Vacuum Technology

Introduction to concepts that should be understood before designing, constructing, modifying, ‘assessing’ or, perhaps even using a vacuum system

Michael McKeown
Outline

Gas–solid surface interactions*
Basics of conductance & pumping
Gas load and its many sources
Outgassing & ways to reduce it
Pump throughput & keeping it high
Equating gas load & throughput
Modeling performance with VacTran

With KTG defines VT
Gas Load = total
Outgassing – major source
Gas-Surface Interactions

References

Overview
   Adsorption-Desorption, Diffusion & Permeation

Arrival at Surface
   Reflection & Sticking

Departure from Surface
   Cosine Distribution
## References

1. *Modern Vacuum Practice*  
   Harris

2. *Vacuum Technology*  
   Roth

3. *Foundations of Vacuum Science and Technology*  
   Lafferty

4. *The Physical Basis of Ultrahigh Vacuum*  
   Redhead

5. *A User’s Guide to Vacuum Technology*  
   O’Hanlon

6. *Vacuum Sealing Techniques*  
   Roth

7. *Handbook of Electron Tube and Vacuum Techniques*  
   Rosebury
Adsorption

‘Satisfying’ surface’s residual forces
Initially molecules of any type adsorbed
More polar molecules preferred

Reduced by higher temperature
Increased by lower temperature *
Desorption – (outgassing)

Opposite of adsorption
Molecule gains sufficient energy to overcome binding energy to peers or surface

Increased by higher temperature
Reduced by lower temperature
**Diffusion**

Gas trapped in bulk interstices  
Concentration gradient near surface  
Stainless steel: atomic H and CO

Increased by higher temperature *  
Reduced by lower temperature
Permeation

Gases permeate through all materials
Gas/Solid usually immeasurably slow*

Gas/Elastomer are measurable*

Permeation rate
\[ P \propto f(\text{partial pressure}) \]
\[ P \propto f(\text{specific for gas & elastomer}) \]
\[ P \propto f(\text{temperature}^q) \]
Molecules Arriving at Surface

1. Reflect back into gas phase with no energy exchange *
2. Reflect back into gas phase with energy exchange *
3. Trapped shallow minimum energy state: *
   Physisorption 0 – 10 kcals/mole
4. Trapped in deep minimum energy state:
   Chemisorption 20 – 100 kcals/mole
5. Chemically react:
   Heat of formation ~100 – 500 kcals/mole
Reflection of Light

Specular Reflection (smooth surfaces)

Diffuse Reflection (rough surfaces)
Gas-Surface Interactions 7

*Molecules Leaving Surface*

- Flux = 1
- Flux = 0.707
- Flux = 0

Cosine Distribution

45°
Reflection of Atoms/Molecules

L – dot Pt/RT He, circle hot Pt/RT He  Pt 1300°C  He 1800°C
R – dot RT Pt/hot He, circle hot Pt/hot He
Reflection of Atoms/Molecules at Large Angles

N2 - Glass
Adsorption of Atoms/Molecules

Accommodation coeff — reflection with energy exchange
Condensation coeff — adsorption into shallow minimum
Sticking probability — adsorption into deep minimum
## Accommodation Coefficients of Atoms/Molecules

<table>
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<tr>
<th>Gas</th>
<th>Substrate</th>
<th>Coefficient</th>
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</thead>
<tbody>
<tr>
<td>He</td>
<td>Ni (298K)</td>
<td>0.385</td>
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<tr>
<td>H2</td>
<td>Ni (298K)</td>
<td>0.249</td>
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<td>Ar</td>
<td>Ni (298K)</td>
<td>0.935</td>
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<tr>
<td>N2</td>
<td>Pt (?)</td>
<td>0.816</td>
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## Condensation Coefficients of Atoms/Molecules

<table>
<thead>
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<th>Gas</th>
<th>Substrate</th>
<th>Coefficient</th>
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<tbody>
<tr>
<td>He</td>
<td>glass at 50°C</td>
<td>0.17</td>
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<tr>
<td>H2</td>
<td></td>
<td>0.57</td>
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<tr>
<td>N2</td>
<td></td>
<td>0.76</td>
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<tr>
<td>O2</td>
<td></td>
<td>0.82</td>
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<tr>
<td>Ar</td>
<td></td>
<td>0.86</td>
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</table>
Gas-Surface Conclusions

Molecules hitting a surface:

- Do NOT reflect like light
- Stick (momentarily ► permanently)
- Desorb with cosine distribution

(Under vacuum: every solid surface desorbs gas)
Basic Pumping Concepts

Flow Regimes

Conductance

Pumping Speed

Conductance plus Pumping Speed

‘Effective’ Pumping Speeds (EPS)

Measuring EPS
Flow Regimes

\( mfp \ -- \ mean \ free \ path \)

Continuum Flow: molecules’ mfp is small compared to characteristic dimensions of vacuum volume

Transitional Flow: molecules’ mfp roughly equal to characteristic dimensions of vacuum volume

Molecular Flow: molecules’ mfp is large compared to characteristic dimensions of vacuum volume

Flow regime (ie pressure) affects conductance
**Conductance**

Passive Components

Ability to transfer gas *volume* in unit *time*

Determined by shape, open area, length, gas, & pressure

*(Volumetric flow measured in: L/s, cfm, m³/hr, L/m)*
Calculating Conductance

- Dushman’s Table
- Transmission Probability
- VacTran®

<table>
<thead>
<tr>
<th>a (cm)</th>
<th>F_o</th>
<th>F_t - Conductance of tube (liters sec⁻¹) for air at 25°C</th>
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<tbody>
<tr>
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<td></td>
<td>L/a = 1  2  4  8  12  16  30</td>
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<tr>
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<td>K = 0.672 0.514 0.350 0.232 0.172 0.137 0.080</td>
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<td>0.1</td>
<td>0.367</td>
<td>0.246 0.188 0.132 0.085 0.063 0.050 0.029</td>
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<td>3.300</td>
<td>2.217 1.664 1.184 0.764 0.567 0.451 0.263</td>
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<td>13.200</td>
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<td>1208.000 922.800 644.900 416.100 308.800 245.700 143.200</td>
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<tr>
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<td>2347.000</td>
<td>1577.000 1205.000 842.400 543.600 403.300 320.800 187.100</td>
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<tr>
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<td>1996.000 1525.000 1066.000 687.900 510.500 406.100 236.800</td>
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<td>3666.000</td>
<td>2464.000 1883.000 1316.000 849.200 630.200 501.300 292.200</td>
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</tbody>
</table>

Aperture, long ducts (1/L), short ducts
Pumping Concepts 4

**Conductance vs Pressure**

**Tubes**
(with no losses)
Conductance vs Pressure

Tubes
(with entrance loss)
Conductance vs Pressure

Tube vs Elbow (of equal ‘length’)
Conductance vs Pressure

Elbow vs Elbow (radius vs miter)
Pumping Concepts 8

*Pumping Speed*

Active Components (Pump/Trap)

Ability to transfer (remove) gas volume in unit time

Determined by gas, pump’s mechanism, and pressure

(*Volumetric flow measured in: L/s, cfm, m³/hr, L/m*)
Pumping Concepts 9

Pumping Speed vs Pressure

![Graph showing pumping speed vs pressure for different stations. The y-axis represents Station Pump Speed (liters/second) ranging from 0 to 70, and the x-axis represents Pressure (Torr) ranging from $10^{-3}$ to $10^{3}$. Three lines correspond to different stations: Aixzen, Pfeiffer_HIFace 80, and Leybold_W151.]
Combining Conductance with Pumping Speed

Units for both: volume / unit time

Combined as reciprocals

1/Conductance + 1/Pump Speed = 1/Effective Pump Speed

1/EPS = 1/PS + 1/C1 + 1/C2 + 1/C3
Effective Pumping Speed

500 L/s pump & infinite conductance

\[
\frac{1}{\text{EPS}} = \frac{1}{500} + \frac{1}{\infty}
\]

\[
\frac{1}{\text{EPS}} = \frac{1}{500}
\]

\[
\text{EPS} = 500 \text{ L/s}
\]
Effective Pumping Speed

500 L/s pump & 500 L/s conductance

\[
\frac{1}{\text{EPS}} = \frac{1}{500} + \frac{1}{500} = \frac{2}{500}
\]

\[
\text{EPS} = 250 \text{ L/s}
\]
Effective Pumping Speed

500 L/s pump & 50 L/s conductance

\[ \frac{1}{\text{EPS}} = \frac{1}{500} + \frac{1}{50} \]
\[ \frac{1}{\text{EPS}} = \frac{11}{500} \]
\[ \text{EPS} = 45 \text{ L/s} \]
Effective Pumping Speed

5000 L/s pump & 50 L/s conductance

\[
\frac{1}{\text{EPS}} = \frac{1}{5000} + \frac{1}{50}
\]

\[
\frac{1}{\text{EPS}} = \frac{101}{5000}
\]

\[
\text{EPS} = 49.5 \text{ L/s } *
\]
Effective Pumping Speed

HP700 + 160mm ID x 10cm tube
Effective Pumping Speed

Delivered Speed vs Pressure

HP700 + 160mm ID x 20cm tube + miter elbow
Effective Pumping Speed

CTI-8: tube 3.92” ID x 2” long
Measuring EPS 1 (i)

1. Measure chamber (base) pressure P1
2. Inject known mass flow rate of gas
3. Measure new (working) pressure P2
4. Calculate pressure difference P2 – P1
   (Convert mass flow units as needed)

Mass flow / Pressure Difference = EPS
**Measuring EPS 1 (ii)**

1. Base pressure $P_1$  \quad 5x10^{-7} \text{ Torr}
2. Mass flow ($N_2$)  \quad 10 \text{ sccm}
3. Working pressure $P_2$  \quad 5x10^{-5} \text{ Torr}
4. $P_2 - P_1$  \quad 4.95x10^{-5} \text{ Torr}

\[
10 \text{ sccm} \times \frac{10}{60} \times \frac{760}{1000} = 1.27 \times 10^{-1} \text{ T.L/s}
\]

EPS  \quad 1.27 \times 10^{-1} \text{ T.L/s} / 4.95 \times 10^{-5} \text{ T}

EPS ($N_2$)  \quad 1600 \text{ L/s}
Measuring EPS 2 (i)

1. Estimate chamber volume \( V \)
2. Inject unknown flow of gas
3. Measure (working) pressure \( P_1 \)
4. Time pressure decay
   - At time = 0 shut off gas flow
   - At time = t sec measure pressure \( P_2 \)

\[
EPS = \frac{V}{t} \times \log_e \left( \frac{P_1}{P_2} \right)
\]
Measuring EPS 2 (ii)

1. Chamber volume 150 L
2. Inject unknown flow of gas
3. Working pressure 4x10^{-4} Torr
4. Time 15 sec
5. Pressure (t = 15 s) 6x10^{-6} Torr

\[
\text{EPS} = \frac{V}{t} \times \log_e \left(\frac{P_1}{P_2}\right)
\]

\[
\text{EPS} = \frac{150}{15} \times \ln\left(\frac{4 \times 10^{-4}}{6 \times 10^{-6}}\right)
\]

EPS 42 L/sec
Conductance & Pumping Speed Conclusions

- **High conductance**: shorter/fatter is better
- **Conductance is too high**: (I wish!)
- **Conductance is too low**: serious (and expensive!)

- **The lowest conductance** wins
- **Pump’s `quoted´ PS**: means very little

**Think: Effective Pumping Speed**
Gas Load

Meaning & Units

Sources of Gas Load
Gas Load

The total *mass* (quantity/amount) of gas entering the vacuum volume in a given time period

*Mass flow measured in: T.L/s, Pa.m³/s, mbar.cc/s, atm.cc/s, sccm (pressure x volume / time)*
Gas Load - Sources

Permeation
Real leaks: from air / not from air
Virtual leaks
Backstreaming
Diffusion
Sublimation/Evaporation
Injected gas*
Outgassing
Gas Load 3

**Gas Load — Permeation**

- Air / H₂O and o-rings
- Air / H₂O and plastic gas line tubing *
- H₂O and Teflon insulators *
- H₂O and plastic cooling lines (in chamber) *
- H₂ / He and glass/silica tubing
- H₂ / CO and high temp. metal tubes *

Permeation rates through o-rings depend on: gas, elastomer, elastomer compounding, partial pressure, temperature.

Reducing permeation? *
Gas Load 4

Gas Load vs Time

Time (Hours)

Gas Load (Torr Liters/Second)

- vItem (high value for nitrogen)
- vItem (high value for oxygen)
- Buna-N (High value for Nitrogen)
- Buna-N (high value for Oxygen)
- vItem (water)
- Buna-N (water)
Gas Load — Real Leaks (air)

- Gaskets at flanges/joints
- Re-welds (in high carbon SS)
- At welds after baking/cleaning
- At welds in cryo conditions
- Blank flanges cut from bar
- Feedthroughs:
  - braze faults/cracked ceramics
  - porous deep drawn weld lips

Detecting leaks?

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Gas Load 6

**Gas Load**—*Real Leaks (not air)*

- Needle valves connected to gas source
- Leaking shut-off valves connected to gas source
- Leaking valve on shut-off mass flow controller
- Gas/water circulation lines inside chamber with cracked tube or leaking joint

*Detecting (not air) leaks?*
Gas Load — Virtual Leaks

Blind tapped holes / non-vented hardware
Multi-strand wire with plastic insulation
Flat surfaces clamped together
Welds on air-side surfaces

Test for virtual leaks?
Gas Load — Backstreaming

Oil vapor:  rotary vane & piston, diffusion, oil ejector
Methane, argon:  ion (and getter?)
Hydrogen, helium, neon:  turbo, cryo, molecular drag
Water vapor:  liquid ring, stream ejector

Check for backstreaming?
Gas Load 9

**Gas Load — Diffusion**

- H₂ & CO: stainless
- H₂: titanium* / palladium
- H₂O: glass
- VOMs: plastics

Pumping H₂ from Ti Bars
Gas Load 10

**Gas Load** — *Sublimation/Evaporation*

**Metals to avoid**
- Mercury, cadmium, zinc
- Cesium, rubidium, potassium, sodium
- Stainless steels (containing non-metals)

**Non-Metals to avoid**
- Phosphorus, arsenic
- Sulfur, selenium

Refer to VP charts
Outgassing

What is it? Units of measure
What are the worst sources?
Main components
Reducing outgassing
Outgassing

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

Outgassing Rate is difference between number of molecules:
- desorbing from the surface (in time ‘t’)
- absorbing on the surface (in time ‘t’)

Outgassing follows exponential decay.

Kurt J. Lesker Company
**Outgassing Rate**

Effective desorption rate from a given surface measured by rate-of-rise test (from a significantly large area)

- After preparation & cleaning in a repeatable way
- At a particular temperature
- After a specified time under vacuum (1 & 10 hours)
- From a specified area

Mass flow/unit area measured in:

\[
T.L\cdot cm^{-2}\cdot s^{-1}; \ mbar.L/cm^2.s; \ Pa.m^3/m^2.s; \ (W.m^{-2})
\]

(Pressure x volume / area x time)
Main Components

Water vapor
Oil/grease (‘hydrocarbons’)
Solvents
VOMs
H₂ and CO
'Other stuff'
Outgassing 4

**Main Components**

- H2
- H2O
- N2/CO/C2H4
- O2
- CO2
Worst Sources

Porous surfaces (ceramics or metals)
Plastics, elastomers, polymers
Previously backstreamed oil
Epoxy glues
Lubricating/sealing/heat transfer greases

and

Us!

(hair, skin cells, dust mites, spit, fingerprints, food)
Outgassing vs Time

1cm² Viton (fresh/baked) + 2000cm² SS (sanded)
Outgassing vs Time

2000 cm² anodized Al & 2000cm² sanded SS
Reducing Outgassing — 1st Steps

1. Make a log book and document everything!
2. Don’t put weird stuff in the chamber
3. Solvent clean everything (no plastic squash bottles) *
4. Vacuum bake before assembly (if possible)
5. Wrap it (in what?) until ready to mount
6. Never touch anything with bare hands!
Reducing Outgassing — 2nd Steps

Vacuum History
Heat
Light
Plasma
(Chemistry)
Reducing Outgassing — Vacuum History

Following initial pump-down, subsequent pump-down times depends on:

• Time at atmosphere when vented
• Venting gas & dryness of vent gas *
• Dry gas flowing while chamber vented *
• Application or process in chamber *
Reducing Outgassing — Heat

Outgassing 11

1L, 1L.s, 100cm$^2$
### Reducing Outgassing — Heat

<table>
<thead>
<tr>
<th>Material</th>
<th>Unbaked ($W/m^2$)</th>
<th>Baked ($W/m^2$)</th>
<th>Time/Temperature</th>
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</thead>
<tbody>
<tr>
<td>Stainless</td>
<td>$6 \times 10^{-7}$</td>
<td>$4 \times 10^{-9}$</td>
<td>30hr / 250°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$3 \times 10^{-10}$</td>
<td>2hr / 900°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$2 \times 10^{-11}$</td>
<td>3hr / 1000°C +</td>
</tr>
<tr>
<td>Aluminum</td>
<td>$6 \times 10^{-7}$</td>
<td>$5 \times 10^{-10}$</td>
<td>15hr / 250°C</td>
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<tr>
<td></td>
<td></td>
<td>$1 \times 10^{-11}$</td>
<td>GD &amp; 200°C</td>
</tr>
<tr>
<td>Copper</td>
<td>$5 \times 10^{-6}$</td>
<td>$2 \times 10^{-9}$</td>
<td>20hr / 100°C</td>
</tr>
</tbody>
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Reduction of Outgassing — UV Light

Graph showing the comparison of UHV pumpdown with and without UV light.
Reducing Outgassing — Plasma / Glow Discharge

Outgassing Mechanisms Include:

- UV stimulated desorption
- Electron stimulated desorption
- Ion bombardment
- Hot atom energy transfer
- Free radical ‘oxidation’
Pump Throughput

Equating Throughput & Gas Load

Flow Conversions & Calculations*
**Gas Load — reminder**

Total *mass* (quantity/amount) of gas *entering* the vacuum volume in a given *time* period

*Mass flow measured in: T.L/s, Pa.m³/s, mbar.cc/s, atm.cc/s, sccm (pressure x volume / time)*
Pump Throughput

Total mass (quantity/amount) of gas leaving the vacuum volume (via the pumps) in a given time period

Mass flow measured in: T.L/s, Pa.m$^3$/s, mbar.cc/s, atm.cc/s, sccm (pressure x volume / time)
Equating Gas Load & Pump Throughput

Gas Load \hspace{2em} Torr.Liter/sec
Pump Throughput \hspace{2em} Torr.Liter/sec

When Gas Load = Pump Throughput * 
\hspace{2em} (Gas In = Gas Out)

Chamber pressure is stable!
Throughput Calculations — 1

Injecting 800 sccm, chamber pressure 2 mbar.
What is the effective pumping speed needed in L/min?

\[
\begin{align*}
\text{EPS} &= \frac{800}{1000} \text{ sL/min} \\
&= \left(\frac{800}{1000}\right) \times 1013 \text{ mbar.L/min} \\
&= 810 \text{ mbar.L/min} \\
&= \frac{810}{2} \text{ L/min} \quad @ \ 2 \text{ mbar} \\
\text{EPS} &= 405 \text{ L/min}
\end{align*}
\]
Injecting 100 sccm, chamber pressure $3 \times 10^{-3}$ Torr. What is the effective pumping speed needed in L/s?

$$\frac{100}{60} \text{ scc/s}$$

$$= \frac{100}{60} \times \frac{1}{1000} \text{ sL/s}$$

$$= \frac{100}{60} \times \frac{1}{1000} \times 760 \text{ Torr.L/s}$$

$$= 1.27 \text{ Torr.L/s}$$

$$= \frac{1.27}{(3 \times 10^{-3})} \text{ L/s} @ 3 \times 10^{-3} \text{ Torr}$$

$$\text{EPS} = 422 \text{ L/s}$$
Injecting 1 sLm, chamber pressure 0.5 mbar.
What is the effective pumping speed needed in m$^3$/h?

\[
\begin{align*}
&= 1 \times 60 \text{ sL/h} \\
&= (1 \times 60) \times (1/1000) \text{ sm}^3/\text{h} \\
&= (1 \times 60) \times (1/1000) \times 1013 \text{ mbar.m}^3/\text{h} \\
&= 60.8 \text{ mbar.m}^3/\text{h} \\
&= 60.8/(0.5) \text{ m}^3/\text{h} \quad @ \ 0.5 \text{ mbar} \\
\text{EPS} &= 122 \text{ m}^3/\text{h}
\end{align*}
\]
Injecting 300 sccm, have 500 L/s pump. What is base pressure in mbar?

\[
\begin{align*}
&= \frac{300}{60} \text{ scc/s} \\
&= \left(\frac{300}{60}\right) \times \left(\frac{1}{1000}\right) \text{ sL/s} \\
&= \left(\frac{300}{60}\right) \times \left(\frac{1}{1000}\right) \times 1013 \text{ mbar.L/s} \\
&= 5.06 \text{ mbar.L/s} \\
&= \frac{5.06}{250} \text{ mbar}
\end{align*}
\]

(assume EPS is \( \frac{1}{2} \) quoted pumping speed)

\[
\text{Base Pressure} = 0.02 \text{ mbar}
\]
Injecting 200 sccm, 400 L/s turbo backed by 3.8 m³/h vane pump. Turbo’s max foreline pressure 2 mbar.
Will this work?

\[
\begin{align*}
\text{Throughput Calculations} & \quad 5 \\
\text{Injecting} & \quad 200 \text{ sccm, 400 L/s turbo backed by 3.8 m}^3/\text{h vane pump. Turbo’s max foreline pressure 2 mbar.} \\
\text{Will this work?} & \\
= & \quad 200 \times 60 \text{ sccm/h} \\
= & \quad (200 \times 60) \times (1/1000) \text{ sL/h} \\
= & \quad (200 \times 60) \times (1/1000) \times (1/1000) \text{ sm}^3/\text{h} \\
= & \quad (200 \times 60) \times (1/1000) \times (1/1000) \times 1013 \text{ mbar.m}^3/\text{h} \\
= & \quad 12.2 \text{ mbar.m}^3/\text{h} \\
\text{(assume EPS equals quoted pumping speed)} & \\
= & \quad 12.2/(3.8) \text{ mbar} = 3.2 \text{ mbar} \\
\text{It will not work!} & 
\end{align*}
\]
Gas Load & Pump Throughput Conclusions

When Gas Load = Effective Pump Throughput

Chamber pressure is stable

If you don’t like that pressure your options are:

1. Reduce gas load
2. Increase pump throughput
Modeling with VacTran®