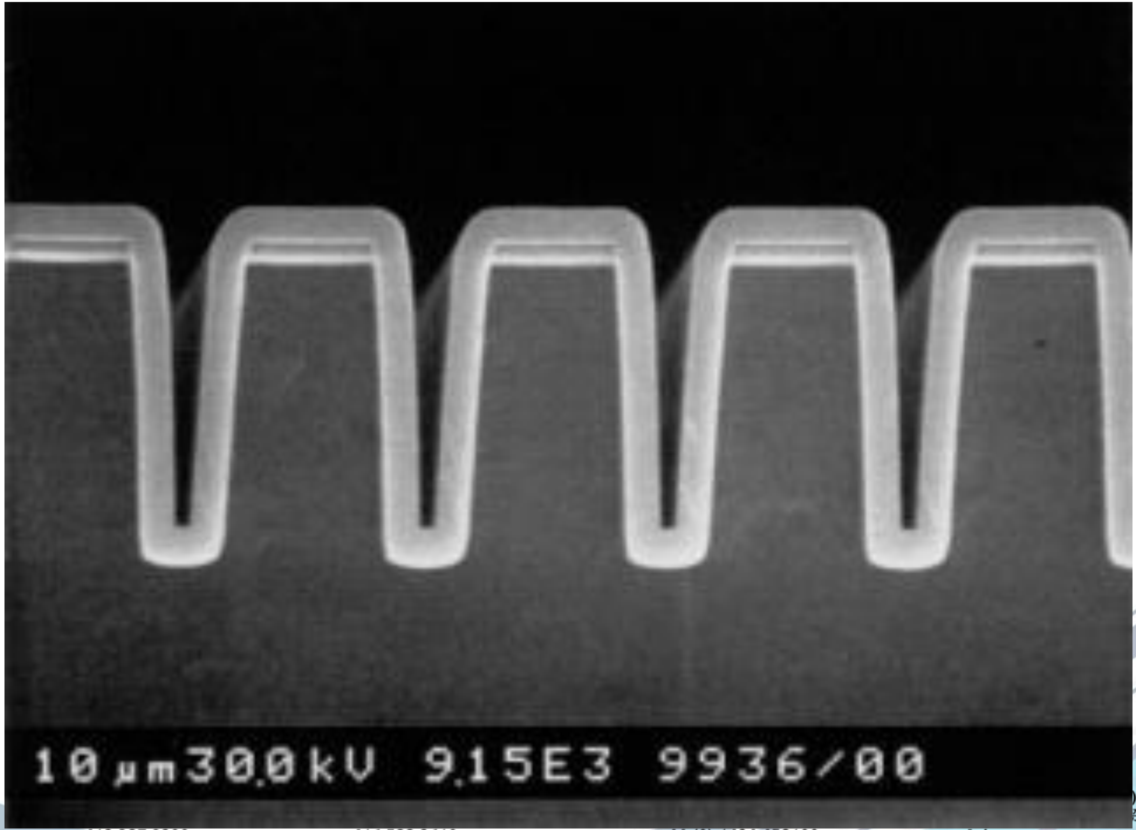
Highly Conformal, Deep trench



Recommendations

- Good vacuum practice
- Good vacuum system design
- Proper materials selection – system and samples
- Protective coatings – system and samples
- Pressure gauges/placement
- Protection from Humans, maintenance

- # THANK YOU!

- Questions?

- For more information on vacuum systems:
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