Helium Purification

By

JLab Cryo Group

Presenter: Mathew Wright

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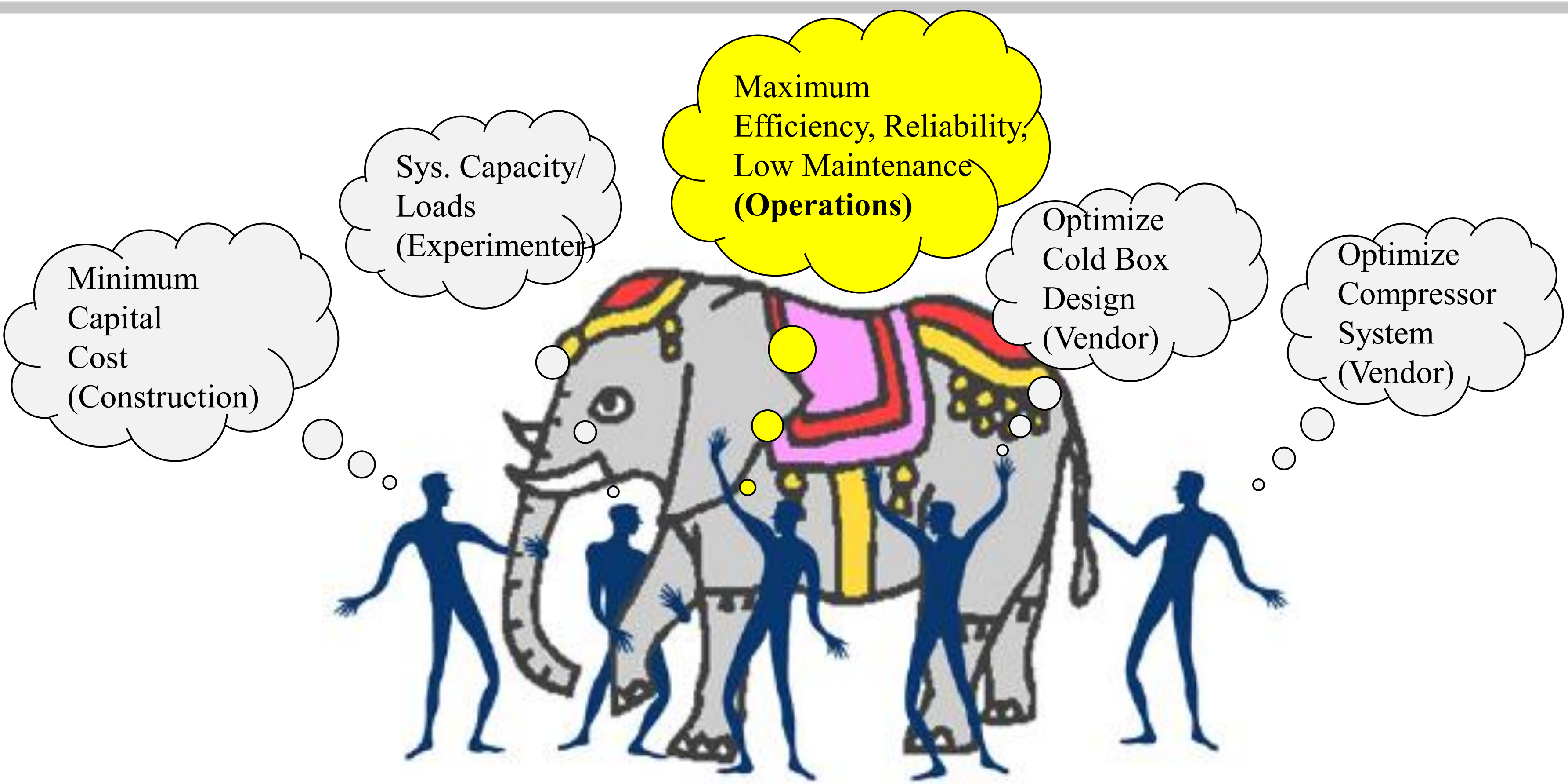
Topics

- Presenter Background
- Background of Helium Purification
- Design Development
    - Carbon Bed
    - Molecular sieve (molsieve) Bed
    - 3 Pass Heat Exchanger
    - LN2 Boiler
- Design Considerations & Code Calculations
- Status

Who is Mathew Wright?



- One’s viewpoint can be based only on their role and focus within a project.
- Easy to believe that one’s goals are mutually exclusive of others.
- Many believe that maximum system efficiency occurs only at one set of fixed operating conditions.

Presenter Background

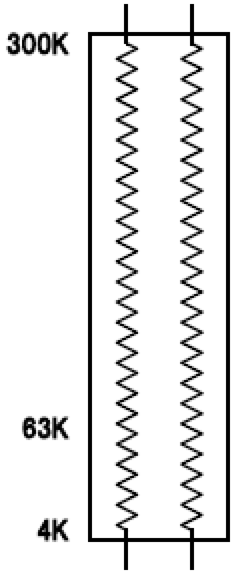
- Finished Associates degree in 1994
- Worked as model maker for a contract company
- Started at Jefferson Lab in 2000 as a mechanical designer
- Started purifier design in 2005
- Graduated with BS ME degree from ODU in 2006
- Promoted from designer to Chief Cryo Operator
  - Stopped all work on new CHL purifier except requirements to graduate w/ masters
- Graduated with MS ME from ODU in 2009
- Commission new CHL purifier in 2011!

What is a helium purifier?

- www.dictionary.com (purify)
  - to make pure; free from anything that debases, pollutes, adulterates, or contaminates
- Jefferson Lab Helium Purifiers
  - Helium purification for ppm level only
  - Removal traces of air (contamination)
    - Nitrogen, oxygen, moisture
  - Molsieve beds at ~300K used to remove H_20
  - Carbon beds at ~80K used to remove N_2 & O_2
- Oil is also a contaminant, is mitigated with other methods
  - Oil Coalescers
  - Charcoal
  - Process Pressures > 13 atmospheres

Why purify helium?

- Build-up of frozen impurities will affect plant capacity and operation
  - Damage moving parts
  - Alter flow characteristics
  - 1 ppm of N_2 in a helium flow @ 15 g/s ~ 4 liters/year LN_2



All contamination will end up in the cold box. If there is a build-up of frozen moisture (273K) or nitrogen (63K) in the process path, the process has to be shutdown, warmed up, and purged to the purification system or even vented.

Where Does Contamination Come From?

- Isolated equipment bled down to atmospheric pressure
  - Barometric pressure changes throughout the day
  - Temperature of the day will affect the pressure in the piping
- Equipment not properly purged of contamination during
  - Installation / Commissioning
  - Maintenance
    - Oil in the pipes exposed to air has an affinity to attract contamination
    - Low Pressures
    - Low Temperatures

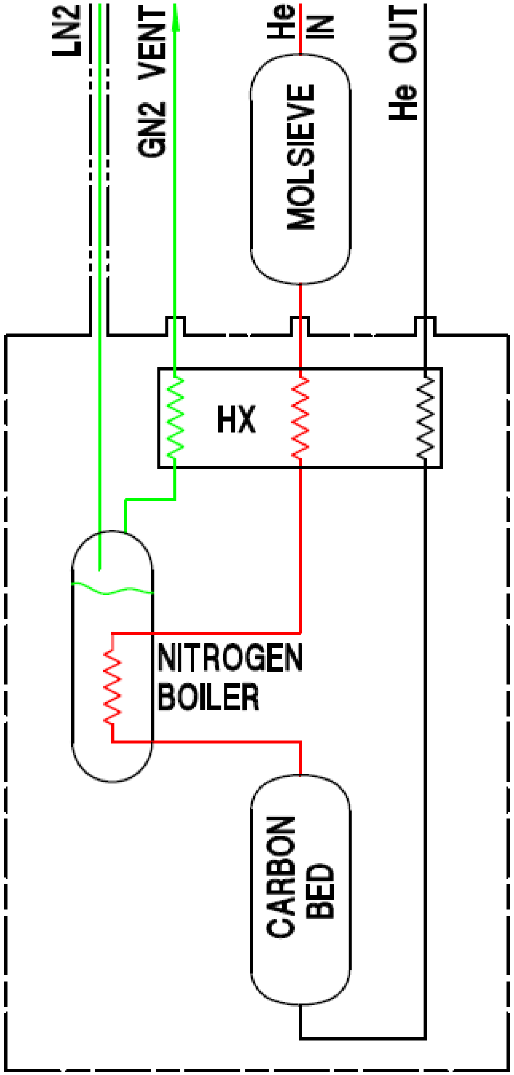
Where Does Contamination Come From?

- Sub-atmospheric operations (0.04 atmospheres – 2.1K)
- Other process fluids used in heat exchangers at higher pressures (Water, LN_2, Hydrogen)
- Make-Up of lost helium (300 liquid liters per day)
  - Note that 5 ppm of N_2 in a helium flow at 300 liquid liters per day ~0.6 liters/year of LN_2
  - Maximum Trace Contamination in Helium Deliveries at Jefferson Lab:

Contaminant	Max Amount	Delivered on 2-15-11
Nitrogen	5 ppm	.635 ppm
Neon	9 ppm	.05 ppm
Hydrocarbon	0.5 ppm	0.1 ppm
CO and CO2	0.5 ppm	0.04 ppm
Argon	1 ppm	.326 ppm
Water	1 ppm	.122 ppm

How is Helium Purified?

- Dirty helium enters the purifier.
- The molsieve bed removes moisture.
- The heat exchanger cools the helium to ~85K.
- The Nitrogen boiler cools the helium to ~80K.
- The carbon bed removes the N_2 and O_2.
- The heat exchanger recovers the refrigeration of the helium exiting the purifier.
- Note that the only LN_2 used during steady state is due to inefficiencies of the heat exchanger and static heat leak.

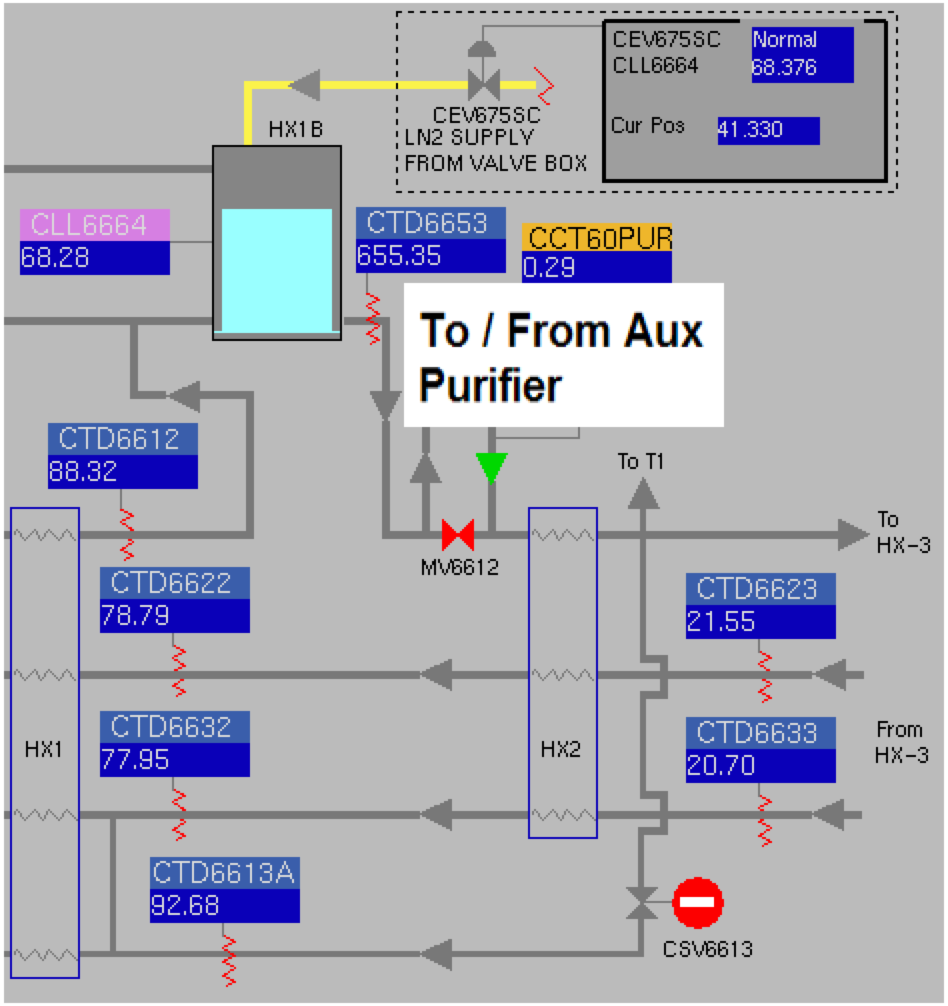


Process Options for Purification

- Full-flow purification:
  - ESR
    - Supported loads, e.g., magnets, tend to be more contaminated than RF cavities.
  - CTF
    - Supported loads use sub-atmospheric room temperature vacuum pumps, which are prone to leaking.
- Slip-flow purification:
  - CHL
    - Guard vacuum and cold compressors protect from most contamination sources.

How is helium purified?

- Full-flow purification devices utilize the total process flow through carbon beds.
- 80K beds to remove O_2 & N_2
- 20K beds to remove neon and hydrogen



The Picture to the right is an example of the full-flow 80K beds that have been added to the ESR cold box after the LN_2 Boiler (~80K).

How is helium purified?

The molsieve and activated carbon granules have many orders of magnitude of surface area in their pores compared to their outer surface area. The surface area has the affinity to adsorb and retain contaminants at certain process conditions but can only adsorb finite amounts of contaminants relative to their weight. Once the saturation capacities are reached, the molsieve and carbon granules need to be regenerated.

- **Molsieve** removes **water vapor** from the helium flow at room temperature.
- **Carbon** removes **nitrogen (air)** at 80K.
  - Made of coconut shell because nature has provided high amount of surface area, better than anything we can cost-effectively synthesize.

Drivers for new Purifier design

- Existing challenges
  - Needs refurbishment after many years of continuous use
  - Valves are hard to identify
  - Valves are hard to reach
  - Too long to regenerate
    - (1 week+)
  - Helium leaks
  - Instrumentation needs repaired
- JLab got a design funded by Linde
- Project facilitated MS degree

JLab New Purifier

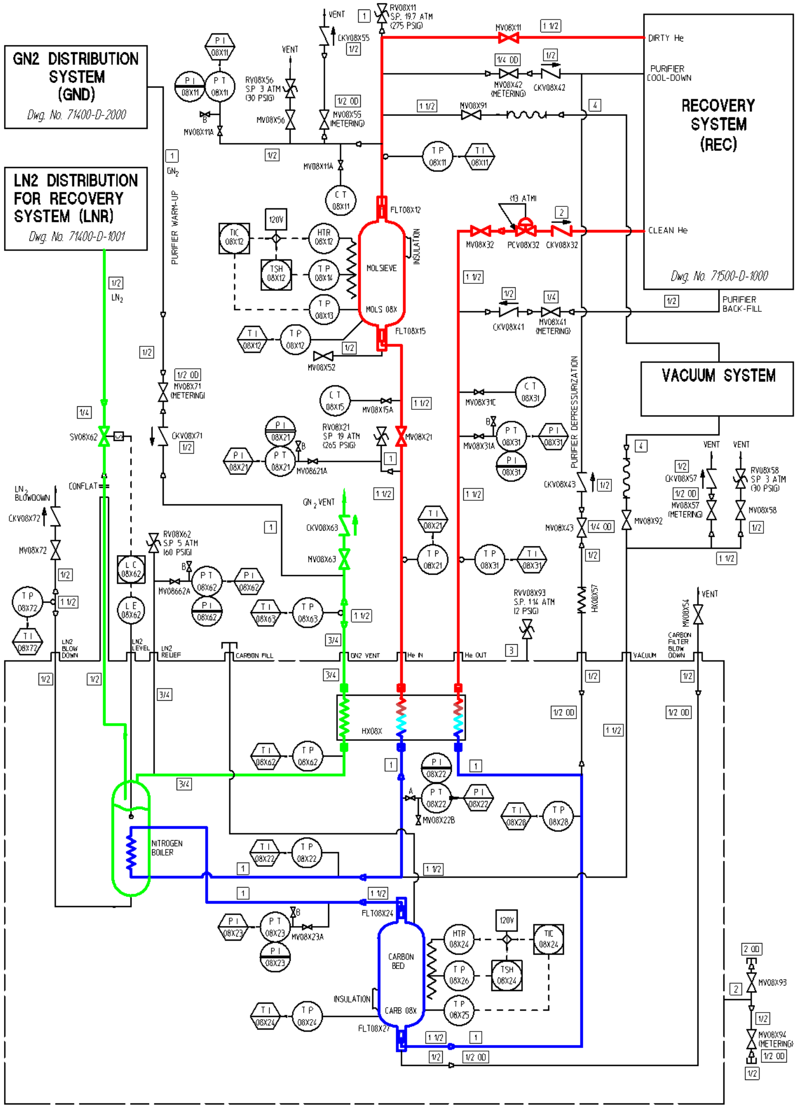
Process Requirements

- 40 g/s nominal to 60 g/s maximum of helium flow
- 10 ppm of water vapor
- 10 ppm of nitrogen
- 4 weeks minimum bed life

Design Basis

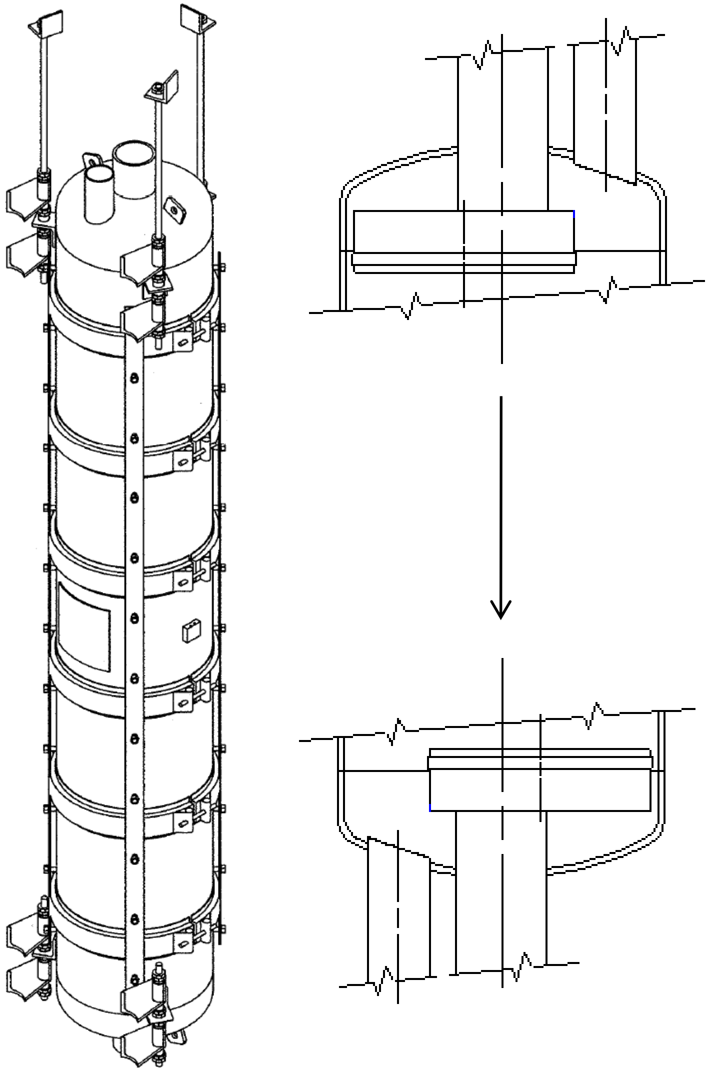
- Ease of Operation
- **Molsieve bed** is maintained at **300K**
- **Carbon bed** is maintained at **80K**
- Regeneration Heating
  - **350ºF** for the **Molsieve**
  - **150ºF** for the **Carbon**
- Shipping
- Ease of Fabrication and Assembly

Purifier Flow diagram



Carbon Bed Design Development

- The size of the carbon bed was determined using the following parameters:
  - Specific adsorbent capacity of carbon
  - Mass concentration of nitrogen in the helium flow
  - Specific weight of adsorbent material
- Bed diameters are derived from:
  - Required volume
  - Practical length-to-diameter ratios of 3 (minimum) to 8 (maximum)
  - Checking velocity and pressure drop constraints



Carbon Bed Design Development

- Specific adsorbent capacity of carbon

$$(\varepsilon_{ij})_{eq} = RT\ln(P_s/p)$$ (Polanyi potential theory)

	Input Value	Output Value	Units
R = Gas Constant for Nitrogen	0.2968		kJ/kg-K
T = Temperture	80		K
P_S = Staturation Pressure	137.0		kPa
p = Partial Pressure of Nitrogen	0.01317		kPa
v = (v ij)eq =		219.6	kJ/kg
Excess Adsorption Energy		1469	cal/mole

$$p = \frac{ppm}{1,000,000}\,p_T$$

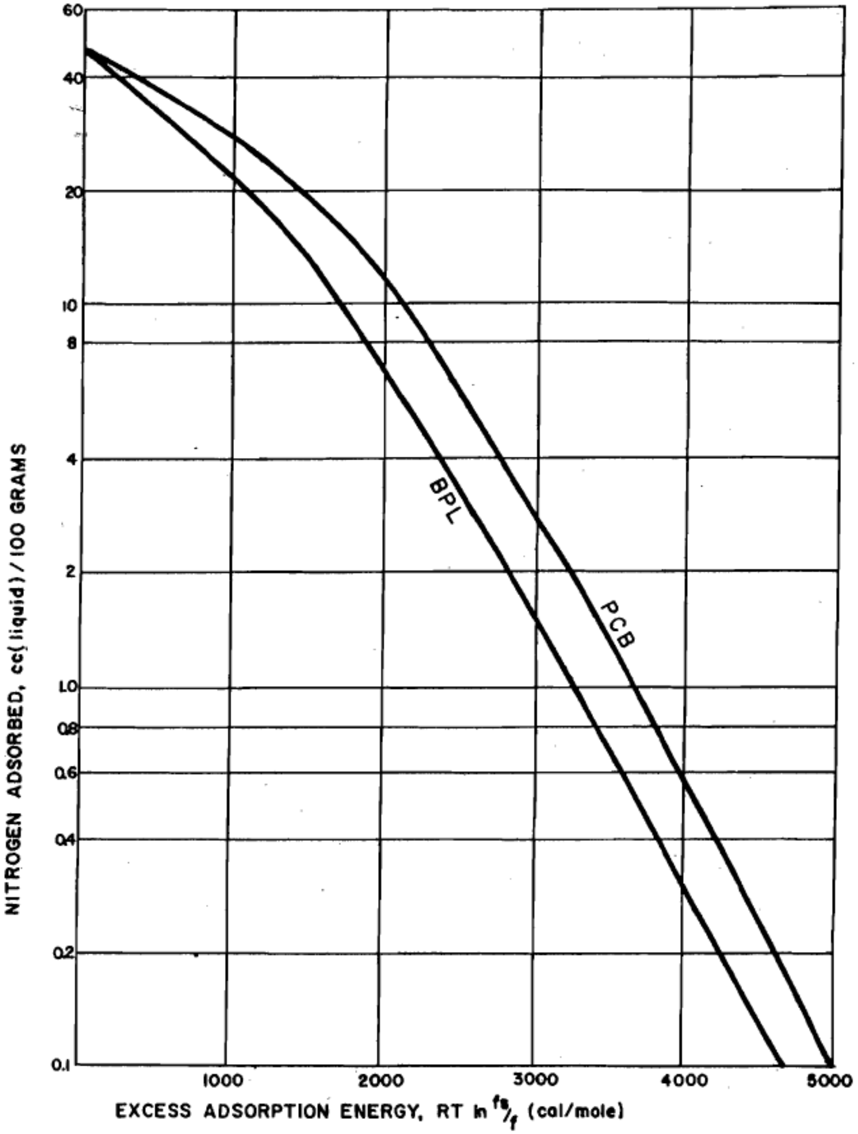
	Input Value	Output value	
Ratio of moles of Nitrogen to He	10.00		ppm
P_T = Total Pressure	13		atm
	1317		kPa
p = Partial Pressure of Nitrogen		0.01317	kPa

Carbon Bed Design Development

- Specific adsorbent capacity of carbon

$$
\begin{aligned}
N_2 \text{ adsorbed} = & -2.1720 \times 10^{-18} * v^5 \\
& + 2.9064 \times 10^{-14} * v^4 \\
& - 1.2939 \times 10^{-10} * v^3 \\
& + 1.3893 \times 10^{-7} * v^2 \\
& - 2.7232 \times 10^{-4} * v \\
& + 1.6802
\end{aligned}
$$

Manes, M. and Grant R.J., "Calculation methods for the Design of Regenerative Cryosorption Pumping Systems," Transactions of the 10th National Vacuum Symposium, 1963, Macmillan, NY, 1963



Carbon Bed Design Development

- Mass concentration of nitrogen in the helium flow

	Input Value	Output Value	Units
Mole Fraction of He	0.99999		moles/moles
Molecular Weight of He	4.0026		kg He / kmol He
Mass (kg) per kmol of Mix He		4.00256	kg/kmol
Mole Fraction of N2	0.00001		moles/moles
Molecular Weight of N2	28.0134		kg N2 / kmol N2
Mass (kg) per kmol of Mix N2		0.00028	kg/kmol
Total mass per kmol of Mix		4.00284	kg/kmol
% of Mass of Nitrogen		0.00007	kg N2 / kg mix
Mass flow rate of He	40		g/s
Mass Flow Rate of N2		0.002799	g/s

Carbon Bed Design Development

- Specific weight of adsorbent material

	Input Value	Output Value	Units
Mass Flow Rate of N_2	0.002799		g/s
Break Through Time	30.00		days
Nitrogen Required to be Adsorbed		7.26E+03	g N_2
Capacity of Carbon to Adsorb Nitrogen	19.52		cm^3_{liq}/100 g C
Density of Nitrogen at T	794		kg/m3
Mass of Carbon		46.81	kg
Density of Carbon	0.44		g/cc
Volume of Carbon		106389	cm^3
		3.76	ft^3

$$Mass\_of\_Carbon = \frac{7.26kg}{\left(794\frac{kg}{m^3}\right)\left(\frac{1m^3}{1000000\ cm^3}\right)\left(195.2\frac{cm^3}{kgC}\right)}$$

Carbon Bed Design Development

- Practical length-to-diameter ratios

$$D = \sqrt[3]{\frac{4V}{\pi R}} \qquad L = \sqrt[3]{\frac{4VR^2}{\pi}}$$

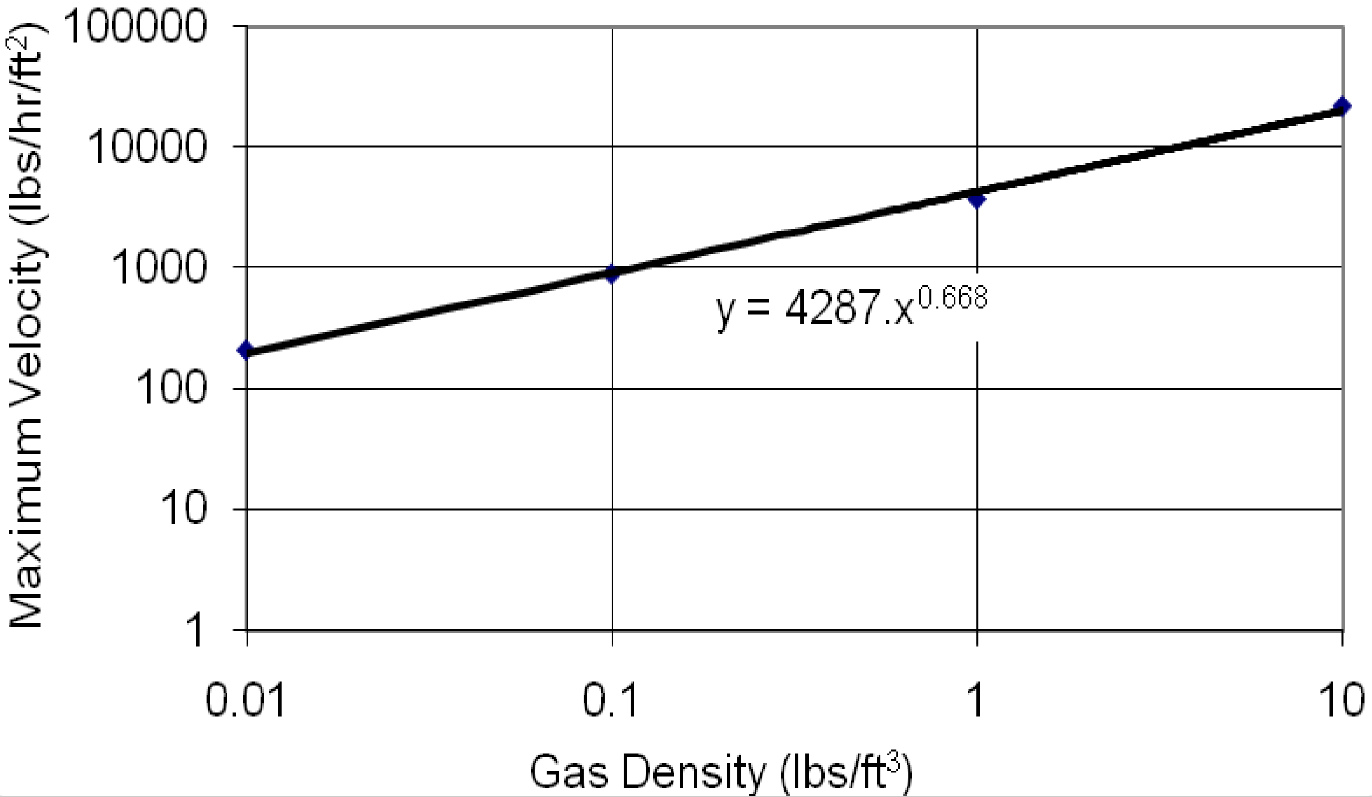
R = L/D	Bed Diameter (inch)	Bed Height (inch)
3	14.02	42.06
4	12.74	50.95
5	11.82	59.12
6	11.13	66.76

New JLab CHL Purifier

Carbon Bed Design Development

- Maximum allowable gas flux velocities for 4/6 granular beds to prevent pulverization and dusting

  for flow from top to bottom $V_{Max} = 4287\,\rho^{.668} = 2638\,\dfrac{\text{lbs}}{\text{hr - ft}^2}$



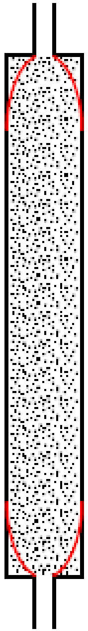
Carbon Bed Design Development

- Calculated Gas Velocities

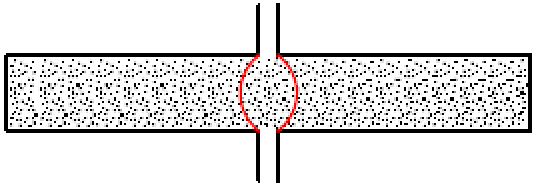
$$v_{bed} = 1455 \frac{\dot{m}}{D^2}$$

R = L/D	Bed Diameter (inch)	Gas Velocity (lbs/hr-ft^2)
3	14.02	296.2
4	12.74	358.8
5	11.82	416.3
8	10.11	569.5
5.8	12.39	379.2

A carbon bed w/ large L/D would have a higher velocity of helium flow.



The carbon in a carbon bed with a very small L/D would not be as effective.



Carbon Bed Design Development

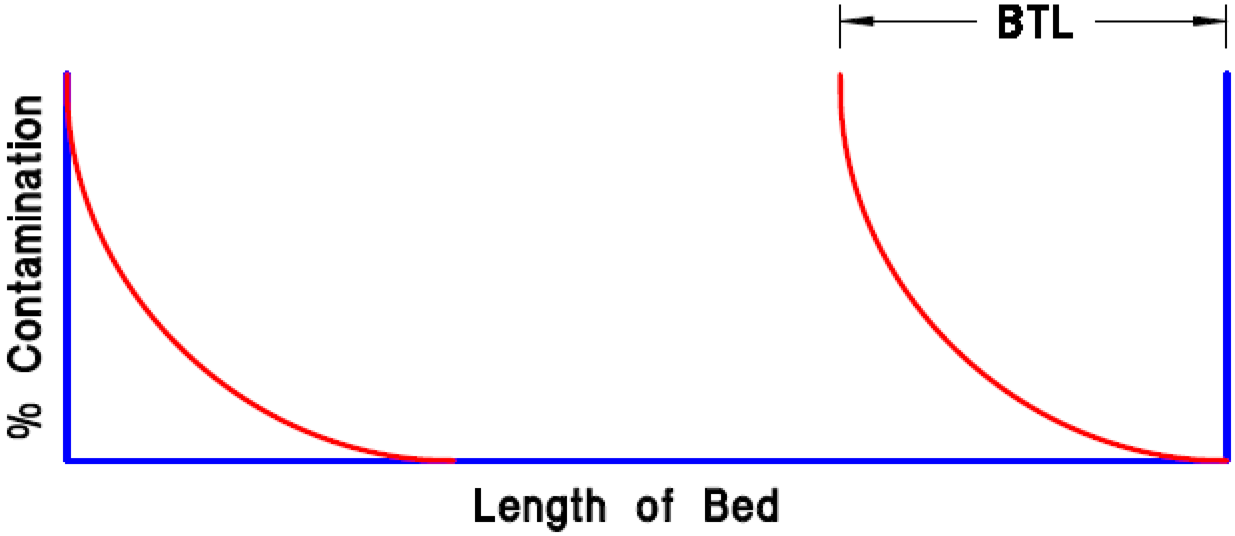
- Pressure Drop
- Ergun Equation

$$\frac{\Delta P}{\mathrm{L}} = \frac{150\,\mu G(1-\varepsilon)^2}{kg\rho D^2} + \frac{1.75 G^2 (1-\varepsilon)}{kg\rho D\varepsilon^3}$$

- Less than 0.1 psi for 6-foot bed height

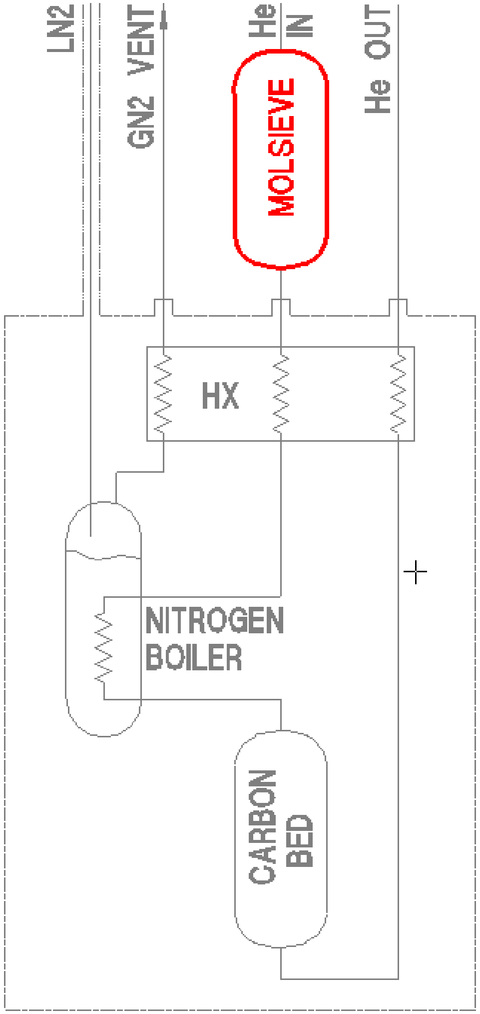
Carbon Bed Design Development

- Final Bed Size
  - 12 NPS SCH-10 (12.39 ID pipe)
  - 6-foot bed height
    - Rounded to nearest foot for ease of fabrication
    - Added ~one foot for break through length (BTL)



Molsieve Bed Design Development

- Bed size for molsieve bed to match carbon-bed size for economical reasons
- Similar to carbon-bed sizing
  - Verify carbon-bed size is adequate for molsieve bed
  - 12.0 lb water adsorbed per 100 lb of activated adsorbent
    - Read from graph provided by vendor
    - Using partial pressure of 0.00191 psi (10ppm @ 13 atm)
  - Yields 77 days before bed needs to be regenerated
    - Compared to 30 days for the Carbon Bed



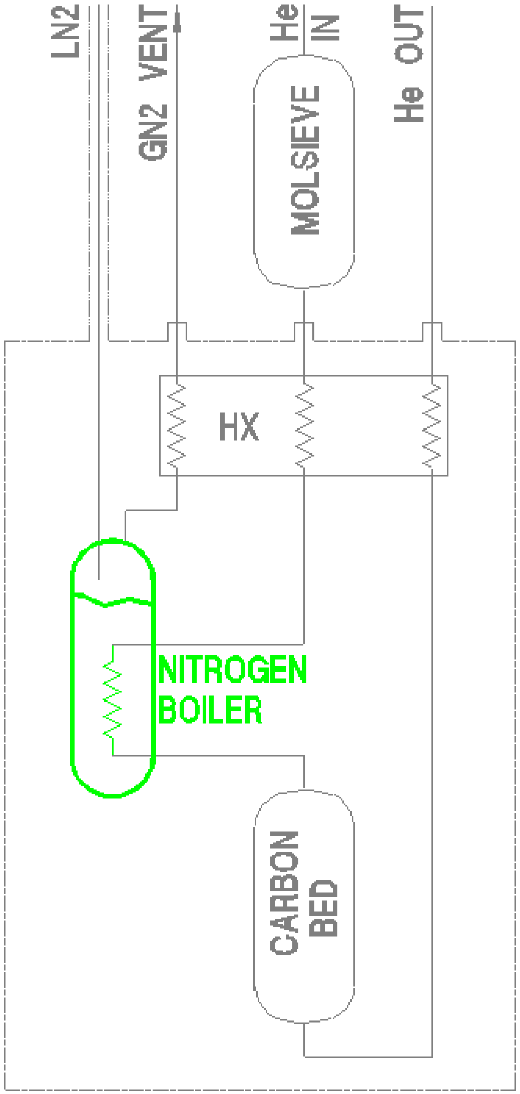
Heat Exchanger Design Development

- Heat exchanger fabricated by vendor to meet following specifications:

HX-Design Data		**Stream-1**	**Stream-2**	**Stream-3**
Design Data	**UNITS**	**Helium**	**Helium**	**Nitrogen**
Flow (gas)	g/s	40	40	5.5
Warm End Temp-A	K	310	300	300
Cold End Temp-A	K	84.6	80	80
Warm End Pressure	Atm	12	11.3	1.1
Maximum Pressure Drop	Atm	0.1	0.1	0.1
Design Pressure	Atm	20	20	20
Design Temperature Max	K	400	400	400
Design Temperature Min	K	60	60	60

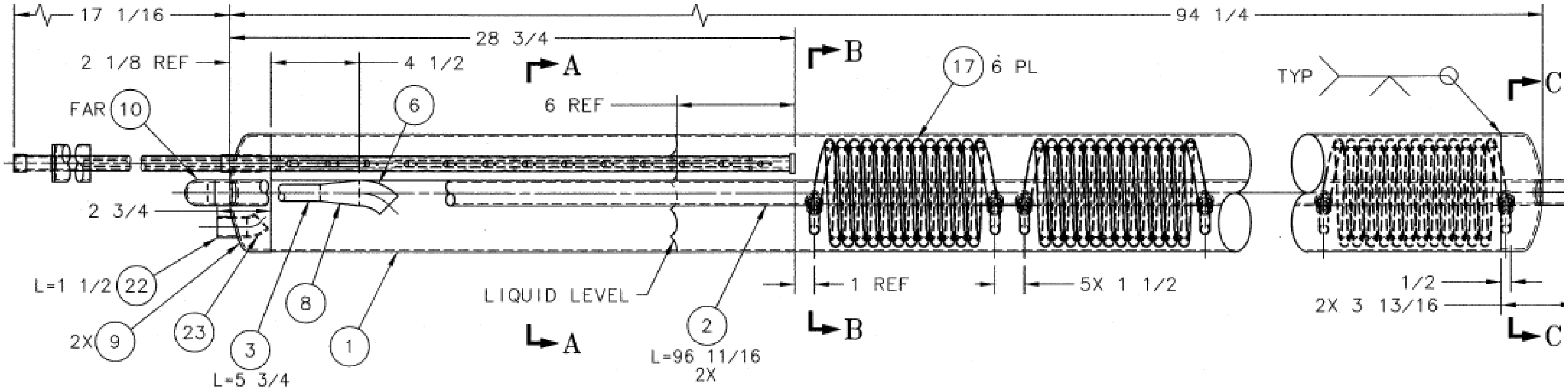
Nitrogen Boiler Design Development

- Tube-in-shell design
- 6 – ½ inch OD x 20-foot long tubes
- Designed not to be a code stamped pressure vessel
  - Able to fabricate at J-Lab
  - Diameter limited to less than 6 inches
- Checked velocity of nitrogen vapor to be less than 20 cm/s
- 5.5 g/s of nitrogen flow specified from heat exchanger yields 6.9 cm/s



Nitrogen Boiler Design Development

- Height required
- All coils are covered with liquid nitrogen
- Minimum 6 inches above the top coil
- 12 inches between top of liquid level and bottom of the liquid fill port
- With LN_2 fill valve closed, there is 30 minutes of stored N_2 to support the process.

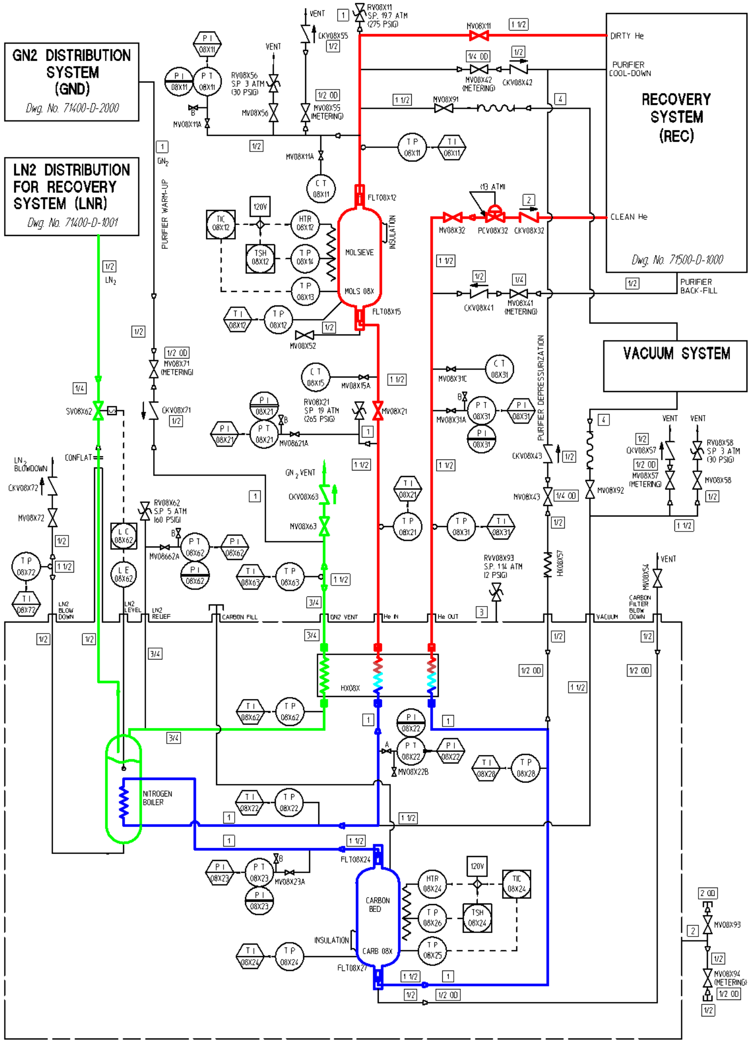


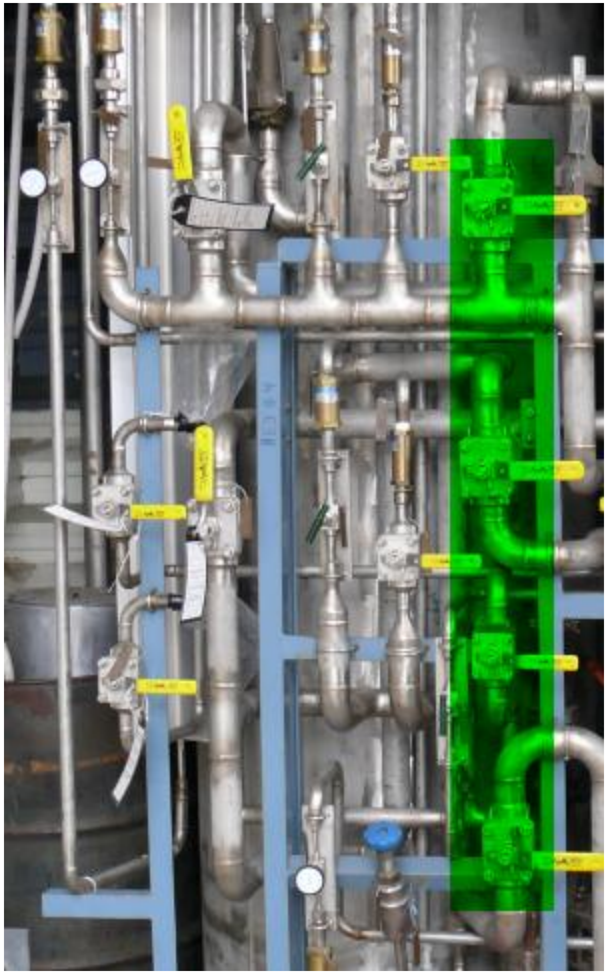
Engineering Design Considerations

1. Modes of Operation
2. Process
    - a) Major components
    - b) Valve sizes
3. Geometry
    - a) Location
    - b) Size
    - c) Orientation
4. Mechanical Supports
    - a) Thermally resistant
    - b) Shipping
    - c) Flexible for thermal expansion
5. Thermal Stability
    - a) Heat leak into the system
    - b) Temperature gradients across valves
        1. Ganni Loops (heat raises)
    - c) Process flow from cold bottom to warm top
6. Mechanical Flexibility in Piping
    1. Stress calculation
    1. Loops
    2. Flex hoses
7. Fabrication Process
    1. Can it be built?

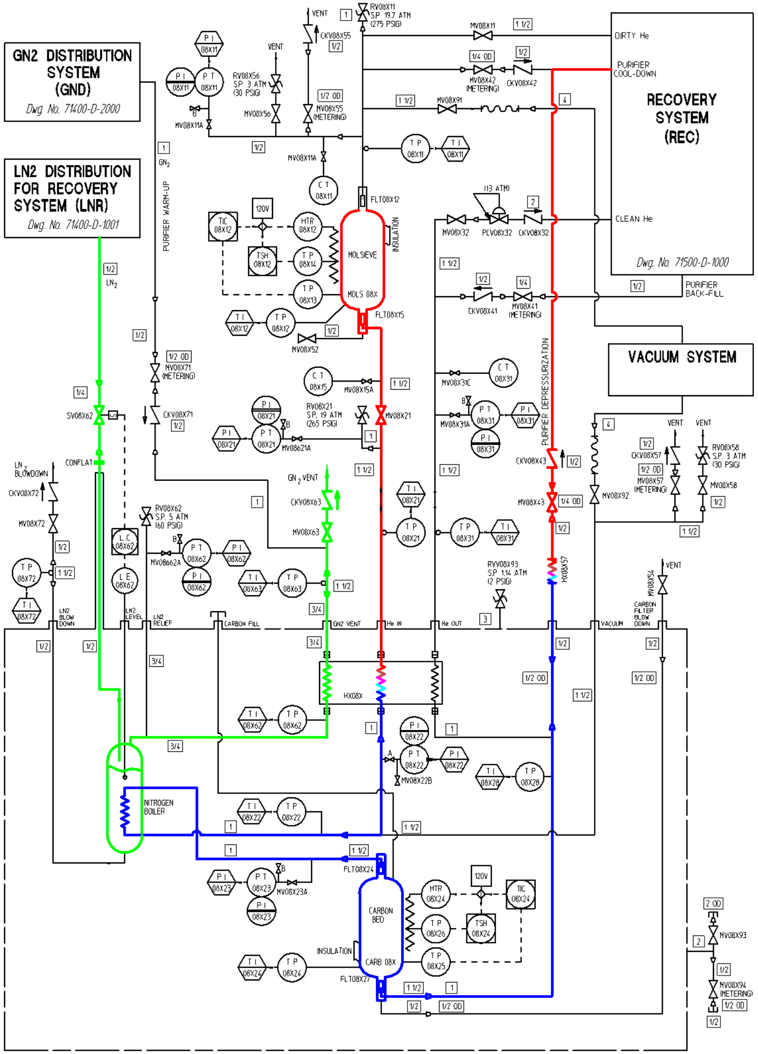
8. Iteration back to step 1

0 - Normal Purifier Process

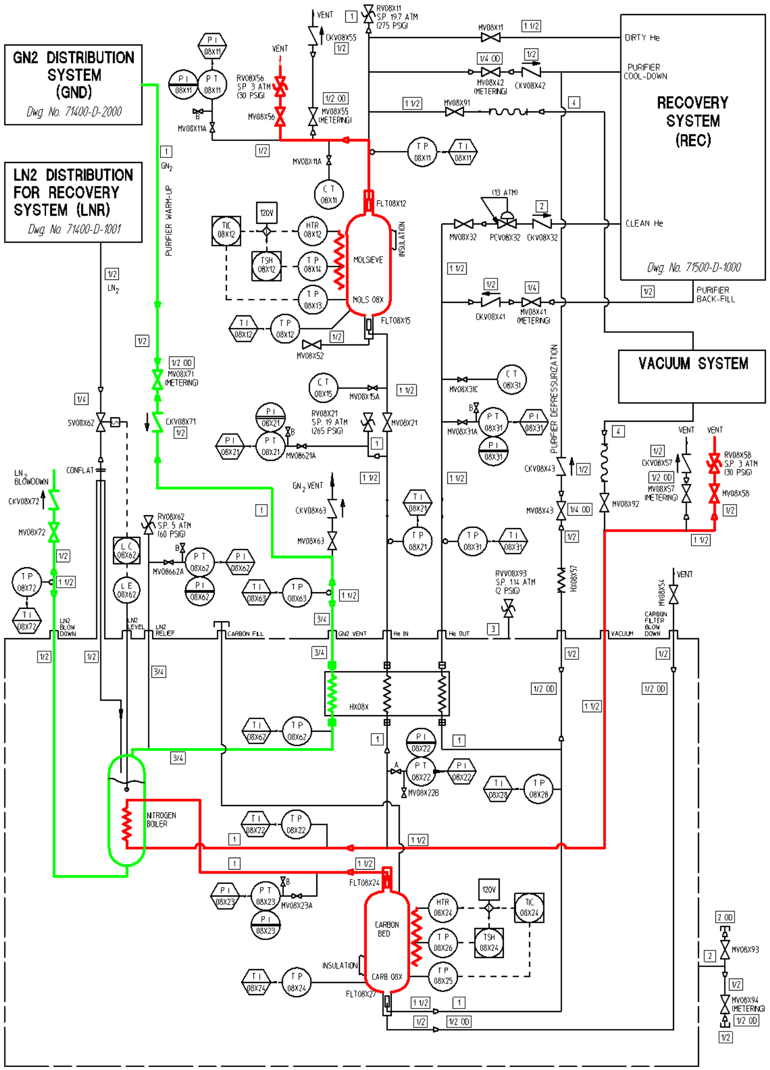


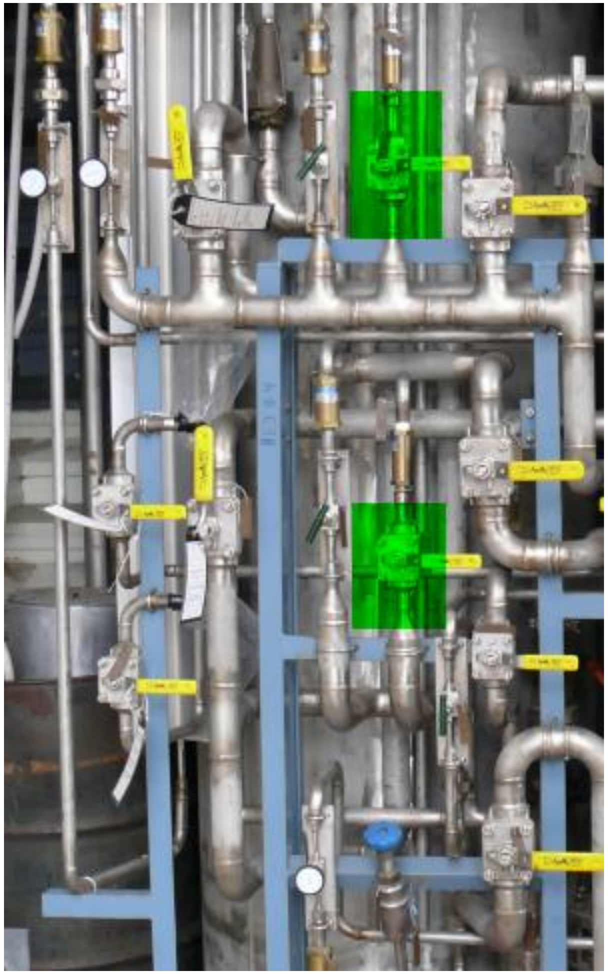


1 - Helium Conservation High Pressure Blow Down

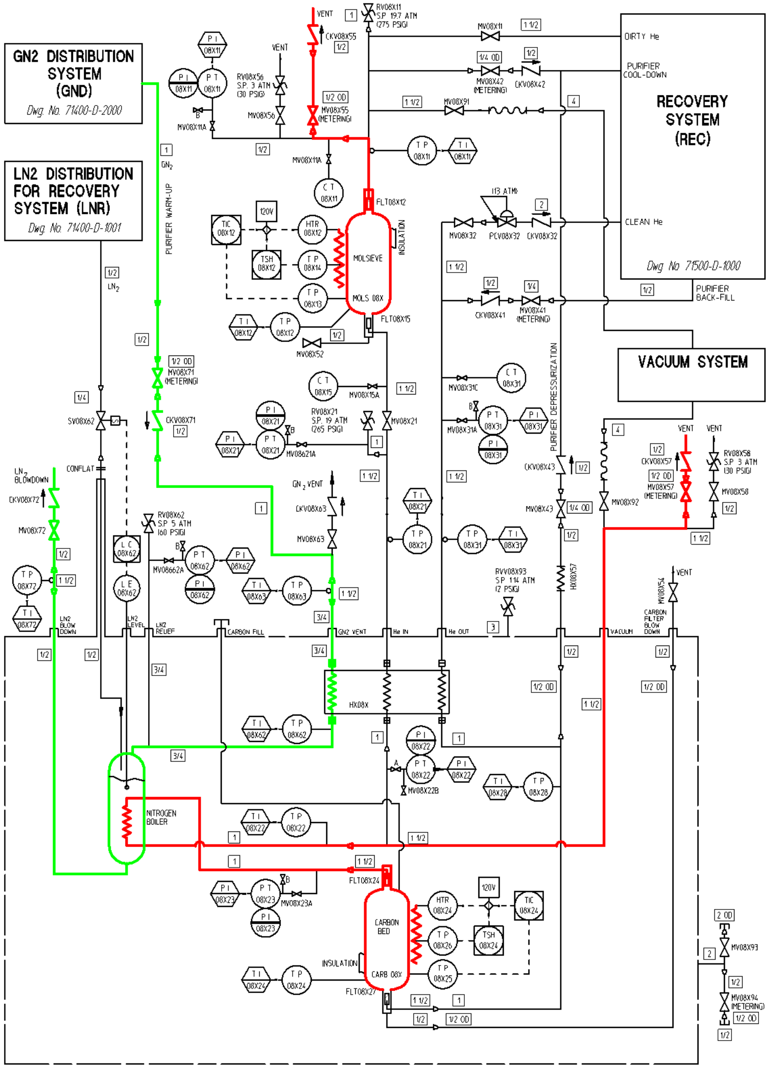


2 - Blow Down to Medium Pressure & Warming

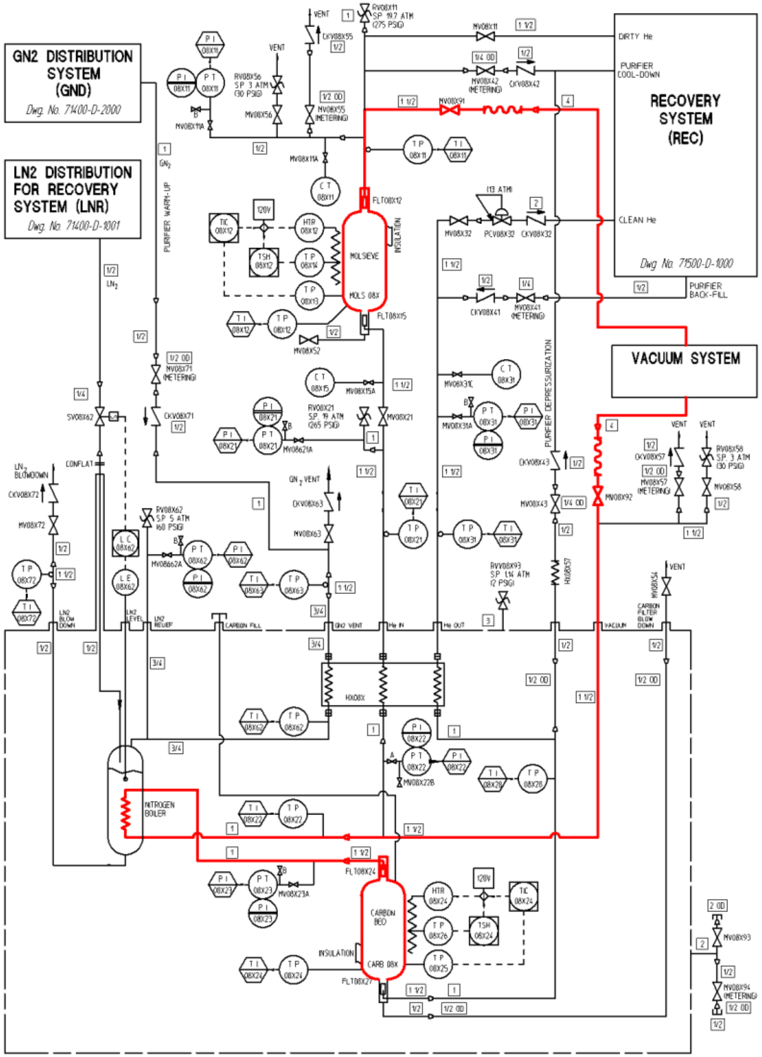


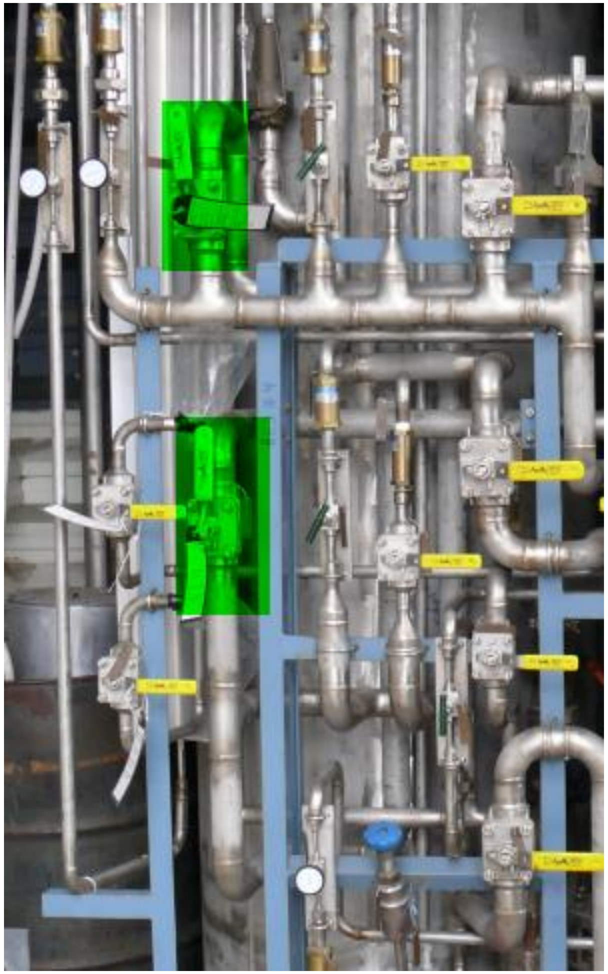


3 - Blow Down to Low Pressure

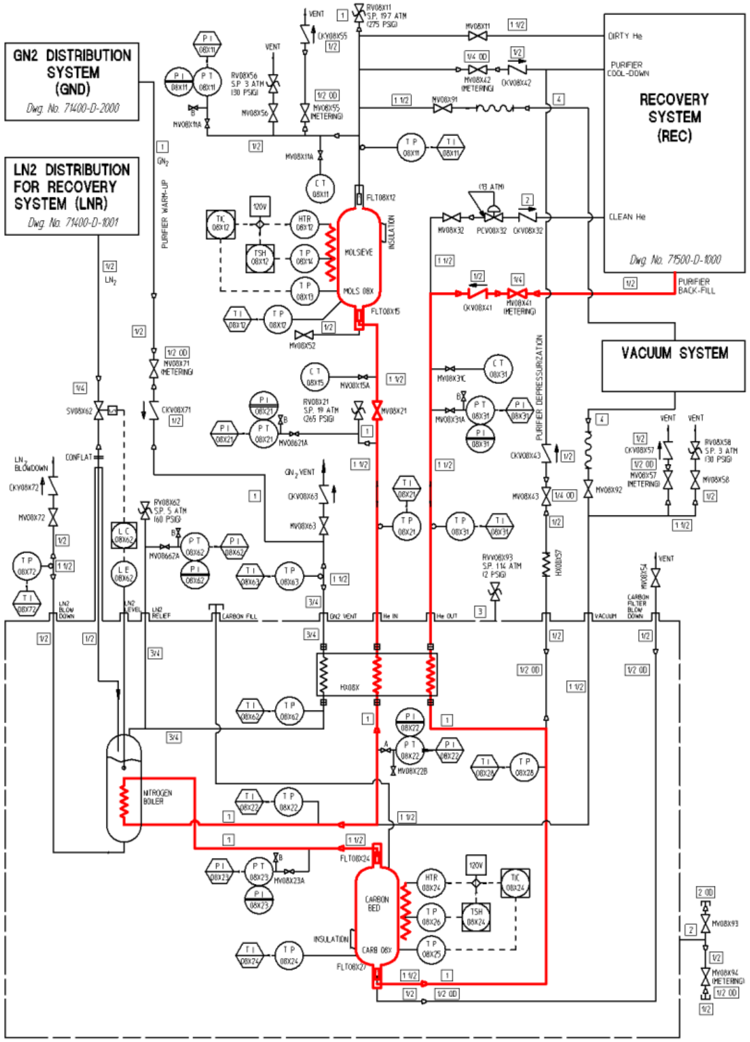


4 – Regeneration Vacuum

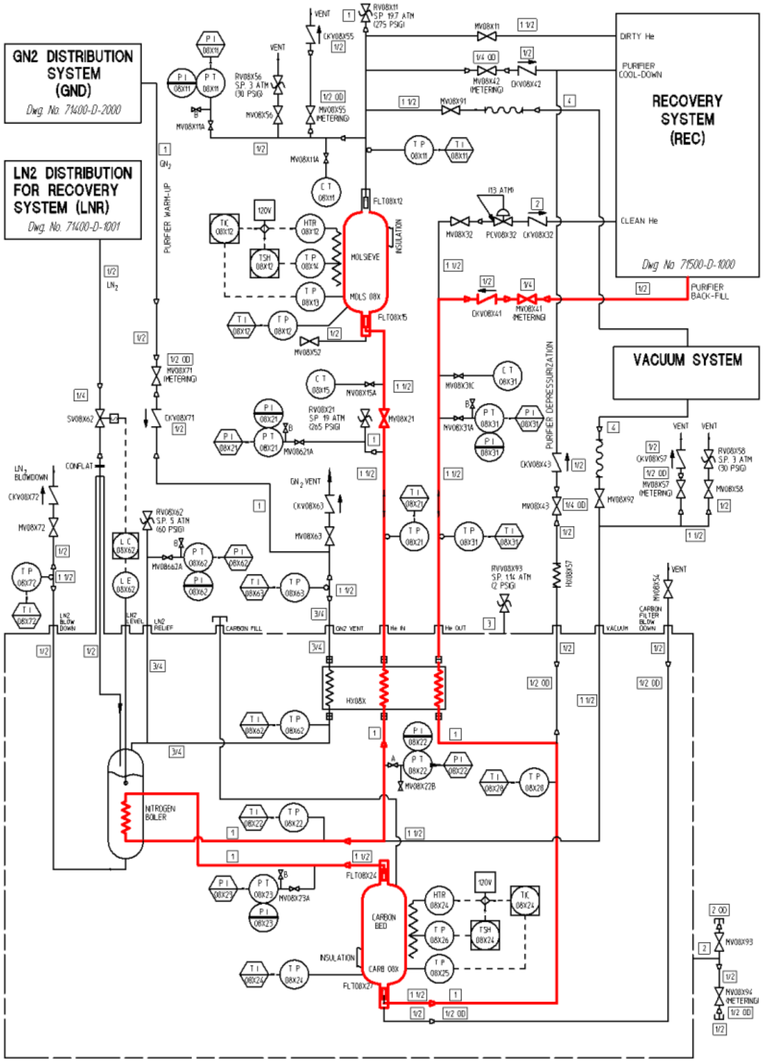




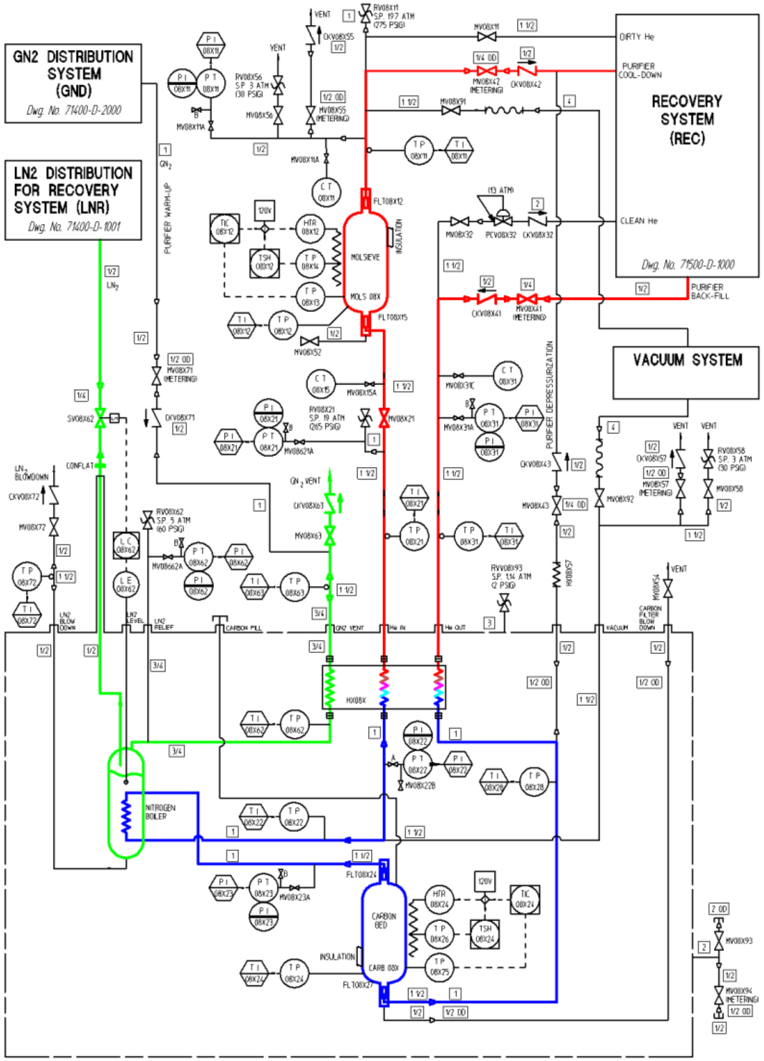
5 – Regeneration Backfill w/ 150ºF/350ºF Heat On



6 – Backfill to Normal Operational Pressure



7 – Cool Down

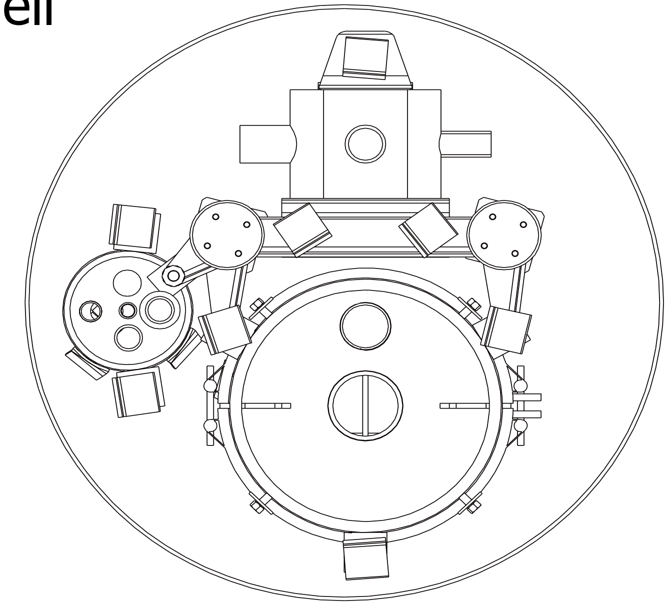

GN2 DISTRIBUTION SYSTEM (GND)
Dwg. No. 71400-D-2000
LN2 DISTRIBUTION FOR RECOVERY SYSTEM (LNR)
Dwg. No. 71400-D-1001
RECOVERY SYSTEM (REC)
Dwg. No. 71500-D-1000
DIRTY He
PURIFIER COOL-DOWN
CLEAN He
PURIFIER BACK-FILL
VACUUM SYSTEM
PURIFIER WARM-UP
PURIFIER DEPRESSURIZATION
MOLSIEVE
MOLS 08X
INSULATION
HX08X
NITROGEN BOILER
CARBON BED
CARB 08X
FLT08X12
FLT08X15
FLT08X24
FLT08X27
RV08X11 SP. 19.7 ATM (275 PSIG)
RV08X21 SP. 19 ATM (265 PSIG)
RV08X62 SP. 5 ATM (60 PSIG)
RV08X56 SP. 3 ATM (30 PSIG)
RV08X58 SP. 3 ATM (30 PSIG)
RVV08X93 SP. 1.14 ATM (2 PSIG)
(13 ATM)
MV08X42 (METERING)
CKV08X42
MV08X41 (METERING)
CKV08X41
PCV08X32
CKV08X32
MV08X32
MV08X91
SV08X62
LN2 BLOWDOWN
CONFLAT
GN2 VENT
He IN
He OUT
CARBON FILL
LN2 RELIEF
LN2 LEVEL
CARBON FILTER BLOW DOWN
VACUUM
VENT
120V

Purifier Regeneration Valve Positions

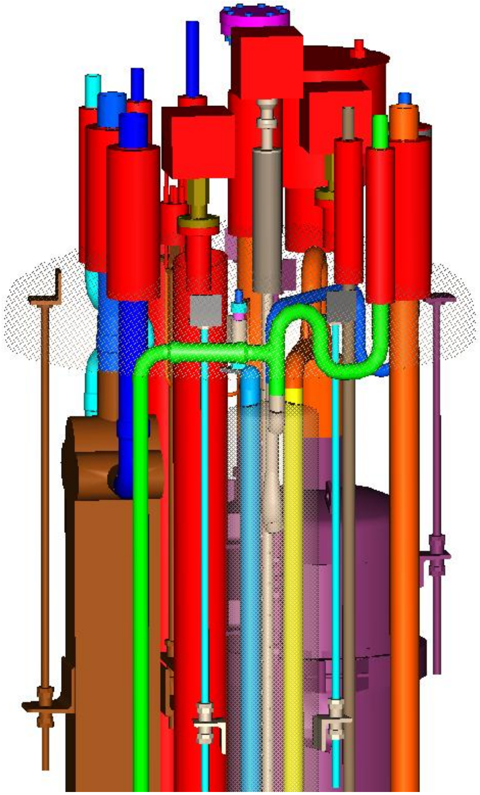
Transient Processes				0	1	2	3	4	5	4	5	4	5	4	6	7	0
Isolation to Molsieve Bed	MV08x 11	ball	1 1/2	**on**	off	off	off	off	off	off	off	off	off	off	off	off	**on**
HX Inlet Isolation	MV08x 21	ball	1 1/2	**on**	**on**	off	off	off	**on**	off	**on**	off	**on**	off	**on**	**on**	**on**
GN2 Vent Isolation from HX	MV08x 63	ball	1	**on**	**on**	off	off	off	off	off	off	off	off	off	off	**on**	**on**
Carbon Bed Discharge Isolation	MV08x 32	ball	1 1/2	**on**	off	off	off	off	off	off	off	off	off	off	off	off	**on**
Carbon Bed Back-Press./Vent Isolation	MV08x 43	ball	1/2	off	**on**	off	off	off	off	off	off	off	off	off	off	off	off
GN2 Supply to HX	MV08x 71	metering	1/2	off	off	**on**	**on**	off	off	off	off	off	off	off	off	off	off
GN2/LN2 Purge/Vent from LN2-Cooler	MV08x 72	globe	1/2	off	off	**on**	**on**	off	off	off	off	off	off	off	off	off	off
Molsieve Bed Blow-Down Isolation	MV08x 55	metering	1/2	off	off	off	**on**	off	off	off	off	off	off	off	off	off	off
Carbon Bed Blow-Down Isolation	MV08x 57	metering	1/2	off	off	off	**on**	off	off	off	off	off	off	off	off	off	off
Molsieve Bed Vacuum Header Isolation	MV08x 91	ball	1 1/2	off	off	off	off	**on**	off	**on**	off	**on**	off	**on**	off	off	off
Carbon Bed Vacuum Header Isolation	MV08x 92	ball	1 1/2	off	off	off	off	**on**	off	**on**	off	**on**	off	**on**	off	off	off
Cool-down & Purge Gas from Molsieve Bed	MV08x 42	metering	1/4	off	off	off	off	off	off	off	off	off	off	off	off	**on**	off
Molsieve Bed Outlet Filter Purge	MV08x 52	ball	1/2	off	off	off	off	off	off	off	off	off	off	off	off	**on**	off
Carbon Bed Outlet Filter Purge	MV08x 54	ball	1/2	off	off	off	off	off	off	off	off	off	off	off	off	**on**	off
Cool-down & Purge Gas to HX	MV08x 41	metering	1/4	off	off	off	off	off	**on**	off	**on**	off	**on**	off	**on**	**on**	off
Molsieve Bed Back-Press./Vent Isolation	MV08x 56	ball	1/2	off	off	**on**	off	off	off	off	off	off	off	off	off	off	off
Carbon Bed Back-Press./Vent Isolation	MV08x 58	ball	1/2	off	off	**on**	off	off	off	off	off	off	off	off	off	off	off
	LN_2	Solenoid		**on**	**on**	off	off	off	off	off	off	off	off	off	off	**on**	**on**
	Heaters			off	off	**on**	**on**	off	**on**	off	**on**	off	**on**	off	off	off	off

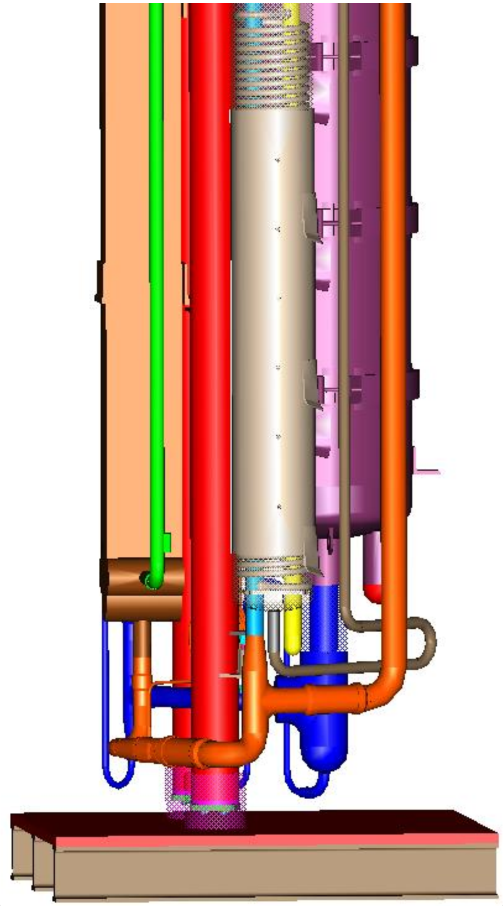
Purifier Layout

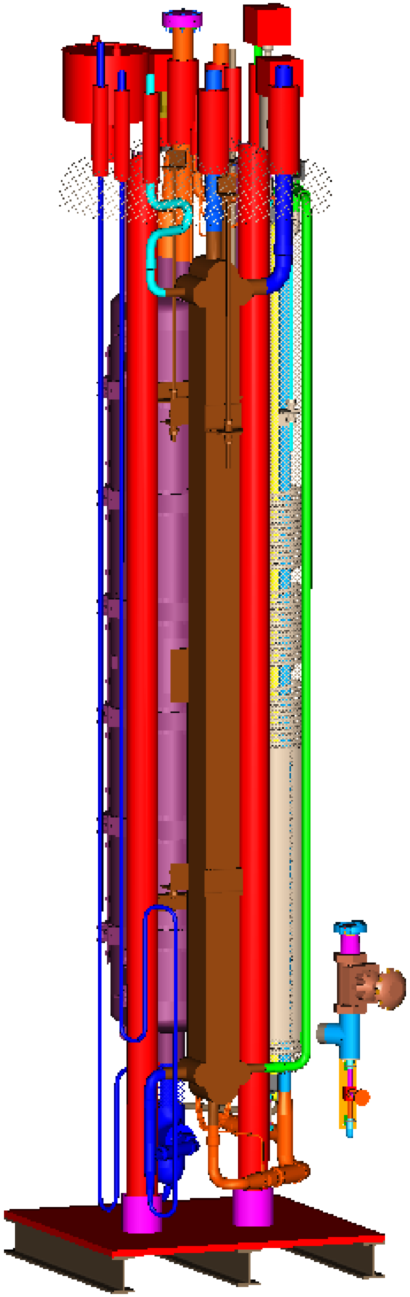
- Maintain the smallest amount of space possible
  - 30-inch diameter vacuum shell used
    - ASME head used on top
    - Flat sheet of steel used on bottom
- Support of components inside vacuum shell
  - Everything is welded to the top head
    - Thermal stability
    - Thermal isolation
    - Fabrication sequence
    - Shipping requirements



Purifier Vacuum Vessel Design Development







Code Calculations

- Code Requirements
  - American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, Section VIII, Rules for Construction of Pressure Vessels
  - ASME Code for Pressure Piping, B31. The ASME Code B31.3, Process Piping
- Vendors required to perform code calculations for:
  - Heat exchanger
  - Carbon / molsieve beds
- Calculations for LN_2 Boiler were subject to ASME code vessel requirements, even though it is not considered a pressure vessel according to the code.
  - Shell thickness (UG-27)
  - Head thickness (UG-32)
  - Head reinforcement area (UG-37)

Code Calculations

- Similar to the LN_2 boiler, the vacuum shell is constrained to the code.
  - Shell thickness (UG-28)
  - Ellipsoidal / torispherical head thickness (UG-33)
  - Ellipsoidal / torispherical head reinforcement area (UG-37)
  - Flat plate thickness (UG-34)

- Minimum pipe wall thicknesses
  - Straight Pipe Under Internal Pressure - ASME B31.3 ¶304.1.2
    - Using Hoop Calculations

Piping Analysis

Hoop Stress

p_i 350 psi

Alowable Stress 16,800 psi

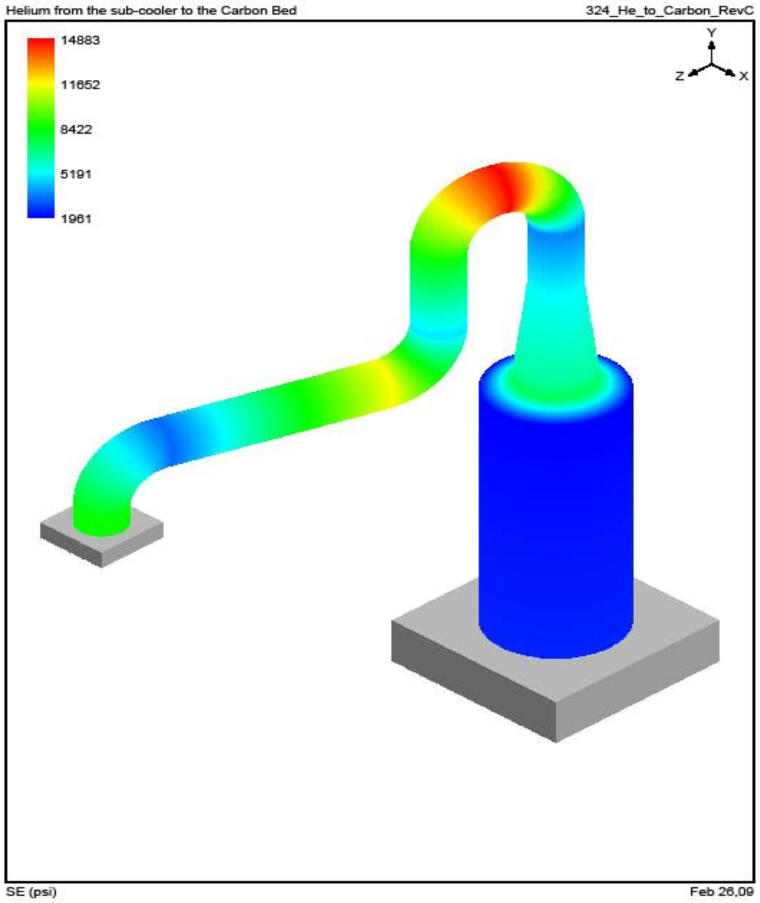
For a thin wall cylinder: t/2r = <.1

Pipe Size	$r_o = D/2$	$r_i = d/2$	is t/2r <.1	Can pipe be considered as a thin wall cylinder	$\sigma_h = \frac{pD}{2t}$	$\sigma_a = \frac{pD}{4t}$
	in	in			psi	psi
3/8 (5)	0.3375	0.2885	**0.085**	**yes**	2411	1205
1/2 (5)	0.42	0.355	**0.092**	**yes**	2262	1131
3/4 (5)	0.525	0.46	**0.071**	**yes**	2827	1413
3/4 (10)	0.525	0.442	**0.094**	**yes**	2214	1107
1 (5)	0.6575	0.5925	**0.055**	**yes**	3540	1770
1 (10)	0.6575	0.5485	**0.099**	**yes**	2111	1056
1 1/4 (5)	0.83	0.765	**0.042**	**yes**	4469	2235
1 1/4 (10)	0.83	0.721	**0.076**	**yes**	2665	1333
1 1/2 (5)	0.95	0.885	**0.037**	**yes**	5115	2558
1 1/2 (10)	0.95	0.841	**0.065**	**yes**	3050	1525
2 (5)	1.1875	1.1225	**0.029**	**yes**	6394	3197
2 (10)	1.1875	1.0785	**0.051**	**yes**	3813	1907
2 1/2 (5)	1.4375	1.3545	**0.031**	**yes**	6062	3031

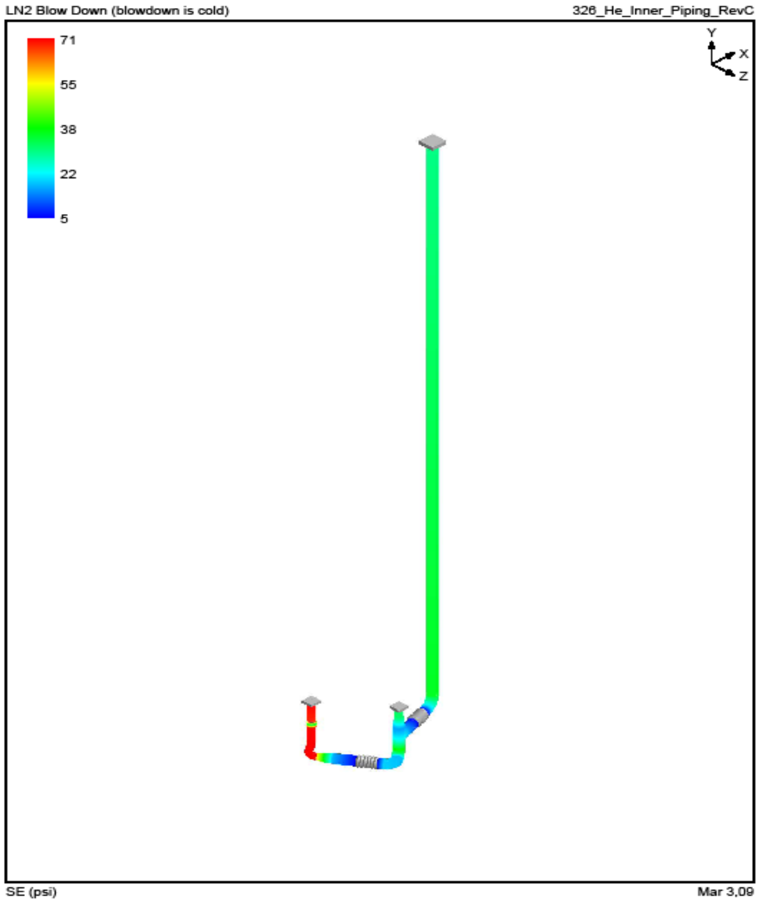
Piping Analysis

- Assumptions:
  - Each pipe spool was evaluated based on maximum theoretical movement due to thermal contractions.
  - Each pipe spool was defined by interface points to the:
    - Vacuum shell
    - Heat exchanger
    - Nitrogen boiler
    - Carbon Bed
- Data were then entered into Caepipe software, and maximum stresses were calculated.

Piping Analysis

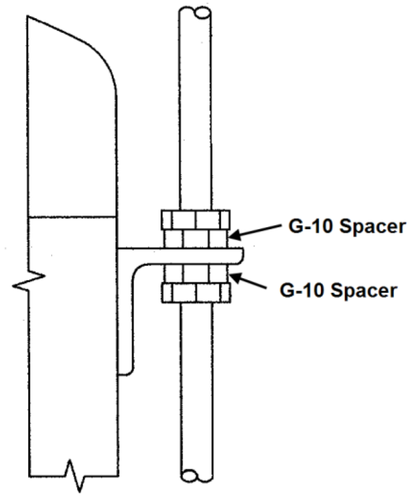


-0324 N2 Boiler to Carbon Bed



-0326 HX to N2 Boiler

Material Selections



- Heat Exchanger
  - Aluminum
    - For heat transfer properties
- Piping
  - SST 304L
    - For low carbon content
      - To minimize the carbon precipitation at the weld joint
      - To reduce non-uniform carbon propagation
    - Regular carbon steel is very brittle at cryogenic temperature
- G-10 where heat insulation is required
- Insulation (MLI & Fiberglass)

Detailed Drawings & Fabrication

- 3D Model utilized
  - Design iterations made to model before generating 2D drawings
    - Reduced design time
  - P&I's utilized to generate model
  - Divided into two sections (vacuum shell &valve rack)
  - All piping and components divided to match fabrication sequence
    - Aided the organization of the piping analysis
    - Helped determine were and how components were built
      - Vacuum shell sections and the valve rack were built in a fabrication shop, moved to an assembly area, and then moved to the final destination.
    - Helped implement ASME requirements

Status

- Vacuum headers are tied in
  - Carbon & molsieve has been regenerated
    - Flowed dry nitrogen through the beds with bed heaters on
    - Pump and backfills completed with N_2 until no indication of moisture
    - Final pump and backfills completed with helium
- Line back to the recovery system has been tied in
  - Trickle flow to recovery system has been established to keep purifiers at positive pressure and to check level of contamination w/ instrumentation on recovery system
- LN_2 and GN_2 have been tied into the system
  - LN_2 system has been tested
- **Ready to tie into the clean and dirty (helium in and out) lines**
  - Requires shutting down the recovery system for ~ a week

Conclusion

- A purifier is a devise that removes trace amounts of contamination (air).
- If the helium is not purified, continuous operations would not be possible.
- Flow rate, level of contamination, temperatures, commercial needs, and ease of operations are the design parameters involved in the design.
- Molsieve and carbon have large surface areas and a large affinity to adsorb moisture, oxygen and nitrogen relative the process conditions.
- The purifier is regenerated using heat, vacuum desorption and backfill process.
- The modes of operations and the design options for the process, geometry, mechanical supports, thermal stability, mechanical flexibility, and fabrication process are considered in the design.
- Key considerations of the fabrication / installation stages are: can it be built, where can it be built, and how will it be installed in the location with the minimum amount of impact to the operating system.