Conservation of Helium while Maintaining High System Purity

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Overview

- Motivation for conserving helium
- Overview of pump & backfilling
- Estimating contamination level on an ideal system
- Notes on instrumentation and plotting logged data
- Pumpdown example calculations
  - Pumping on a perfect system
  - Pumping on a system with pumping resistance
  - Pumping on a system with pumping resistance and air leaks
- Applied example at Fermilab
- Summary of best practices

Motivation for Conserving Helium

- Helium is a non-renewable resource
  - Existing known helium reserves will eventually be depleted
- Helium is byproduct of uranium and thorium radioactive decay
- Helium generally derived from natural gas, since helium released during radioactive decay can be trapped in the same natural formations as natural gas
  - Reduced fossil fuel extraction to mitigate climate change could result in a reduced supply of helium
- Only a few locations in the United States have natural gas deposits with >= 0.3% helium
  - Dilute concentrations make separating helium expensive
  - Limited number of locations where extracting helium is economically feasible
- Helium is an international commodity and geopolitical conflict can lead to helium shortages and/or price spikes
- Limited number of helium supply facilities and distributors
  - Force majeure facility closures can cause restrictions on quantities of helium being delivered

Motivation for Conserving Helium

- Finding ways to conserve helium now starts the accrual of financial savings and conserves a finite resource
- Reducing helium losses now also mitigates the impact of future helium price increases and/or shortages

Pump & Backfilling

- One method of reducing helium losses is to carefully evaluate pump & backfill procedures to achieve desired purity levels with the minimum amount of helium loss
- Cleanup of a helium cryogenic system is accomplished by vacuum pumping the gas, initially air or nitrogen, out of the system and backfilling with helium.
  - Dry nitrogen purge is typically used first to remove residual water.
  - Pump & backfilling is not very effective at removing water
  - At 300K, water vapor pressure is 35 mbar = 27 torr = 1 in Hg
  - Having water vapor present will also add to required number of cycles due to the increase in ultimate pumping pressure
- Multiple cycles are required to get down to a contamination level acceptable as an input to a LN_2 cooled charcoal adsorber (< 50 PPM_v).
  - For systems that are leak tight, it is best to do fewer cycles to a deeper vacuum level to conserve helium
  - For systems with leaks, it is best do more cycles to a shallower vacuum level

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Estimating Contamination Level on Ideal System

$$Contamination\ [PPM_v] = \left(\frac{P_{end}}{P_{start}}\right)^{cycles}\ x\ 10^6$$

- P_{start} is the starting pressure of the pump cycle, generally atmospheric pressure
- P_{end} is the ending pressure of the pump cycle
- *Cycles* is the number of pump and backfill cycles

- In general, somewhere between 2 and 5 cycles will be required
- The backfill pressure will be just slightly above atmospheric pressure

Estimating Contamination Level on Ideal System

170 mbar (5 inHg)

Pump and Backfill Cycles		
Cycle	Ideal Purity PPM_v	Helium m3
1	1.7E+05	8
2	2.9E+04	17
3	4.9E+03	25
4	8.4E+02	33
5	1.4E+02	42
6	2.4E+01	50

35 mbar (1 inHg)

Pump and Backfill Cycles		
Cycle	Ideal Purity PPM_v	Helium m3
1	3.4E+04	10
2	1.2E+03	19
3	3.9E+01	29
4	1.3E+00	39
5	4.5E-02	48
6	1.5E-03	58

5 mbar (0.15 inHg)

Pump and Backfill Cycles		
Cycle	Ideal Purity PPM_v	Helium m3
1	5.0E+03	10
2	2.5E+01	20
3	1.3E-01	30
4	6.3E-04	40
5	3.1E-06	50
6	1.6E-08	60

- Pumping to 5 mbar instead of 170 mbar
  - Reduces number of cycles required from 6 to 2 for an equivalent final purity (~25 PPM_v)
  - Results in a 60% reduction in helium usage for system cleanup
  - Values less than 50 PPM_v in yellow boxes, values less than 10 PPM_v in green boxes

Optimizing Pump & Backfill Parameters

- Different people at different times and locations have selected various pressure and time criteria to stop pumping and various numbers of pump and backfill cycles
  - There are many ways to achieve the desired helium purity level, but some pump & backfill methods are more efficient in the use of helium and time than others
- Reviewing literature and cryogenic engineering handbooks yielded little useful information on optimizing pump & backfill parameters using calculations and experimental data
- This presentation documents a methodology and tool that can be used to help optimize pump & backfill parameters to achieve the desired helium purity with a minimum loss of helium a and minimum time required to complete the procedure

Instrumentation

- A critical component necessary for optimizing pump & backfill procedures is the pressure/vacuum transmitter
  - Transmitter must be capable of reading accurately at the desired vacuum level and should be on a periodic calibration cycle
  - Transmitter readings should be read through a control system where the reading can be plotted in real-time
- Transmitter Location
  - Ideally the transmitter is placed at the opposite end of the pumped volume from the vacuum pump for continuous accurate readings
  - If it is unavoidable to place the transmitter on the pumping line, then there needs to be an isolation valve between the pump and the transmitter.
    - Drawback is that the isolation valve will need to be periodically closed and enough time must be given for the vacuum pressure to equalize across the pumped volume.
      - Process will take longer,
      - More time at vacuum pressures provides more time for air to leak in
      - More difficult to determine when pumpdown becomes non-linear on a semi-log plot

Plotting of Signal

- Plotting of pressure in real-time on a semi-log scale is important
  - The pressure versus time plot on a semi-log scale is ideally a straight line
- Once the vacuum pressure begins deviating from a straight line on a semi-log plot the pumping should be stopped
- Potential reasons for deviations from a straight line
  - There is a leak into the system from atmosphere
  - There is residual water vapor on metal surfaces that is being de-adsorbed
  - There is water in the vacuum pump oil degrading its performance
  - The ultimate pressure of the vacuum pump has been reached

Case 1: Perfect System

- Assumptions
  - Ideal gas
  - No pumping resistance
  - No external leaks into the system
  - No residual entrained water vapor

- Mass balance equations

Mass balance

$$\frac{dM}{dt} = \dot{m}_{In} - \dot{m}_{Out} \tag{1}$$

where M = mass of gas in the system
$\dot{m}_{In}$ = mass flow rate leaking into the system
$\dot{m}_{Out}$ = mass flow rate pumping out of the system

$$M = \frac{PV}{RT} \quad \text{from the ideal gas law} \tag{2}$$

where P = system pressure
V = system volume
R = gas constant
T = gas temperature

For a system with no leaks $\dot{m}_{In} = 0$

Pumping capacity

$$\dot{m}_{Out} = \rho Q = \frac{PQ}{RT} \tag{3}$$

where ρ = system gas density
Q = vacuum pump volume flow capacity

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Case 1: Perfect System

- Assumptions
  - Ideal gas
  - No pumping resistance
  - No external leaks into the system
  - No residual entrained water vapor
- Equations to calculate the pressure inside the volume over time during pumping

Combining (1), (2) and (3)

$$\frac{V}{RT}\frac{dP}{dt} = -\frac{PQ}{RT} \quad (4)$$

Rearrange as an integral

$$\int_{P_1}^{P_2}\frac{dP}{P} = -\frac{Q}{V}\int_0^t dt \quad (5)$$

Integrate

$$ln\left(\frac{P_2}{P_1}\right) = -\frac{Q}{V}t \quad (6)$$

$$\frac{P_2}{P_1} = e^{-\frac{Q}{V}t} = e^{-\frac{t}{\tau}} \quad (7)$$

Where $\frac{V}{Q}$ is the time constant, τ, of the system. $\frac{Q}{V}t$ needs to be dimensionless.

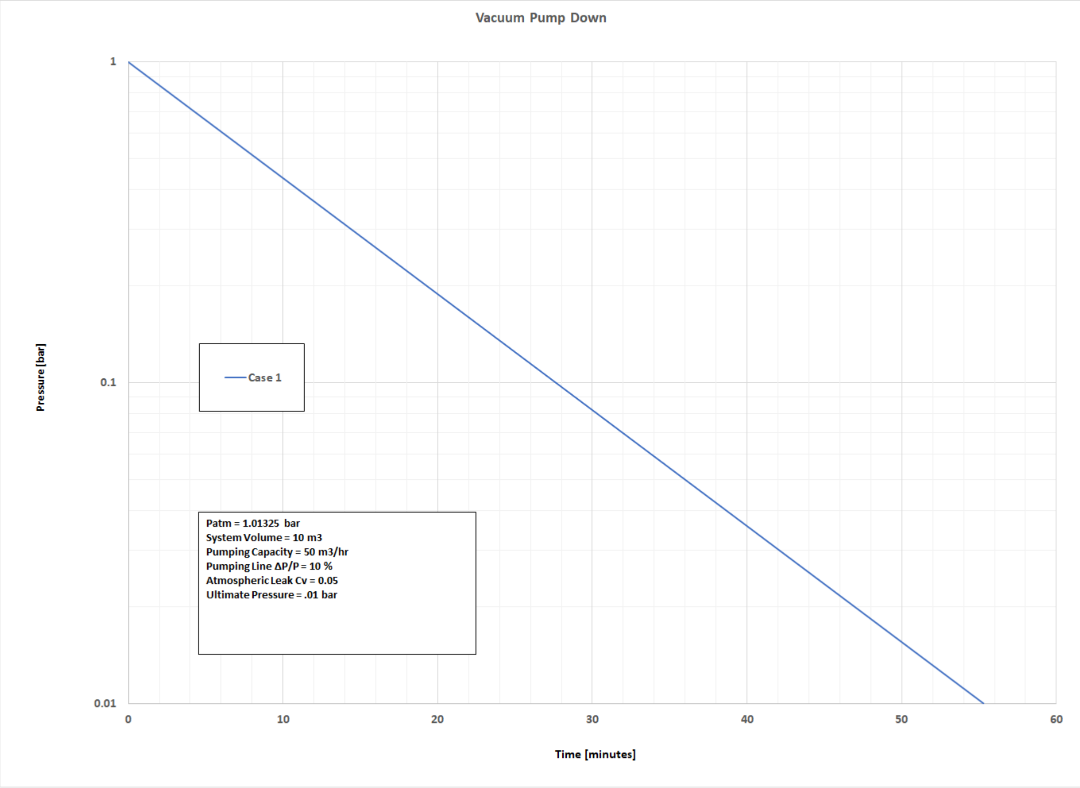
P_1 is the pressure at the start of pump down

P_2 is the pressure at the end of the pump down

Case 1: Perfect System

- Assumptions
  - Ideal gas
  - No pumping resistance
  - No external leaks into the system
  - No residual entrained water vapor

- Pump down process is linear on a semi-log plot



Pumpdown of Initial Volume of Air

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Case 2: System with Pumping Resistance

- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - No external leaks into the system
  - No residual entrained water vapor

- Additional mass balance equations
- To simplify this presentation the ΔP will be assumed to be 10% of the volume pressure
- The X term can be easily manipulated to match experimental data

Pumping capacity is now based on the vacuum pump inlet pressure which is now lower than the pumped volume.

$$\dot{m}_{Out} = \rho_P Q = \frac{P_P Q}{RT} \qquad (8)$$

where
ρ_P = gas density at the vacuum pump inlet
P_P = pressure at the vacuum pump inlet = $P - \Delta P$
Q = vacuum pump volume flow capacity

Combining (1), (2) and (8)

$$\frac{V}{RT}\frac{dP}{dt} = -\frac{P_P Q}{RT} = -\frac{(P - \Delta P)Q}{RT} \qquad (9)$$

The pressure drop between the volume and the pump will be based on the volume pressure in order to simplify the integration.

$$\Delta P = \frac{fL}{D}\frac{\rho V^2}{2} = \frac{fL}{D}\frac{Q^2}{2A^2}\frac{P}{RT} = XP \qquad (10)$$

where
f = Darcy-Weisbach friction factor
L = effective length of pumping line, use four time the actual length for corrugated hose
D = Inside diameter of the pumping line
V = flow velocity in the pumping line = $\frac{Q}{A}$
A = inside cross-sectional area of the pumping line
R = gas constant
T = gas temperature
$X = \frac{fL}{D}\frac{Q^2}{2A^2}\frac{1}{RT} = \frac{\Delta P}{P}$ for the pumping line, used to simplify subsequent formulas

Case 2: System with Pumping Resistance

- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - No external leaks into the system
  - No residual entrained water vapor

- Assumptions
  - Including pumping resistance reduces the effective pumping capacity
  - The system time constant is now *τ* = *V*/(1-*X*)*Q*, so a larger Δ*P* results in a larger time constant
  - Note that *X* changes with each cycle since it will be based on air for the first cycle and predominately helium on subsequent cycles.

Rearrange as an integral

$$\int_{P_1}^{P_2} \frac{dP}{P(1-X)} = -\frac{Q}{V}\int_0^t dt \quad (11)$$

Integrate assuming friction factor is constant

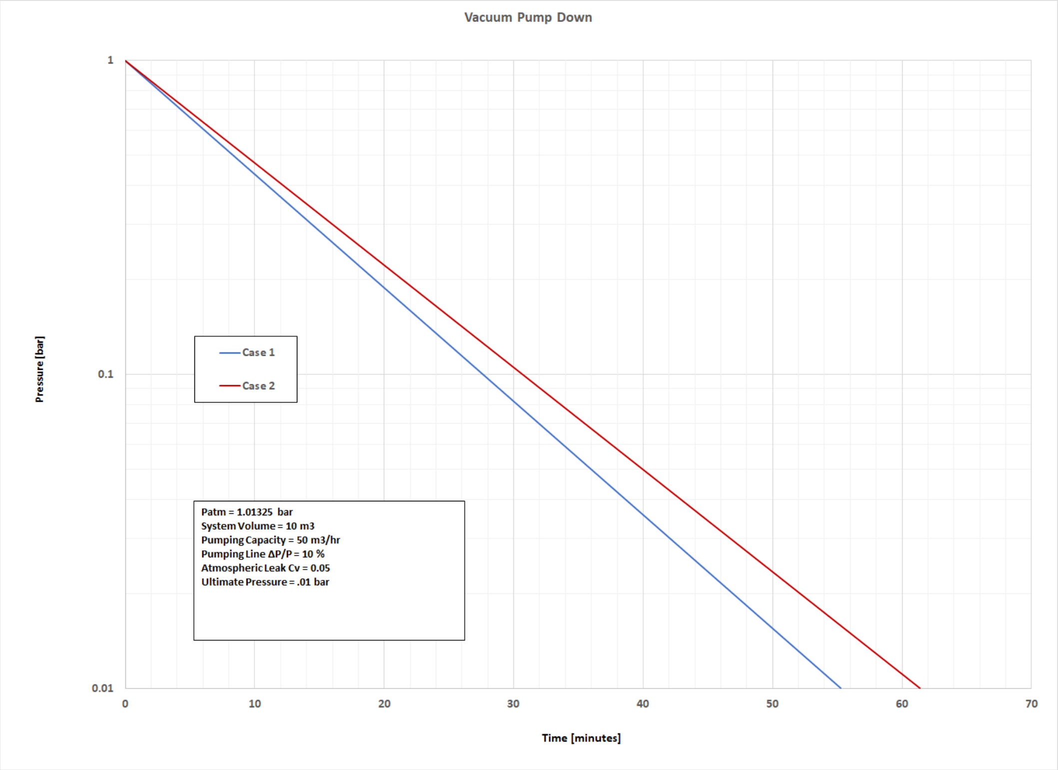
$$\frac{ln\left(\frac{P_2}{P_1}\right)}{1-X} = -\frac{Q}{V}t \quad (12)$$

$$t = -\frac{V}{(1-X)Q}\, ln\left(\frac{P_2}{P_1}\right) \quad (13)$$

$$P_2 = P_1\, e^{-\frac{(1-X)Q}{V}t} \quad (14)$$

Case 2: System with Pumping Resistance

- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - No external leaks into the system
  - No residual entrained water vapor
- The slope of the line changes with pumping resistance included, but trend remains linear on a semi-log plot



Pumpdown of Initial Volume of Air

Case 3: System with Pumping Resistance & Air Leak

- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - External leak into the system from dry atmospheric air
  - No residual entrained water vapor
- Assume choked flow through leak throughout pumpdown
- The initial pump down period where flow through the leak isn't choked is relatively unimportant ($\dot{m}_{Out}$ >> $\dot{m}_{in}$)
- Knowing the size of the leak and where the pumpdown starts significantly deviating from being linear on a semi-log plot is important
- Air leak rates can be unknown or change over time, so the effective valve coefficient C_v may have to be empirically determined by adjusting the model to determine the effective C_v that matches experimental data

For case 3, $\dot{m}_{In}$ is no longer zero. The flow of atmospheric air into the volume will be treated as flow through a control valve, equating the leak as a control valve C_v.

The leak will spend more time as a choked flow during the pumping process. Based on ISA 75.01.01, choked compressible flow through a valve can be characterized as:

$$\dot{m}_{In} = N_6\, C_v\, Y \sqrt{F_\gamma\, X_T\, \rho_A\, P_A} \quad (15)$$

where
- N_6 = numerical constant from ISA 75.01.01
- $N_6' = \frac{2.73}{3600\sqrt{1000}}$ for pure SI units, $\dot{m}_{In}[=]\frac{kg}{s}$, $\rho[=]\frac{kg}{m^3}$, $P[=]Pa$
- C_v = equivalent valve C_v representing the leak. This is dominated by flow area, not perimeter, so equate it to an area based on C_v = 30 has an area of 1 in^2
- Y = gas expansion factor, see ISA 75.01.01. It is a function of downstream pressure until the flow becomes choked and varies from 2/3 to 1. The pump down starts with Y=1 and decreases until the leak is choked flow where Y=2/3. The pump down will spend most of the time in the choked flow regime. It seems reasonable to use a constant value of Y=2/3. If the pressure dependance is considered, it significantly complicates the integration of the resulting differential equation.
- ρ_A = density of atmospheric air
- P_A = pressure of atmospheric air
- F_γ = specific heat ratio, C_p/1.4, which is 1 for air or any diatomic gas
- X_T = ratio of ΔP / P_{In} for choked flow condition. Assuming a value of X_T = 0.5 seems reasonable for the inefficient leak area geometry and is consistent with ideal gas flow through an orifice without pressure recovery.
- $G = N_6'\, C_v\, Y \sqrt{F_\gamma\, X_T\, \rho_A\, P_A}$ used to simplify subsequent formulas. Note that this is a constant, which is not technically correct until the volume pressure is ~½ atmospheric pressure (choked flow).

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Case 3: System with Pumping Resistance & Air Leak

- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - External leak into the system from dry atmospheric air
  - No residual entrained water vapor

- The math becomes more complicated including air leaks, but analytical solutions can still be determined to estimate pressure over time during pumpdown

Combining (1), (2), (8), (10) and (15)

$$\frac{V}{RT}\frac{dP}{dt} = G - \frac{P\,(1-X)Q}{RT} \qquad (16)$$

Rearrange

$$\frac{dP}{dt} = \frac{GRT}{V} - \frac{P\,(1-X)Q}{V} \qquad (17)$$

$$\int_{P_1}^{P_2} \frac{dP}{\frac{GRT}{V} - \frac{P\,(1-X)Q}{V}} = \int_{t_1}^{t_2} dt \qquad (18)$$

Substitute $E = \frac{GRT}{V}$ and $F = \frac{(1-X)Q}{V}$

$$\int_{P_1}^{P_2} \frac{dP}{E - PF} = \int_{t_1}^{t_2} dt \qquad (19)$$

Integrate

$$\ln\left(\frac{FP_2 - E}{FP_1 - E}\right) = -Ft \qquad (20)$$

$$t = -\frac{1}{F}\ln\left(\frac{FP_2 - E}{FP_1 - E}\right) \qquad (21)$$

$$\frac{FP_2 - E}{FP_1 - E} = e^{-\frac{(1-X)Q}{V}t} \qquad (22)$$

$$P_2 = \frac{E + (FP_1 - E)\,e^{-\frac{(1-X)Q}{V}t}}{F} \qquad (23)$$

Case 3: System with Pumping Resistance & Air Leak

- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - External leak into the system from dry atmospheric air
  - No residual entrained water vapor
- It is important to stop pumping before getting to the ultimate pressure after the first pump & backfill cycle has been performed
- As the trend deviates from a linear line on a semi-log plot air the flow rate of air leaking in is becoming significant relative to the flow rate of the air-He mixture being pumped out
- Leaving the vacuum pump on while slowly approaching the ultimate pressure defeats the effect of previous pump and back fill cycles

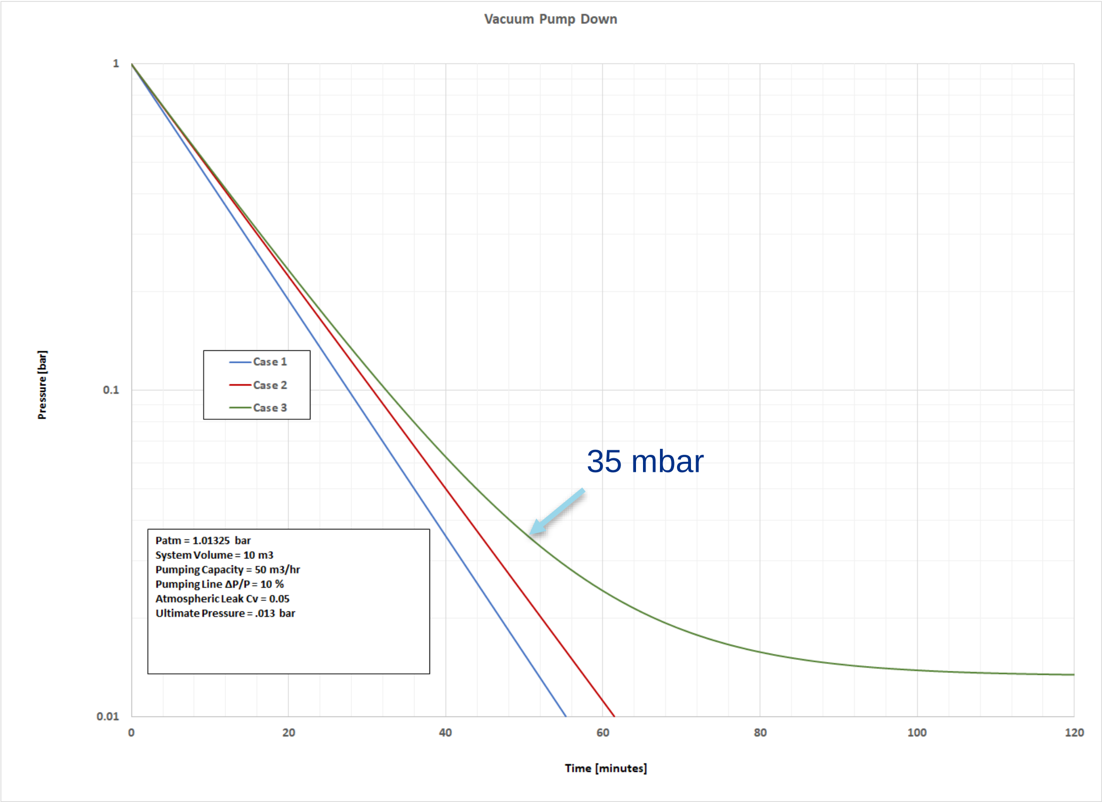
Any leak into the system means that there is a pressure at which the pumping flow is equal to the incoming leak. It is not possible to pump below that pressure without changing the system. This ultimate pressure can be determined by setting $\dot{m}_{In} = \dot{m}_{Out}$ and solving for P.

$$\dot{m}_{In} = N_6' \, C_v \, Y \sqrt{F_\gamma \, X_T \, \rho_A \, P_A} = \dot{m}_{Out} = \rho(1 - X)Q = \frac{P(1-X)Q}{RT} \quad (24)$$

$$P_{Ultimate} = \frac{RT}{(1-X)Q} N_6' \, C_v \, Y \sqrt{F_\gamma \, X_T \, \rho_A \, P_A} \quad (25)$$

Case 3: System with Pumping Resistance & Air Leak

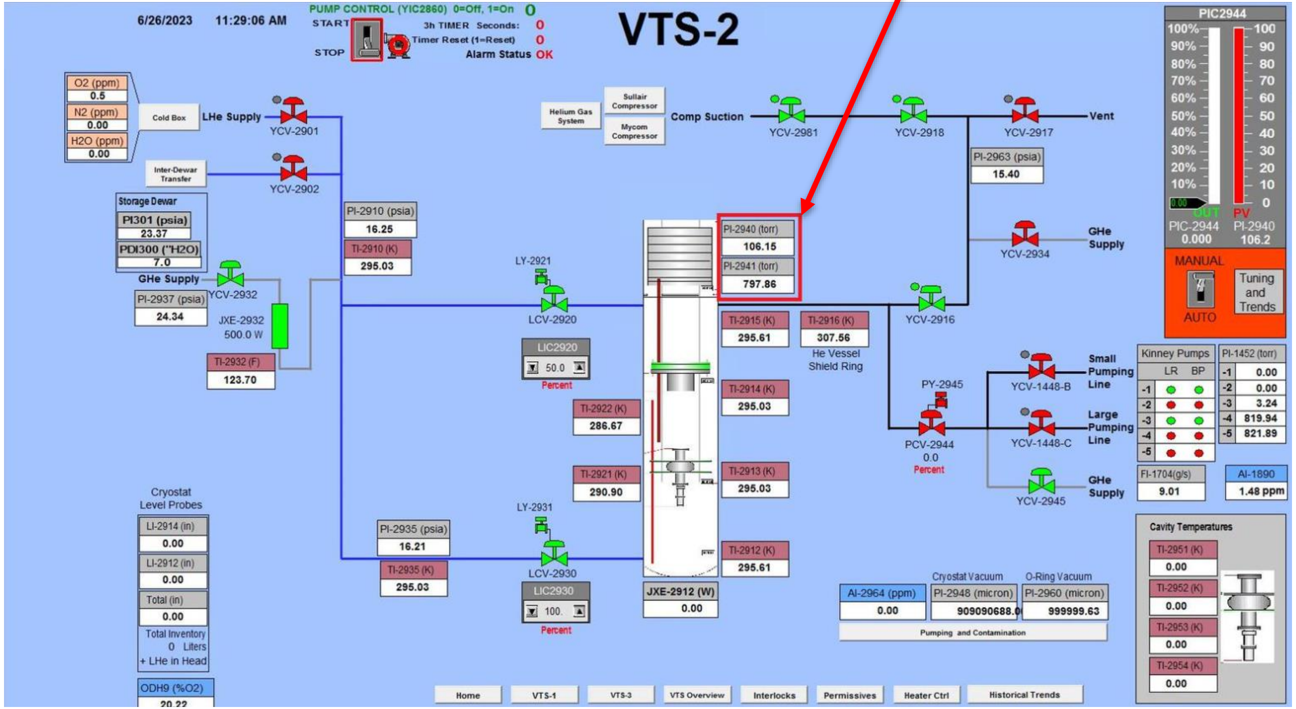
- Assumptions
  - Ideal gas
  - Flow resistance between vacuum pump and pumped volume
  - External leak into the system from dry atmospheric air
  - No residual entrained water vapor
- Based on the assumed inputs the ultimate pumping pressure for this case is 13 mbar
- The 2nd hour of pumping trying to reach 13 mbar is basically just slowly defeating the effect of previous pump & backfill cycles. Air leaking in is displacing the helium-air mixture going out through the vacuum pump, so contamination is actually increasing during this 2nd hour
- The best solution would be to stop pumping at about 35 mbar, then use 3 cycles to get under 50 PPM_v or 4 cycles to get under 10 PPM_v for a final contamination level



Pumpdown of Initial Volume of Air

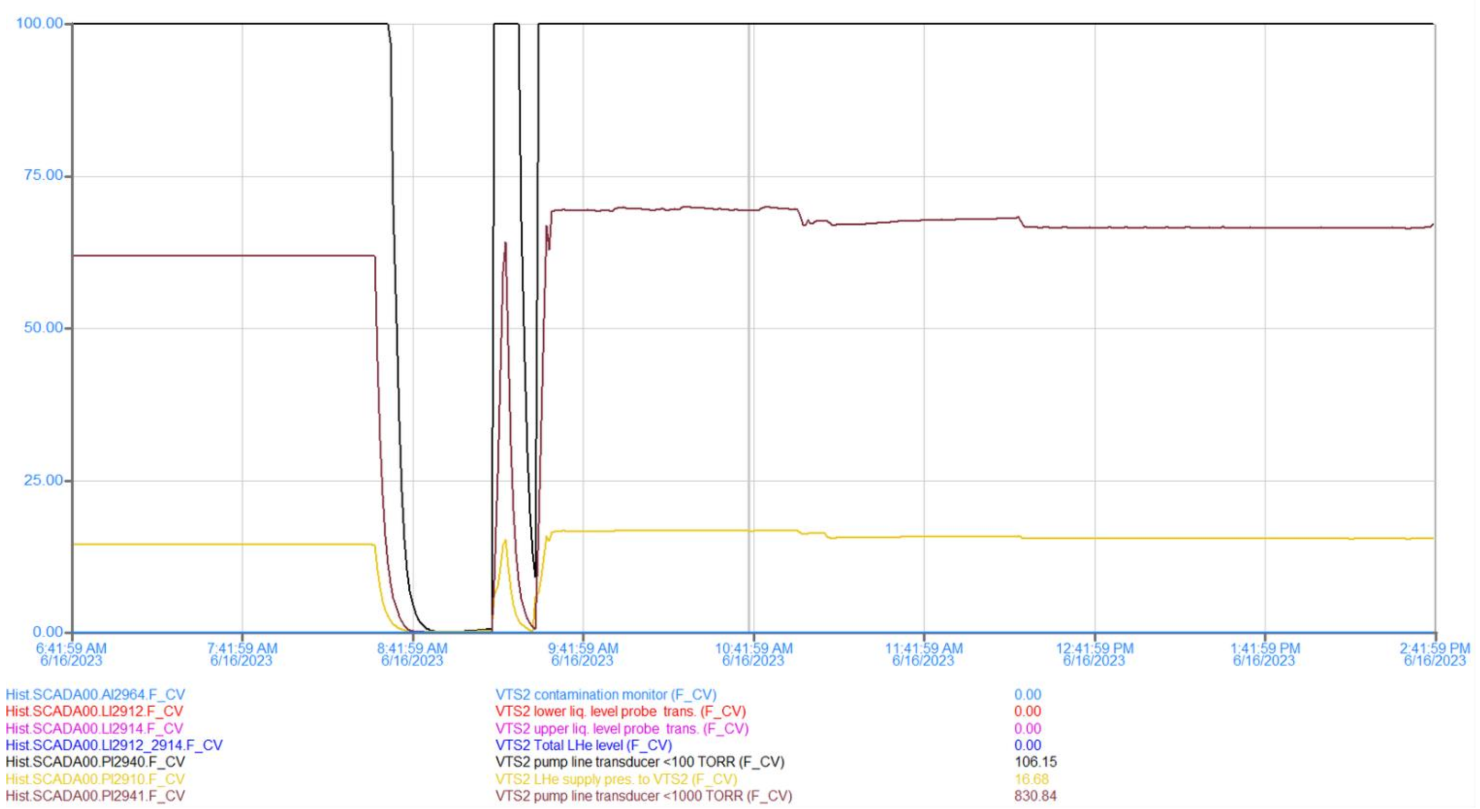
Example at Fermilab

Pressure Transmitters



Vertical Test Stand – 2 for testing SRF cavities

Example at Fermilab



Using only 2 pump & backfill cycles there is no detectable residual contamination!

Summary of Best Practices

- Start by purging with dry nitrogen to remove water and get the best achievable ultimate vacuum for the system
- Use helium leak detector after first backfill and where possible locate and repair leaks. This minimizes air in-leak during future pumping cycles
- Use pressure transmitter and real-time semi-log plot to determine when to stop pumping
- Don’t pump on the volume overnight during pump & backfill!
- Based on the pressure when pumping is stopped, calculate the number of cycles necessary to achieve desired purity level
- Based on experimental data from Fermilab test stands, as few as 2 pump & backfill cycles can reduce the contamination below levels detectable by commercial oxygen analyzers

Acknowledgments

- Special thanks to Jay Theilacker, now recently retired, for developing this calculation template
- This is yet another example of Jay’s many valuable contributions to the cryogenics community over the past 4+ decades

- Contact mjwhite@fnal.gov for calculation template and questions

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