贫氩的获取和提纯

蒙大卫
费米实验室 – 粒子物理部/机械系

中国，北京，高能物理所 - 2011 年 2 月 13-16 日

Depleted Argon Extraction and Purification

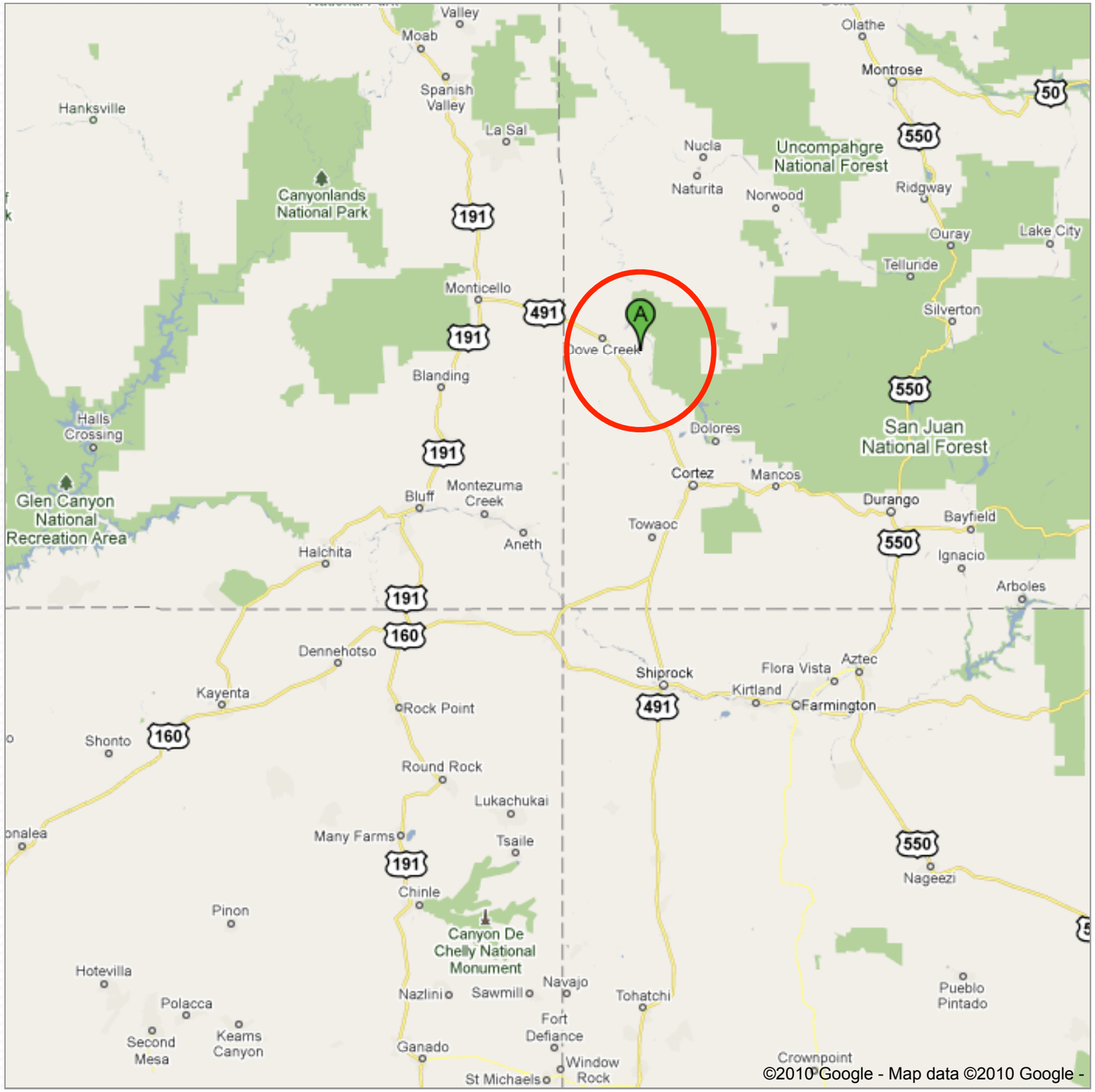
David Montanari
Fermilab - PPD/Mechanical Dept.

IHEP, Beijing, China - February 13-16, 2011

Outline

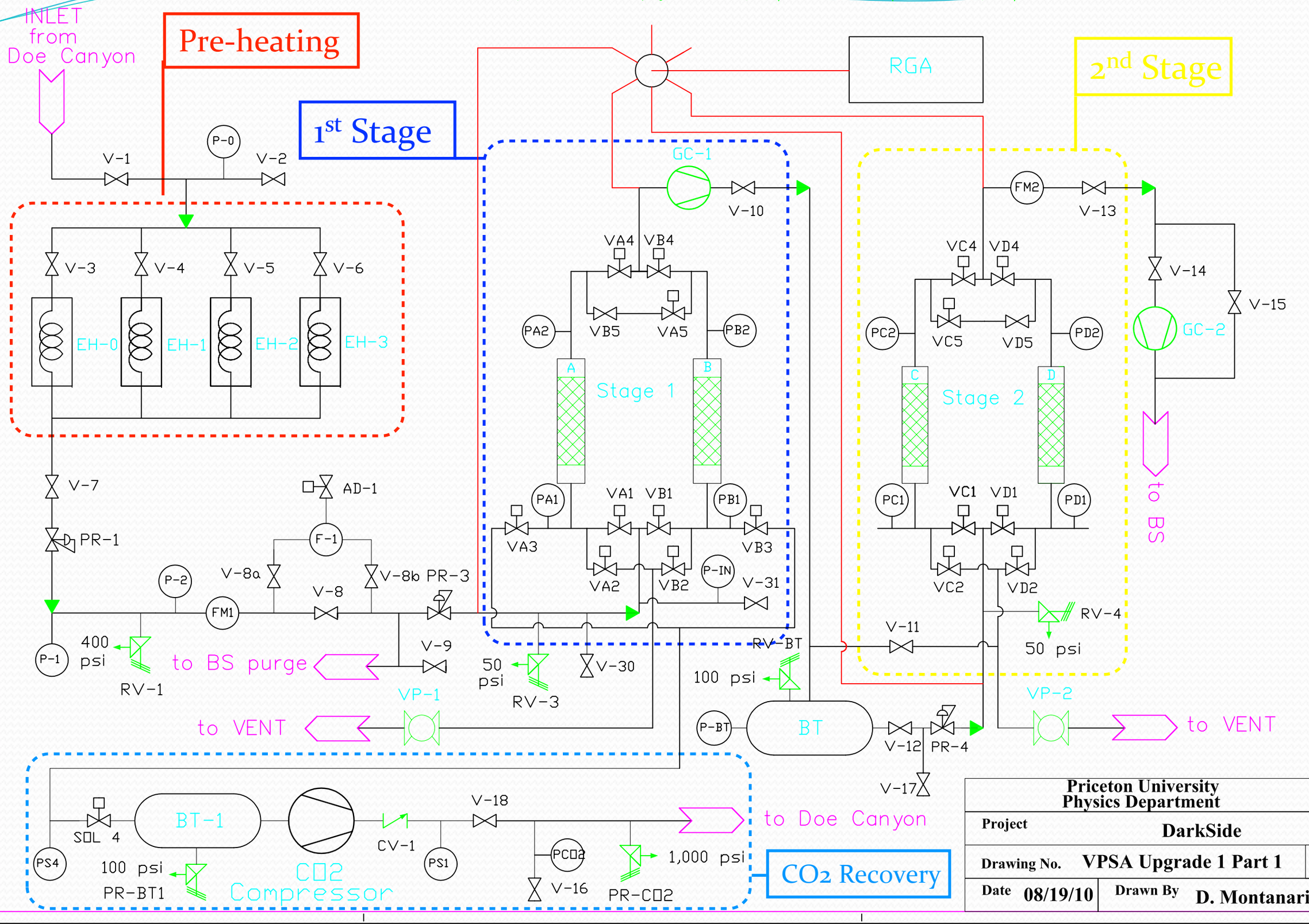
- Intro
- Argon Extraction Plant
- Cryogenic Distillation Plant

Intro


A
Valley
Moab
Spanish Valley
Hanksville
La Sal
Canyonlands National Park
191
Monticello
491
Blanding
Halls Crossing
Glen Canyon National Recreation Area
Bluff
Montezuma Creek
Aneth
Halchita
Dennehotso
160
Kayenta
Rock Point
Shonto
Round Rock
Lukachukai
Many Farms
Tsaile
Chinle
Canyon De Chelly National Monument
Pinon
Hotevilla
Polacca
Second Mesa
Keams Canyon
Nazlini
Sawmill
Navajo
Ganado
Fort Defiance
Window Rock
St Michaels
Tohatchi
Crownpoint
Olathe
Montrose
50
550
Nucla
Naturita
Uncompahgre National Forest
Norwood
Ridgway
Ouray
Lake City
Telluride
Silverton
Dove Creek
Dolores
Cortez
Mancos
San Juan National Forest
Durango
Bayfield
Towaoc
Ignacio
Arboles
Shiprock
Flora Vista
Aztec
Kirtland
Farmington
Nageezi
Pueblo Pintado
©2010 Google - Map data ©2010 Google -

P&ID

EH-0 Electric Heater | EH-1,2,3 Electric Heaters | Stage 1 VPSA Vacuum to 25 psia | Stage 2 VPSA Vacuum to 20 psia | BS Boosting Station 1 to 4,200 psig | PR-1 850 to 250 psi Max IN 2,400 psi | PR-3 250 to 15 psig Max IN 500 psi | PR-4 80 to 6.5 psig Max IN 2,000 psi | RV-BS 4,250 psi | AD-1 Automatic Time Drained Valve



Priceton University Physics Department		
Project	DarkSide	
Drawing No.	VPSA Upgrade 1 Part 1	v.4
Date 08/19/10	Drawn By D. Montanari	

Plant

- Pre-heating system to avoid CO_2 phase transition
- Double stage Vacuum Pressure Swing Adsorption (VPSA) system:
  - 1st Stage removes CO_2
  - 2nd Stage removes most of the N_2
- CO_2 recovery compressor
- Gas Booster to pressurize final storage cylinders
- 15 cylinders crate with gas panel (fill and ship)
- Multi-inlet Mass Spectrometer to measure the composition of the gas from several important points of the plant:
  - Inlet
  - After 1st Stage
  - After 2nd Stage
- Fully automated and LabVIEW controlled
- Leak tightness: $10^{-8}\ mBar/l \cdot s$
- Video Surveillance System

Gas Composition

Component	Feed	Outlet
Ar	480 ppm	**3-4%**
CH_4	5,700 ppm	~800 ppm
CO_2	>96%	n.d.
H_2	~150 ppm	~100 ppm
H_2O	880 ppm	n.d.
He	4,300 ppm	~72%
N_2	2.4%	~24%
O_2	n.d.	**n.d.**

Data

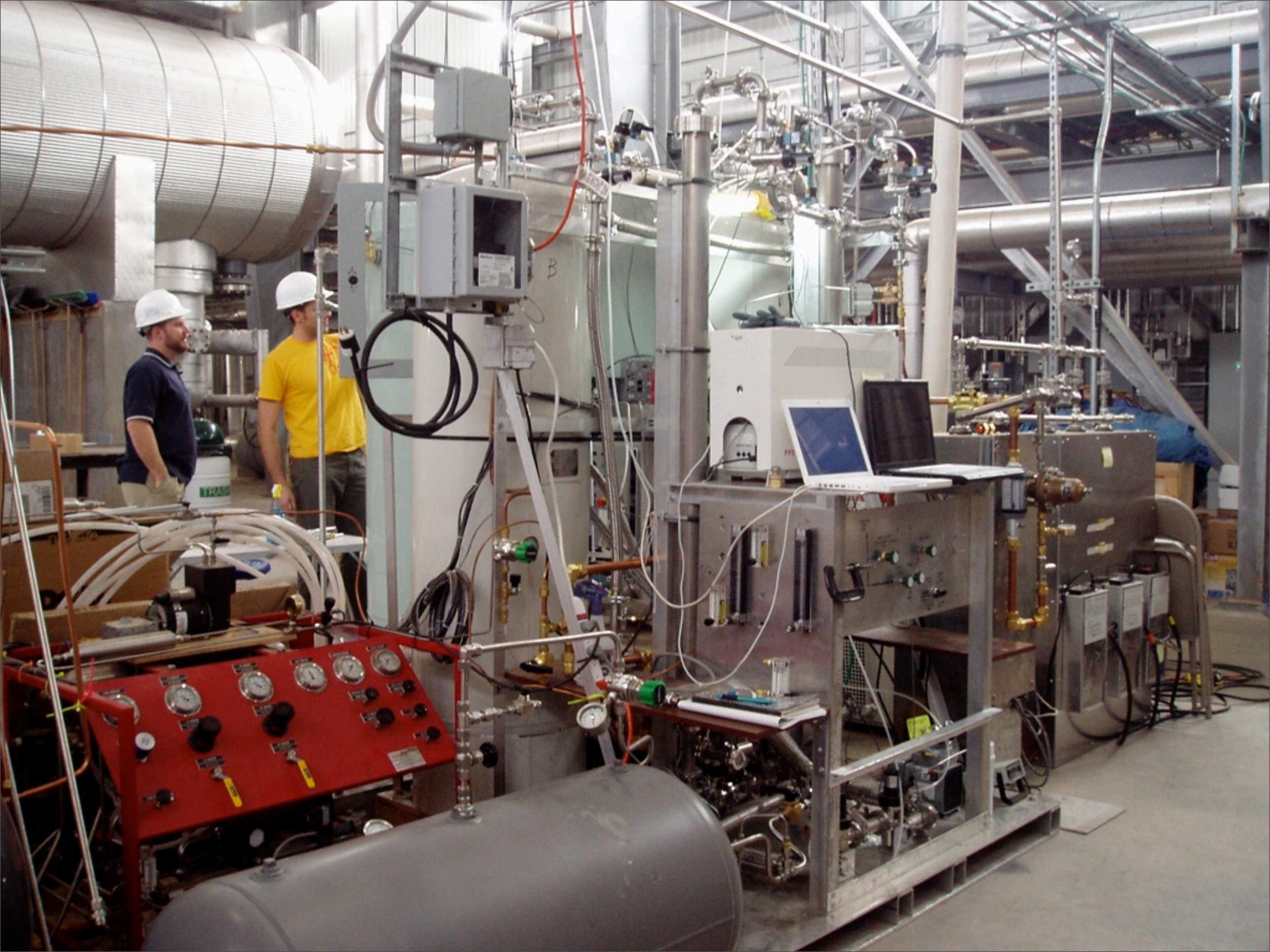
	Feed	After 1st Stage	Outlet
Pressure	780 psig	14.7 psig	10 psig
Temperature	68F	-	60F
Flow Rate	100 SCFM	~50 l/m	8 l/m (FM2)
Duty Cycle	~50%	~67%	~80%
Argon Mass	1.5 Kg/d	1.2-1.3 Kg/d	**0.7-0.8 kg/d**
Ar recovery	-	~80%	~75%

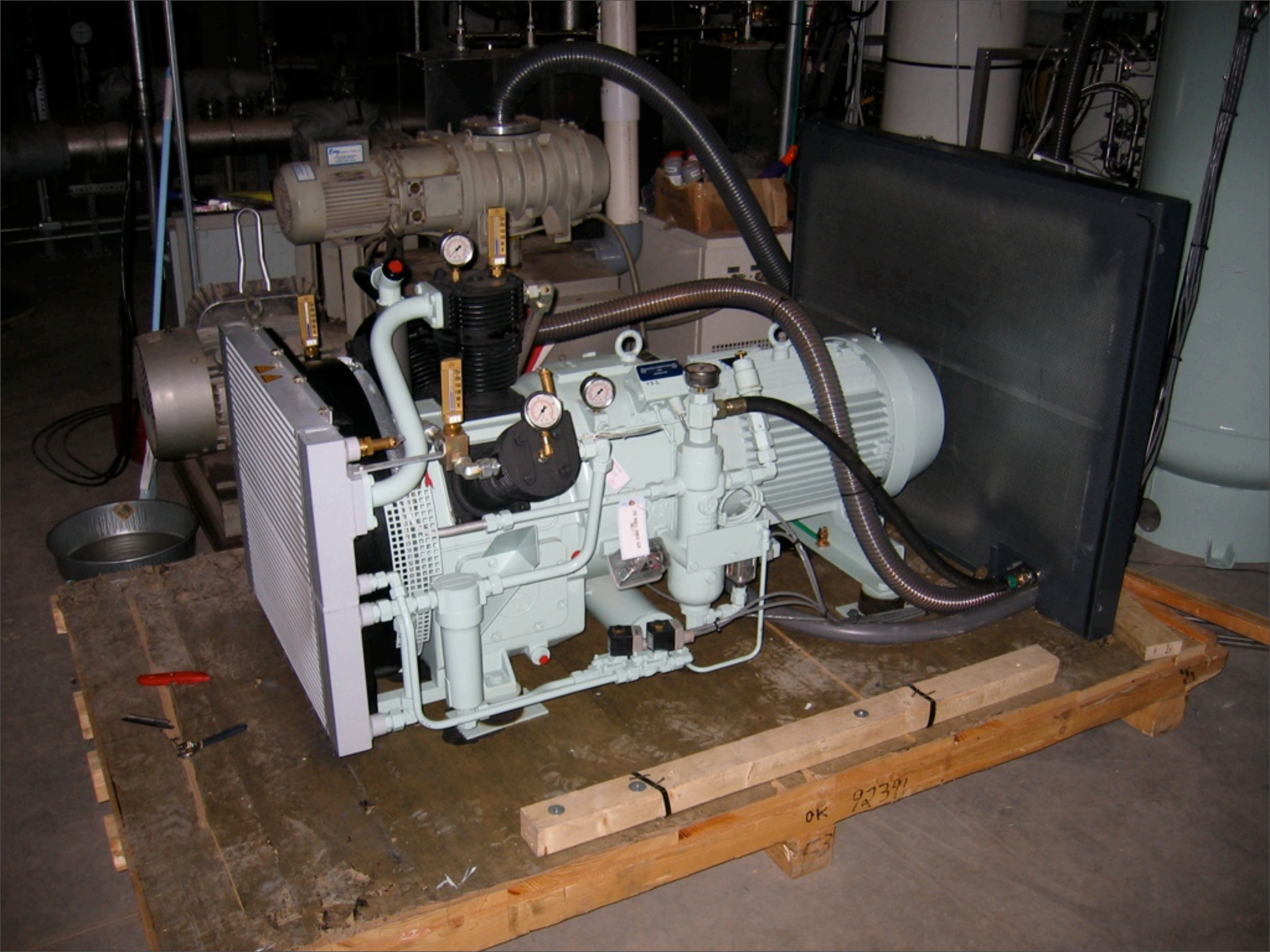
Depletion

The depletion factor of $^{39}Ar/^{40}Ar$ from this underground source is greater than 25 compared to atmospheric Argon

PICS

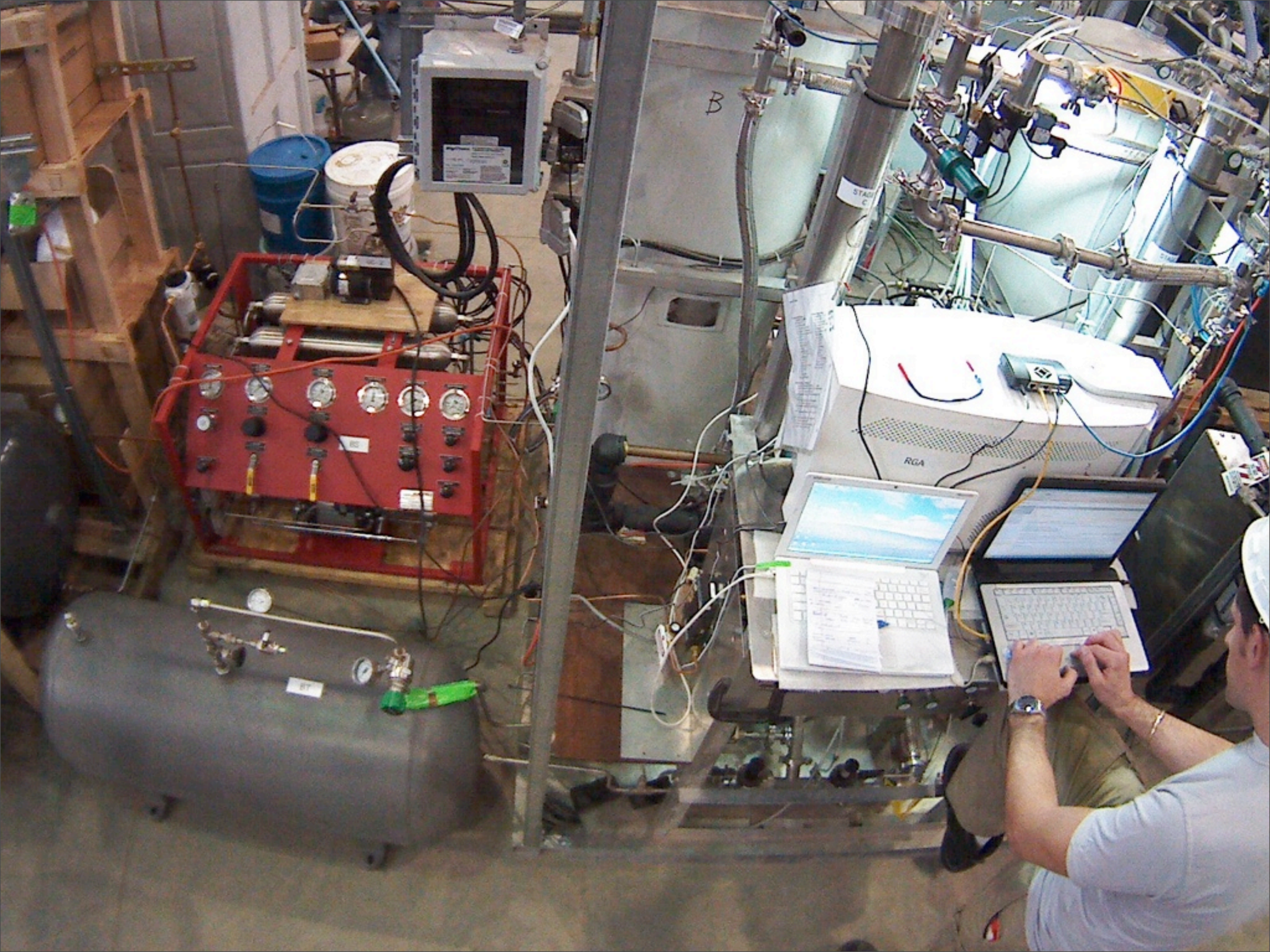
CAUTION




OK 92391

DELL
FRAGILE
PURGE PRESSURE
L.P. BOOSTER AIR PRESSURE
H.P. BOOSTER AIR PRESSURE
INLET PRESSURE
INTERSTAGE PRESS
OUTLET PRESSURE
PURGE REGULATOR
L.P. BOOSTER AIR REGULATOR
H.P. BOOSTER AIR REGULATOR
INTERSTAGE PRESSURE
OUTLET PRESSURE
L.P. BOOSTER AIR SHUT-OFF
L.P. BOOSTER
VENT
PURGE SHUT-OFF
MAXPRO

MAXUS


B
RGA

EXIT

CAUTION

Summary

- The plant is fully automated and can run 24/7
- The Argon collected from this plant is air-free and it shows a reduction factor greater than 25 in ^{39}Ar content compared to atmospheric Argon
- More accurate measure is in progress in the 1kg detector
- The plant has produced ~40 kg of depleted Argon so far
- 2 crates shipped to Fermilab for further purification
- Preliminary storage 25 ft below ground
- The plant has been recently upgraded and it is ready to re-start production with a collection rate of 0.7-0.8 kg/d of depleted Argon (this week)
- The plant supervisor is currently on the field with the new operator (Start-up & Training)

What's next

- Re-start production (this week)
- Training of the new operator on the field
- Target: to produce 120 kg of depleted Argon in the forthcoming months

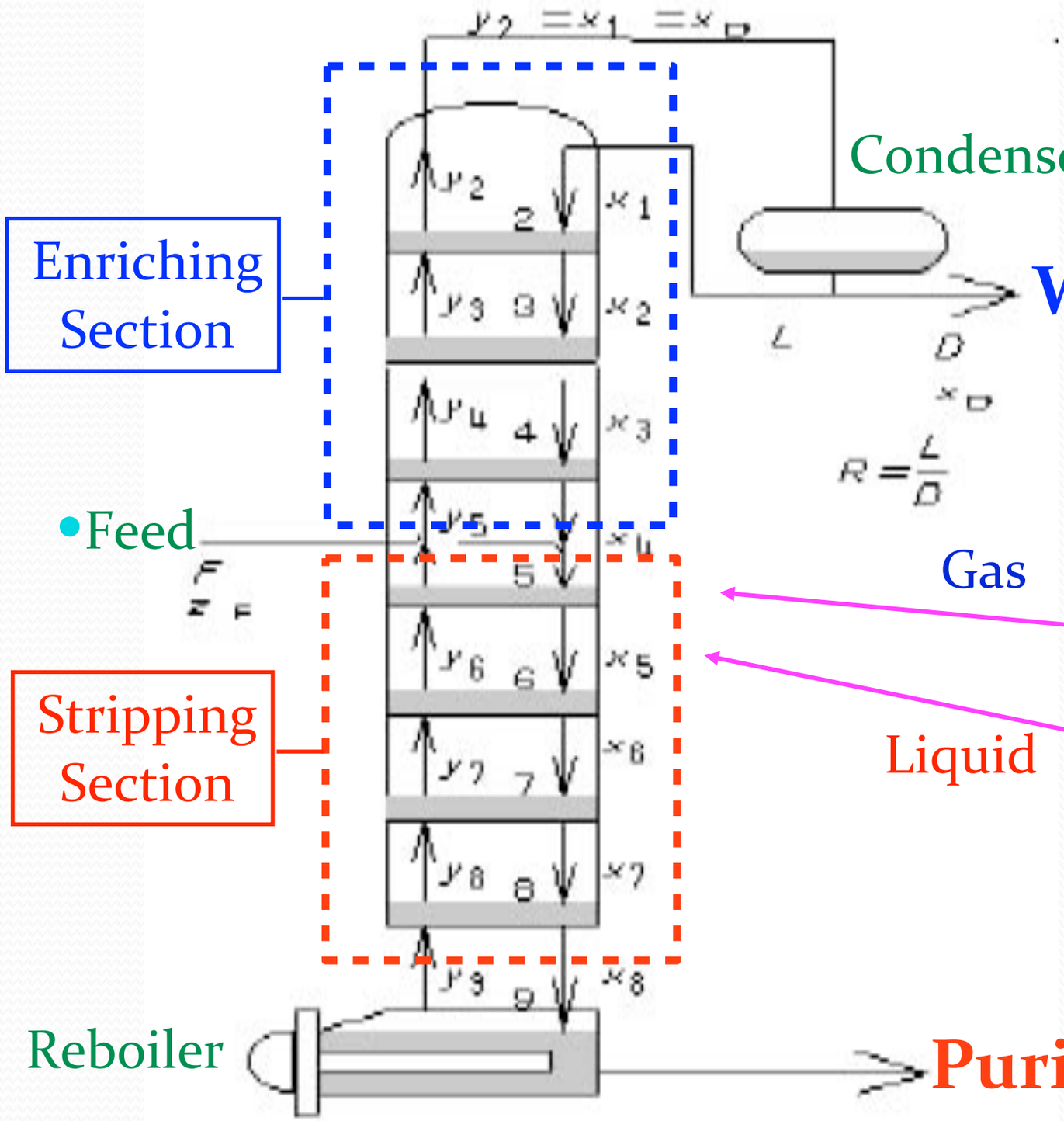
- Expansion of the plant being planned to reach ~10 kg/day of Depleted Argon, suitable for ton-scale detectors

Cryogenic Distillation Column

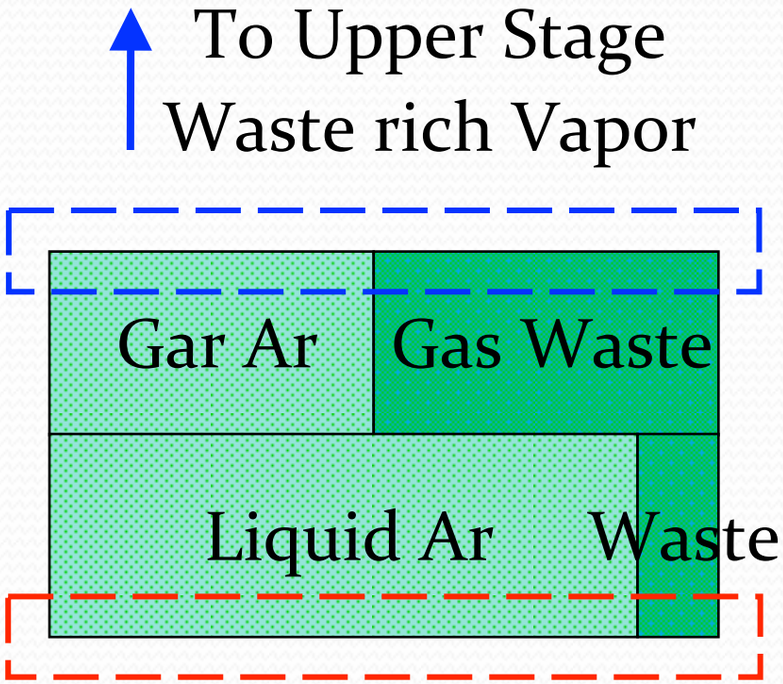
Design Parameters

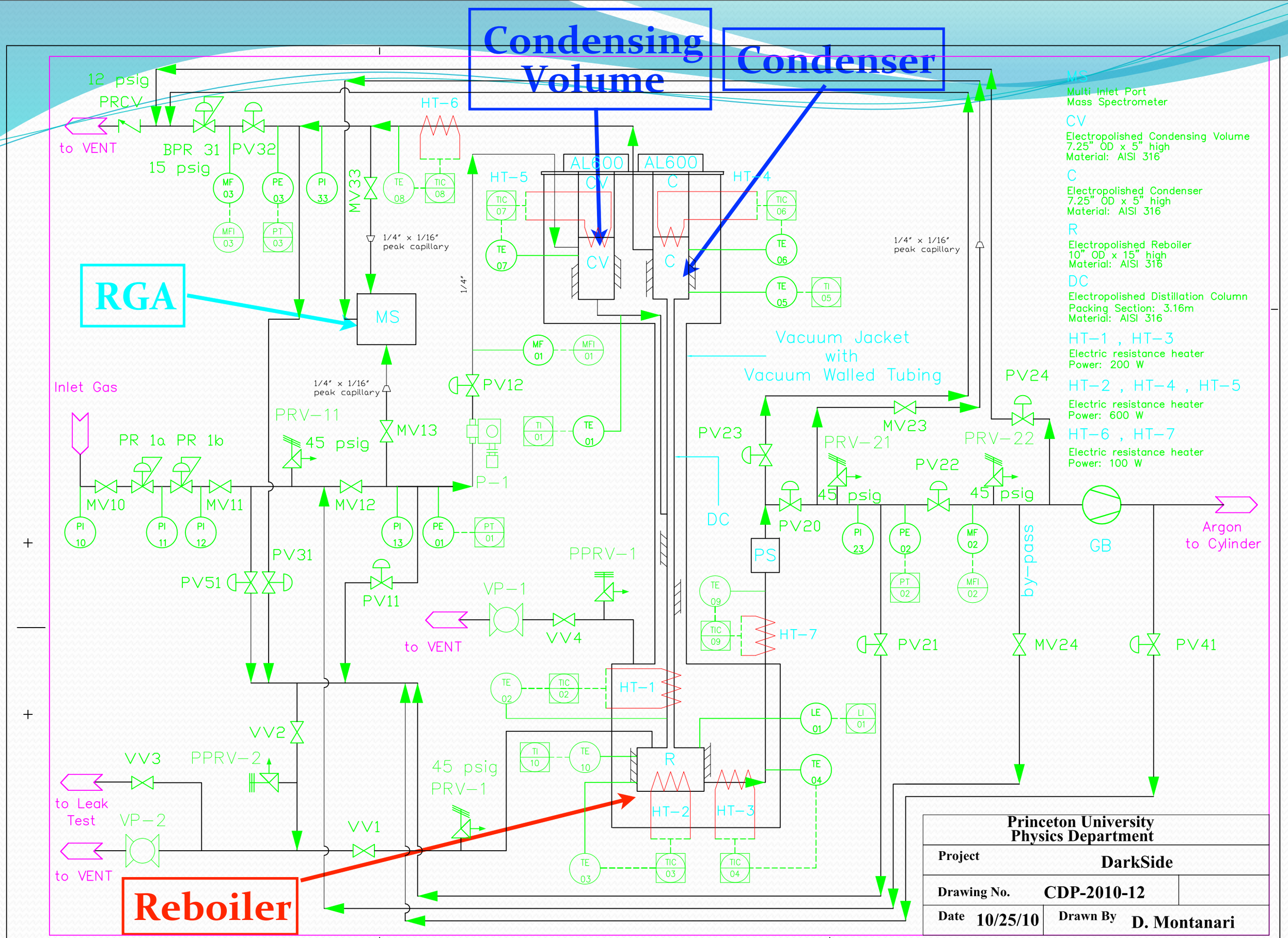
Variable	Value
Throughput	10 kg/d
Ar Purification	**1 ppm N_2 and He**
Ar Recovery	**95%**
Feed Quality	Ar ≥ 5%
Working Pressure	30 psia
Expected HETP	75 mm
Inefficient Packing	80 mm
Inter-bed height	50 mm

How does it work?



- Multiple Stages in a tower
- Each stage is in Vapor – Liquid Equilibrium



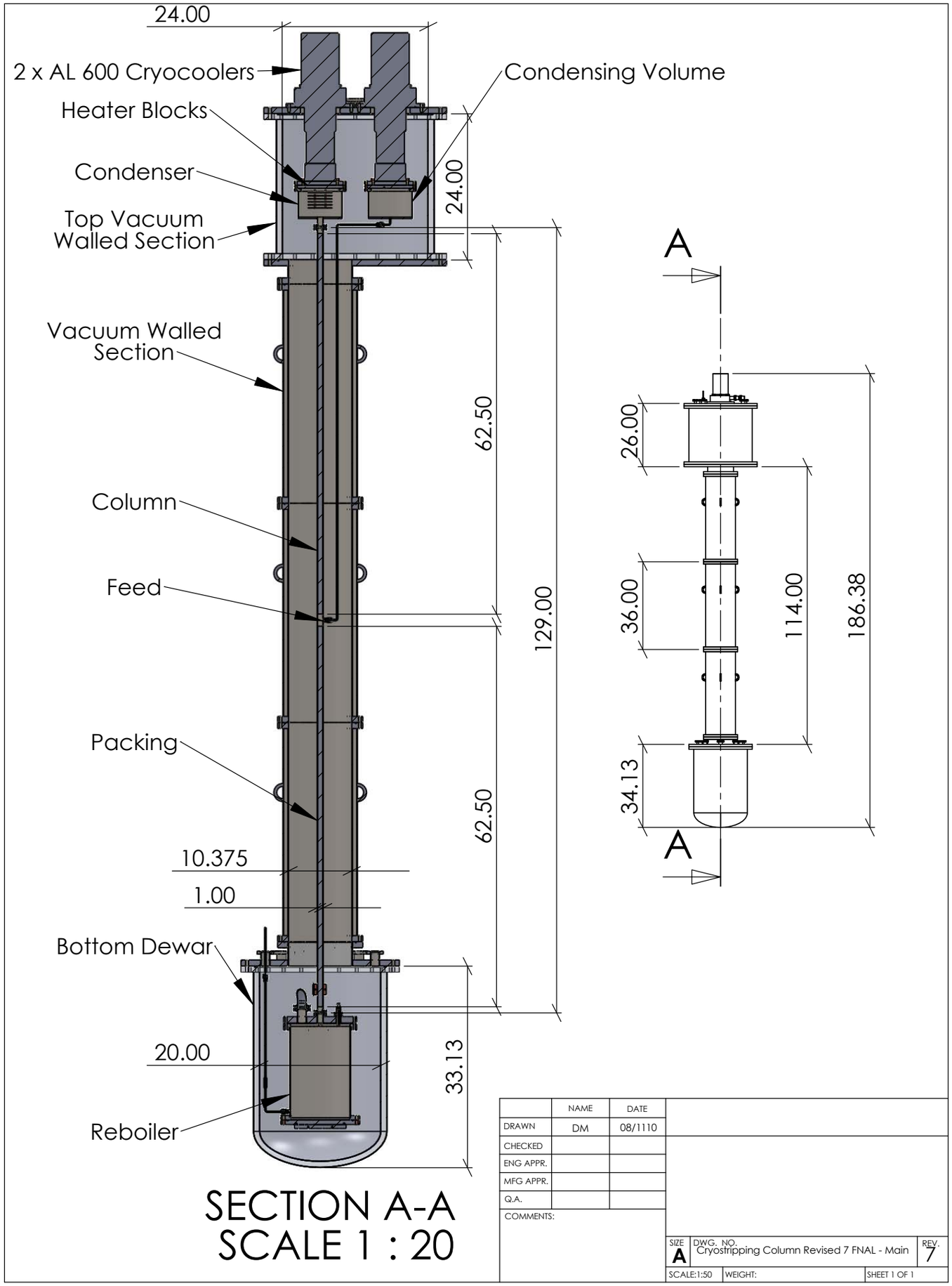

Condensing Volume
Condenser
RGA
Reboiler
12 psig
PRCV
to VENT
BPR 31
15 psig
PV32
HT-6
MV33
1/4" x 1/16" peak capillary
MS
AL600
CV
C
HT-5
HT-4
Vacuum Jacket with Vacuum Walled Tubing
Inlet Gas
PR 1a
PR 1b
PRV-11
45 psig
MV13
PV12
P-1
MV10
MV11
MV12
PV31
PV51
PV11
PPRV-1
VP-1
VV4
to VENT
HT-1
VV2
VV3
PPRV-2
to Leak Test
VP-2
VV1
45 psig
PRV-1
R
HT-2
HT-3
DC
PS
PV23
PV20
PRV-21
PV22
PRV-22
MV23
PV24
GB
Argon to Cylinder
by-pass
HT-7
PV21
MV24
PV41
MS
Multi Inlet Port Mass Spectrometer
CV
Electropolished Condensing Volume
7.25" OD x 5" high
Material: AISI 316
C
Electropolished Condenser
7.25" OD x 5" high
Material: AISI 316
R
Electropolished Reboiler
10" OD x 15" high
Material: AISI 316
DC
Electropolished Distillation Column
Packing Section: 3.16m
Material: AISI 316
HT-1 , HT-3
Electric resistance heater
Power: 200 W
HT-2 , HT-4 , HT-5
Electric resistance heater
Power: 600 W
HT-6 , HT-7
Electric resistance heater
Power: 100 W
Princeton University
Physics Department
Project
DarkSide
Drawing No.
CDP-2010-12
Date
10/25/10
Drawn By
D. Montanari

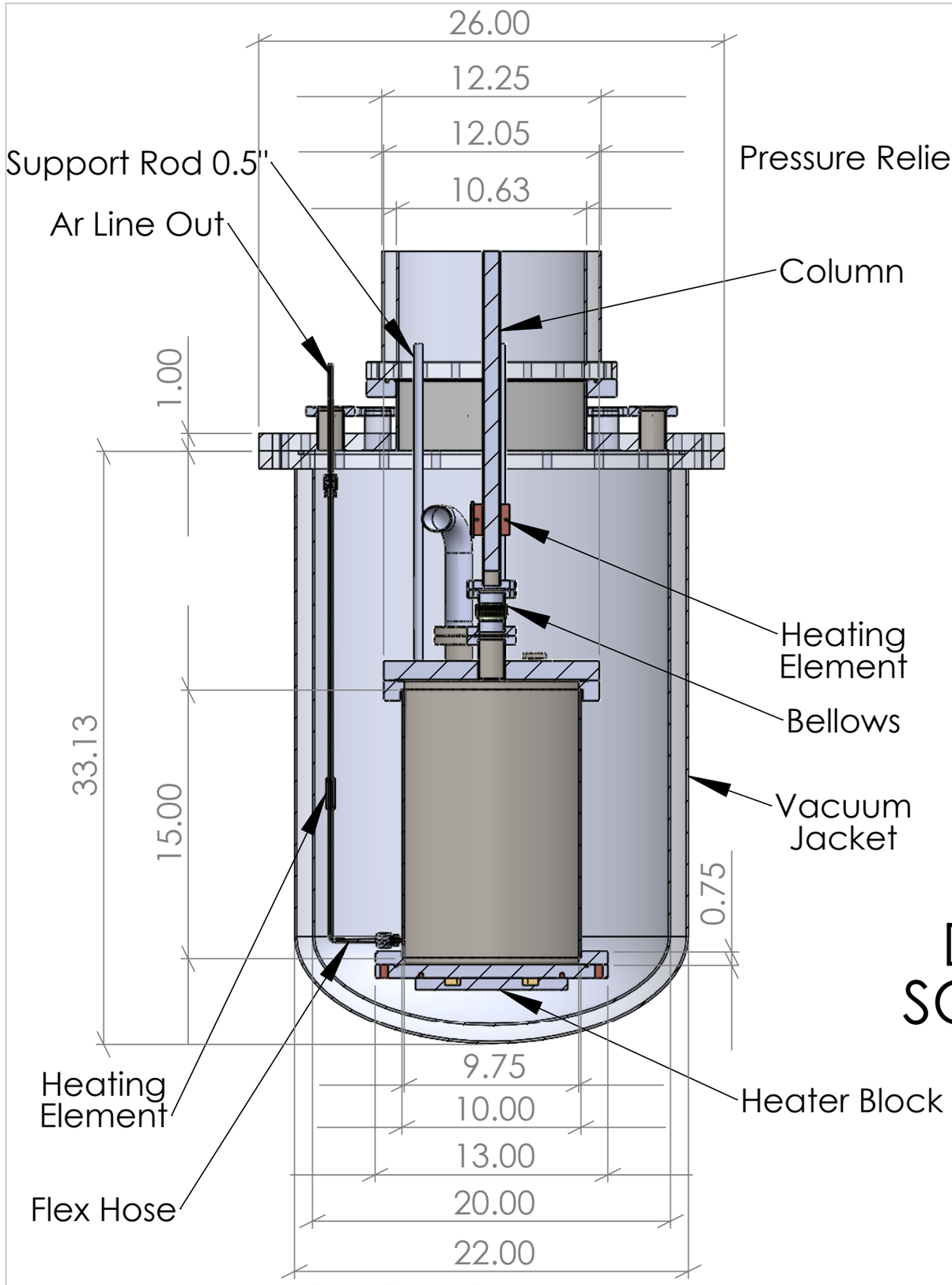
Technical Requirements

- Cooling and Heating Power:
  - Runs at ~85K
  - Bakes out at ~370K
- Thermal stress and deformation
- Fine Temperature Control
- Good Insulation to reduce heat loss
- **Air-free system (Depletion)**
- **Leak tightness**: $10^{-8}\ mBar \cdot l/s$
- Maximum Flexibility (inlet concentration may vary)
- Easily tunable
- Scalability (scale up in few years)

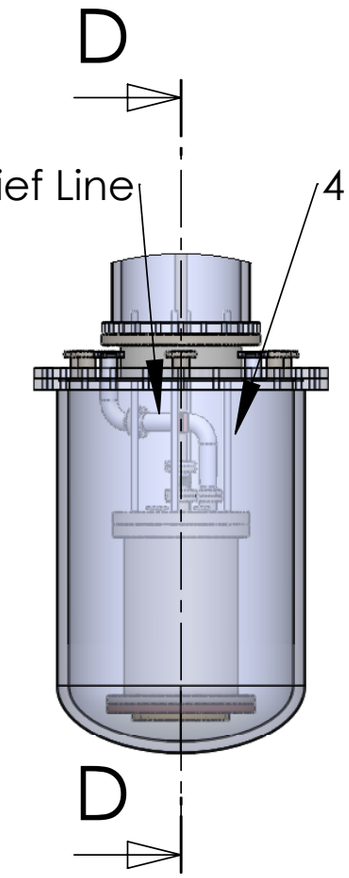
Plant

- Two 600 W cryocoolers (Condensing Volume and Condenser) coupled with heaters to finely control the temperature
- T and Mass Flow controllers
- Distillation column: 1” OD x 129” high
- 58 stages of Sulzer Laboratory Packing (0.834” OD)
- Feed: high pressure inlet cylinders
- Condenser-Booster to pressurize the gas into final storage cylinders
- Multi-inlet Mass Spectrometer to measure the composition of the gas from several important points of the plant:
  - Inlet
  - Argon product
  - Waste
- Fully automated and LabVIEW controlled

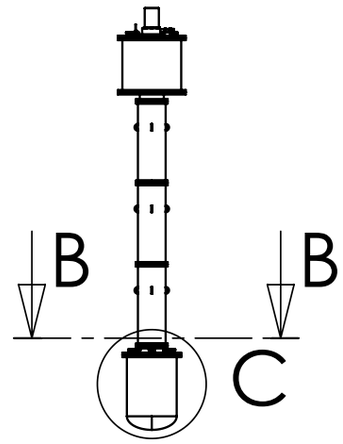

24.00
2 x AL 600 Cryocoolers
Condensing Volume
Heater Blocks
Condenser
Top Vacuum Walled Section
24.00
Vacuum Walled Section
62.50
Column
Feed
129.00
Packing
62.50
10.375
1.00
Bottom Dewar
20.00
33.13
Reboiler
A
26.00
36.00
114.00
186.38
34.13
A
SECTION A-A
SCALE 1 : 20
NAME
DATE
DRAWN
DM
08/1110
CHECKED
ENG APPR.
MFG APPR.
Q.A.
COMMENTS:
SIZE
A
DWG. NO.
Cryostripping Column Revised 7 FNAL - Main
REV.
7
SCALE:1:50
WEIGHT:
SHEET 1 OF 1

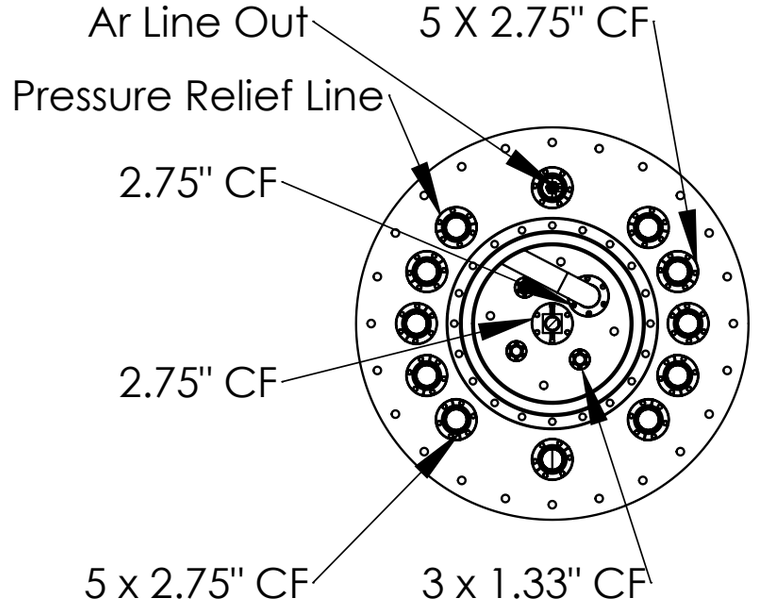


SECTION D-D
SCALE 1 : 10



DETAIL C
SCALE 1 : 20





SECTION B-B
SCALE 1 : 15

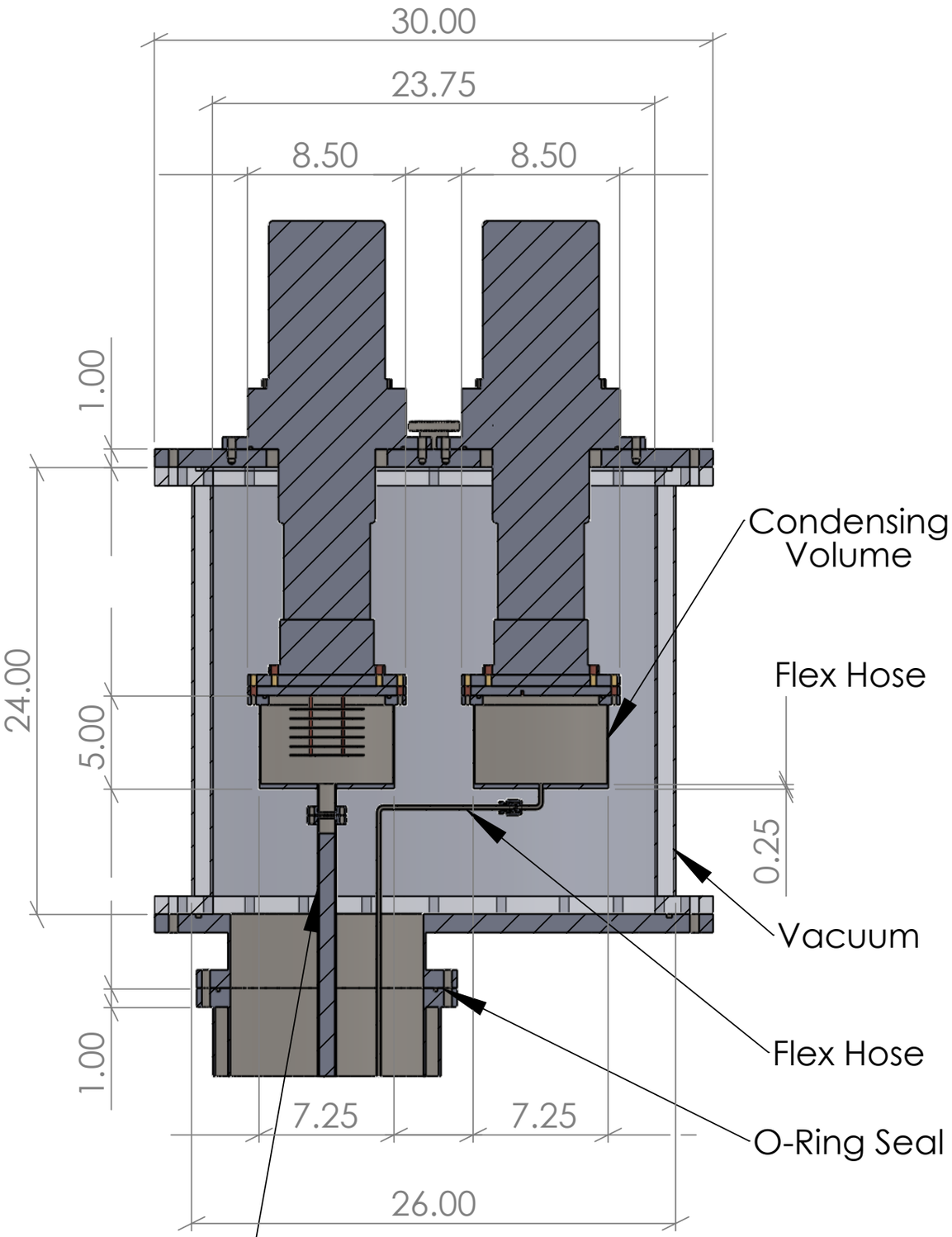
	NAME	DATE
DRAWN	DM	09/07/10
CHECKED		
ENG APPR.		
MFG APPR.		
Q.A.		
COMMENTS:		

TITLE:

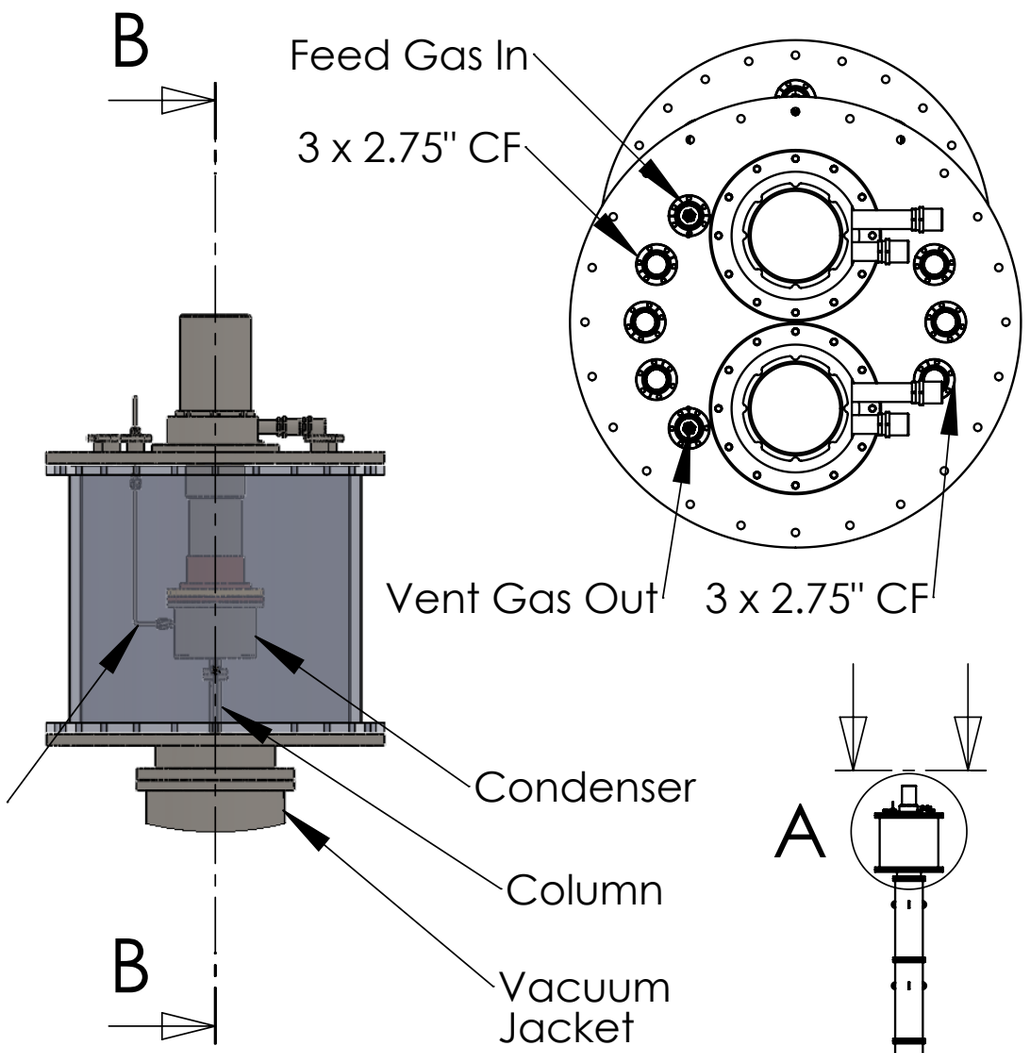
SIZE	DWG. NO.	REV
A	Cryostripping Column Revised 9 FNAL - Top	9

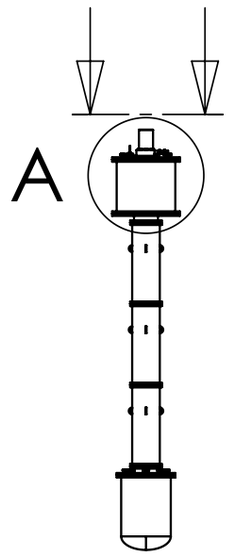
SCALE: 1:100 WEIGHT: SHEET 1 OF 1

5 4 3 2 1



SECTION B-B
SCALE 1 : 10





DETAIL A
SCALE 1 : 20

	NAME	DATE
DRAWN	DM	08/11/10
CHECKED		
ENG APPR.		
MFG APPR.		
Q.A.		
COMMENTS:		

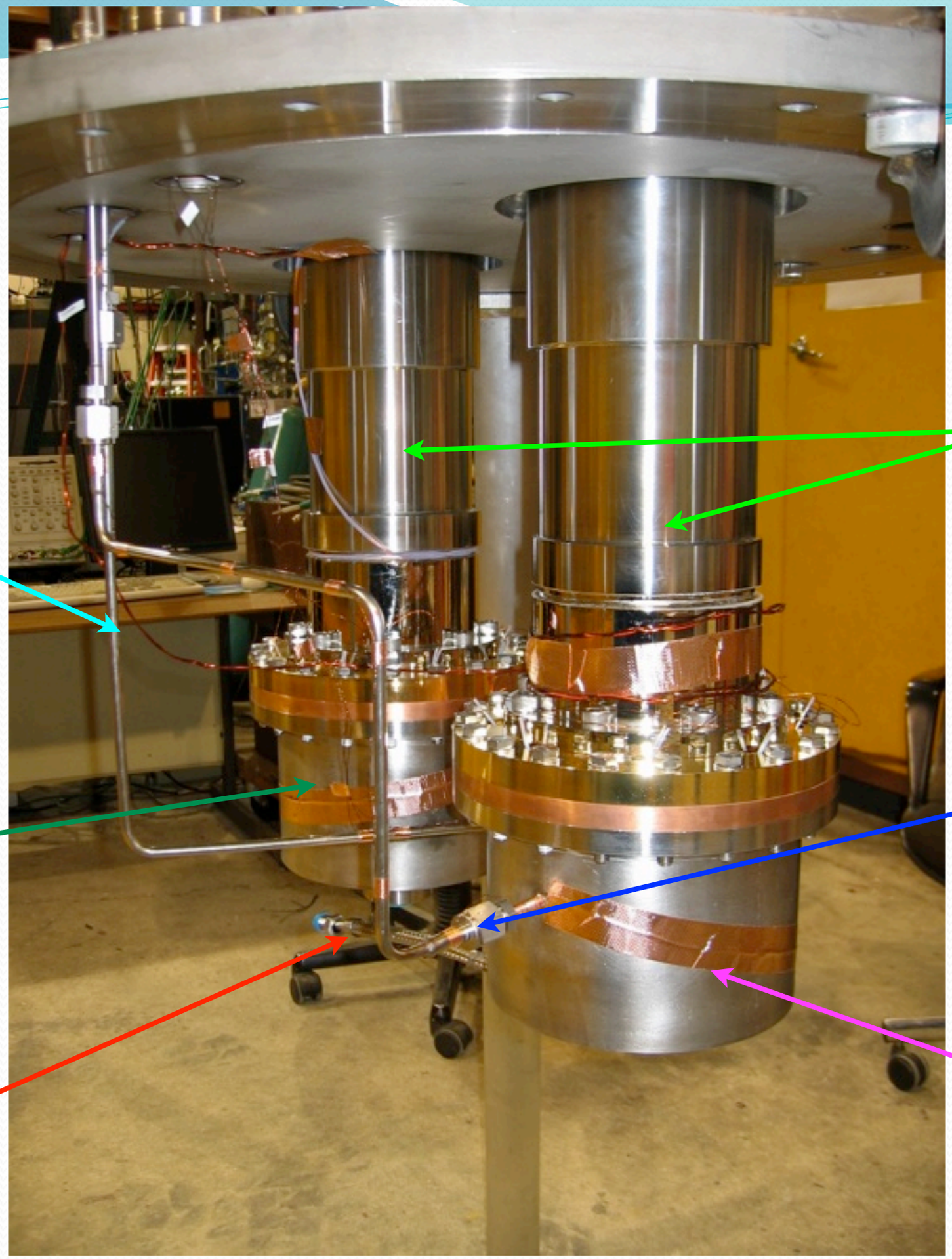
TITLE:

SIZE	DWG. NO.	REV
A	Cryostripping Column Revised 8 FNAL - Top	7

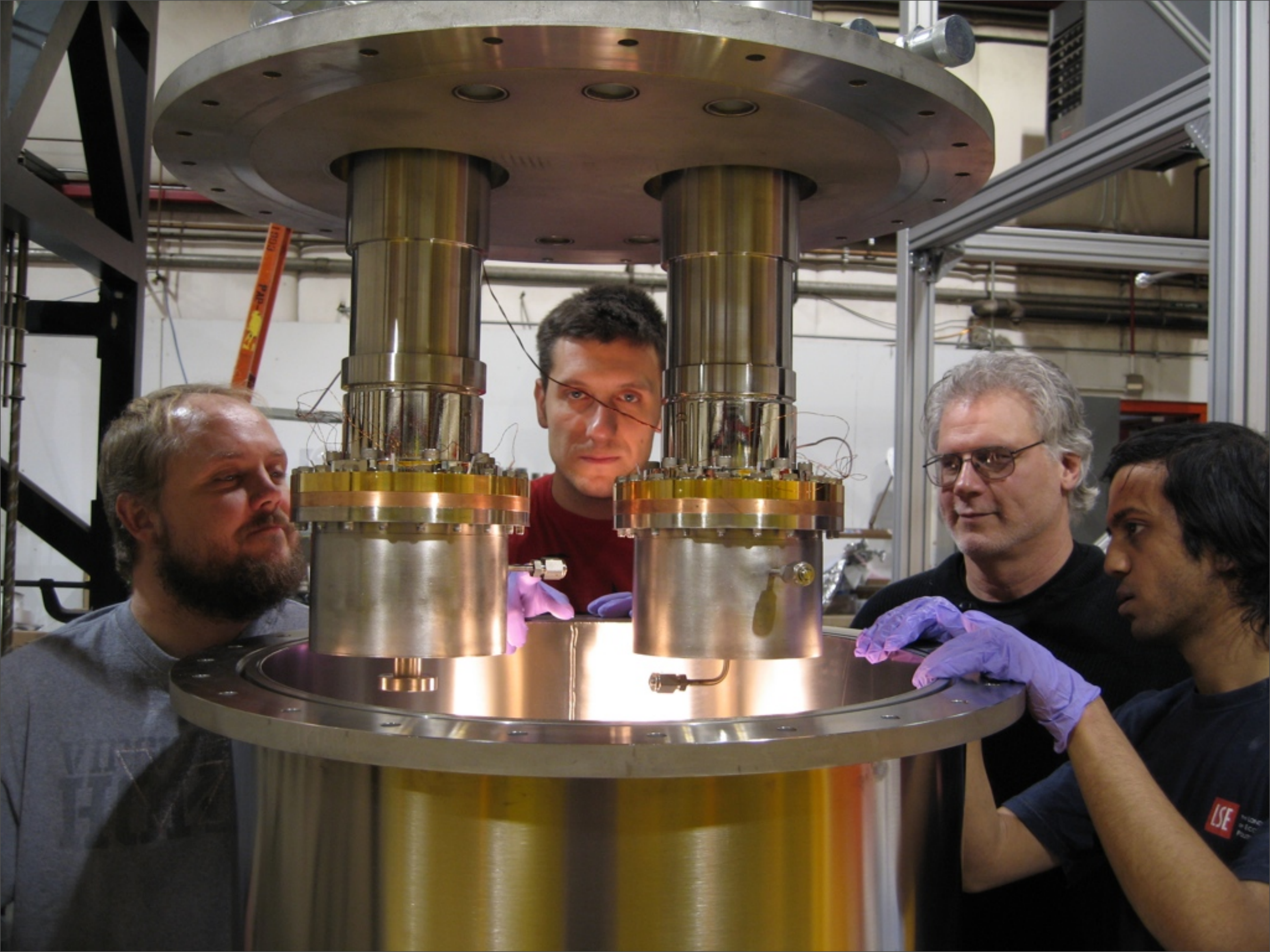
SCALE: 1:100 WEIGHT: SHEET 1 OF 1

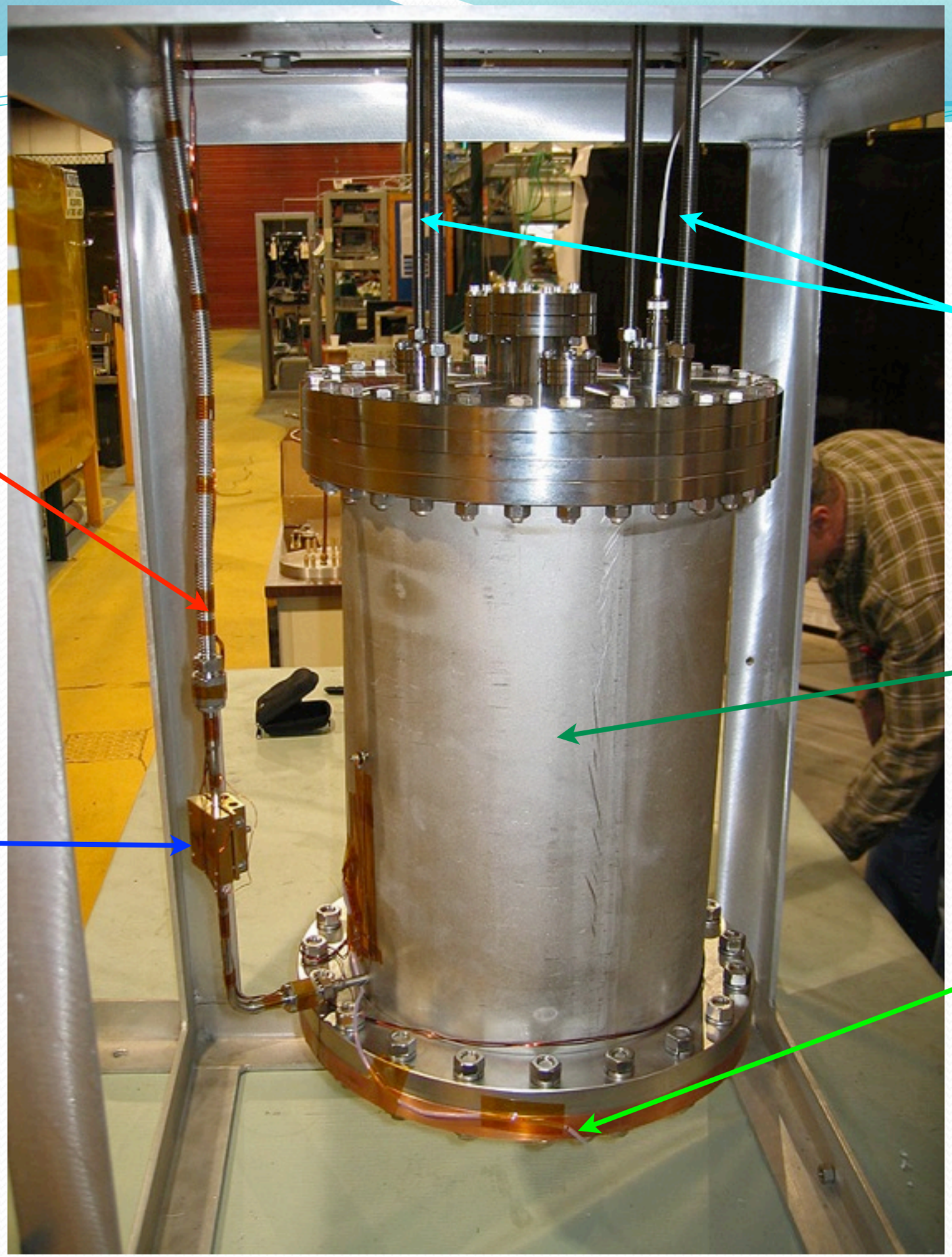
5 4 3 2 1

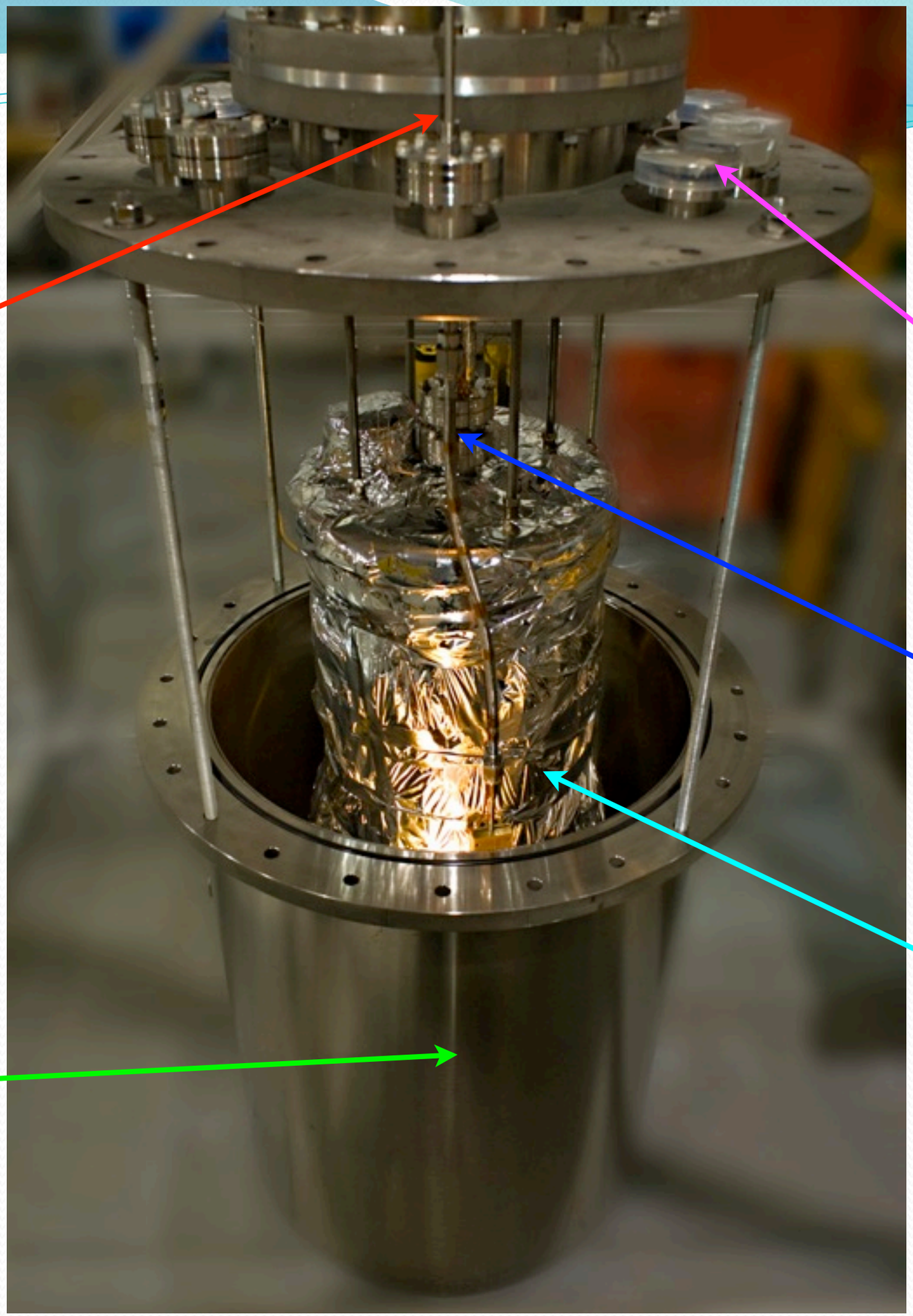
PICS


Cold Heads
Vent
Condenser
Inlet
Condensing Volume
Feed

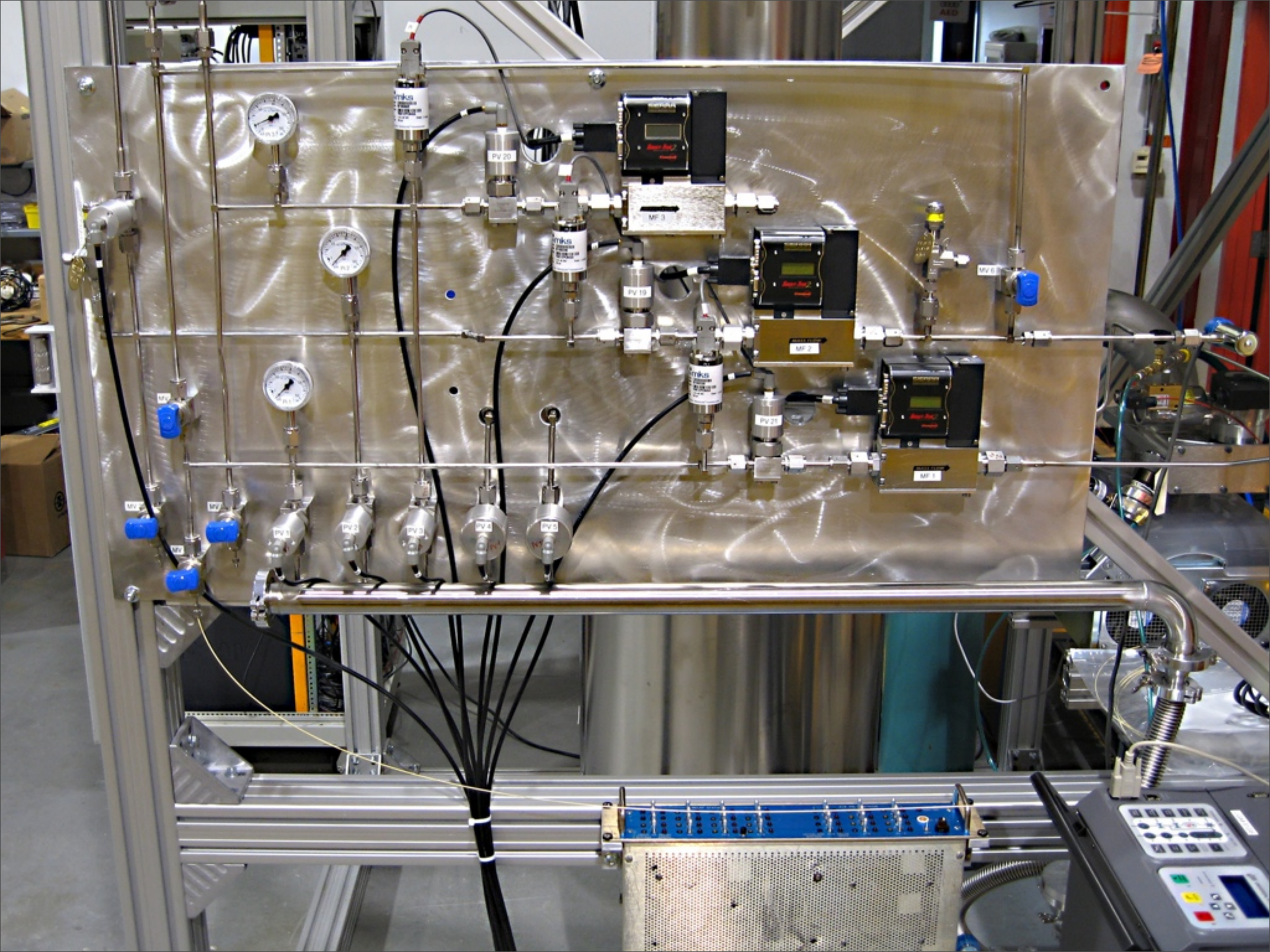
CONDENSER
CRYOMECH
CONDENSING VOLUME
CRYOMECH
HIGH
HIGH
LOW




Ar line out
Reboiler support rods
Reboiler
Heater
Flange with heater


Ar line out
Feedthroughs
Bellows
MLI
Bottom
Vacuum Jacket

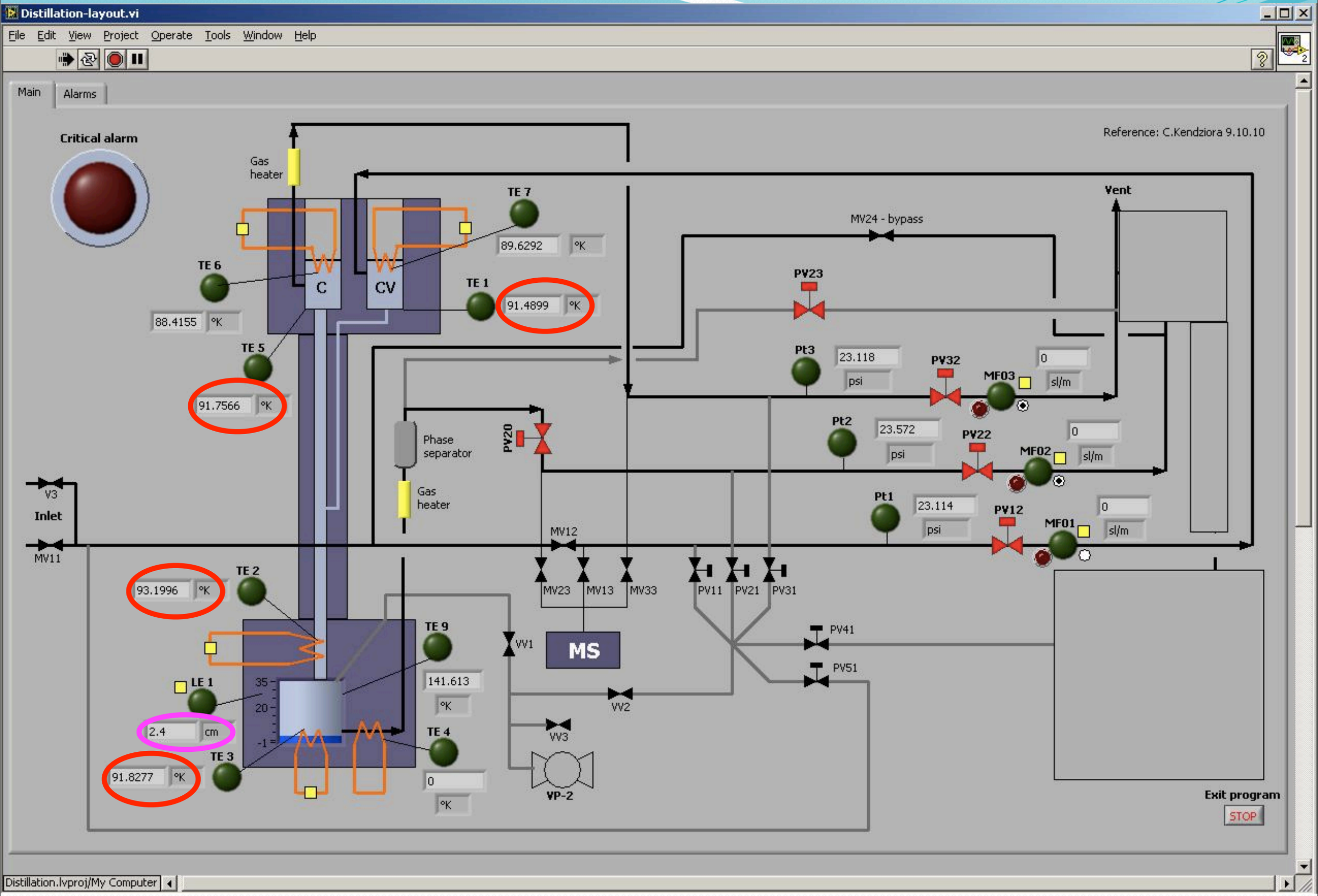
NON-FLAMMABLE GAS
2
gas

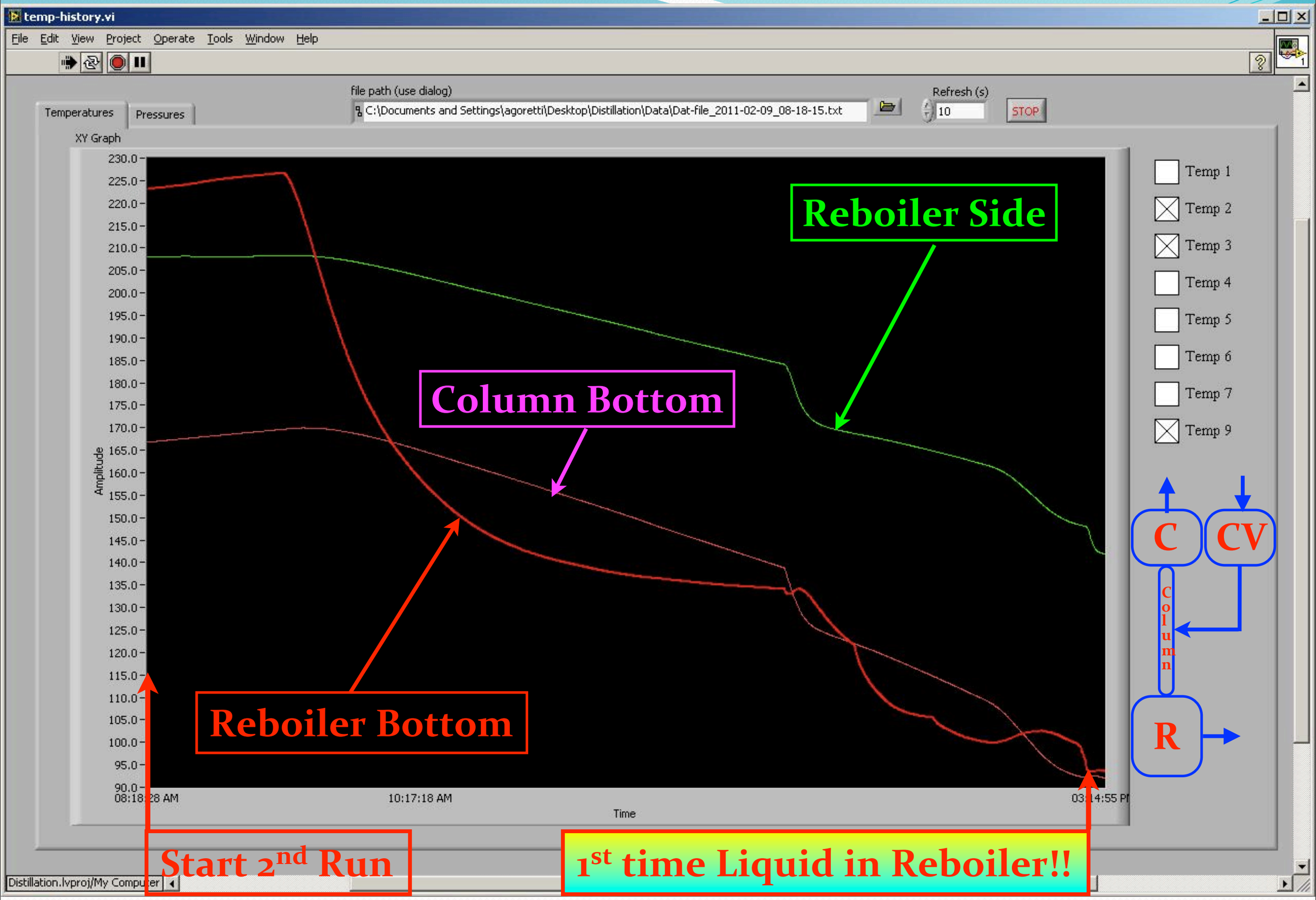

PV 20
MF 3
PV 19
MF 2
PV 21
MF 1
MV 6
PV 1
PV 2
PV 3
PV 4
PV 5

COMMISSIONING

First Commissioning

- Feed: pure Ar gas
- Temporary rotameters to set/read flow rate
- Performed two runs:
  - 12 hours
  - Turned the cryocoolers OFF overnight (8 hours) to check the behavior of the system
  - 10 hours


Distillation-layout.vi
File Edit View Project Operate Tools Window Help
Main
Alarms
Critical alarm
Reference: C.Kendziora 9.10.10
Gas heater
TE 7
89.6292 °K
TE 6
88.4155 °K
C
CV
TE 1
91.4899 °K
TE 5
91.7566 °K
Vent
MV24 - bypass
PV23
Pt3
23.118 psi
PV32
MF03
0 sl/m
Phase separator
PV20
Pt2
23.572 psi
PV22
MF02
0 sl/m
Gas heater
V3
Inlet
MV11
Pt1
23.114 psi
PV12
MF01
0 sl/m
MV12
MV23 MV13 MV33
PV11 PV21 PV31
TE 2
93.1996 °K
TE 9
141.613 °K
LE 1
2.4 cm
TE 4
0 °K
TE 3
91.8277 °K
VV1
MS
PV41
PV51
VV2
VV3
VP-2
Exit program
STOP
Distillation.lvproj/My Computer


temp-history.vi
File Edit View Project Operate Tools Window Help
file path (use dialog)
C:\Documents and Settings\agoretti\Desktop\Distillation\Data\Dat-file_2011-02-09_08-18-15.txt
Refresh (s)
10
STOP
Temperatures
Pressures
XY Graph
Amplitude
Time
08:18:28 AM
10:17:18 AM
Temp 1
Temp 2
Temp 3
Temp 4
Temp 5
Temp 6
Temp 7
Temp 9
C
CV
Column
R
Reboiler Side
Column Bottom
Reboiler Bottom
Start 2nd Run
1st time Liquid in Reboiler!!
Distillation.lvproj/My Computer

temp-history.vi

File Edit View Project Operate Tools Window Help

file path (use dialog)

C:\Documents and Settings\agoretti\Desktop\Distillation\Data\Dat-file_2011-02-09_08-18-15.txt

Refresh (s) 10 STOP

Temperatures | Pressures

XY Graph 2

Amplitude: 33.0, 32.0, 31.0, 30.0, 29.0, 28.0, 27.0, 26.0, 25.0, 24.0, 23.0, 22.0, 21.0, 20.0, 19.0, 18.0, 17.0, 16.0

Time: 08:18:28 AM, 01:01:00 PM, 03:15:38 PM

Pt 1
Pt 2
Pt 3
Pt 4

Liquid in the CV

Start 2nd Run

Distillation.lvproj/My Computer

temp-history.vi

File Edit View Project Operate Tools Window Help

file path (use dialog)

C:\Documents and Settings\agoretti\Desktop\Distillation\Data\Dat-file_2011-02-09_18-19-13.txt

Refresh (s) 10 STOP

Temperatures | Pressures

XY Graph 2

Amplitude: 41.0, 40.0, 39.0, 38.0, 37.0, 36.0, 35.0, 34.0, 33.0, 32.0, 31.0, 30.0, 29.0, 28.0, 27.0, 26.0, 25.0, 24.0, 23.0, 22.0, 21.0, 20.0, 19.0, 18.0, 17.0, 16.0, 15.0, 14.0

06:19:2[illegible] PM 11:36:40 PM 08:05:09 A[illegible]

Time

Pt 1
Pt 2
Pt 3
Pt 4

Equilibrium

Cryocoolers OFF

Distillation.lvproj/My Computer

Commissioning First Data

Variable	Set Point	Value
Feed Pressure	10 psig	**23-24 psia**
Feed Flow Rate	~5 L/m	~5 L/m
Condensing Volume T	89.5K	**±0.4K**
Condenser T	88.25K	**±0.4K**
Reboiler T	91.5-95K	**±0.7K**
Column Bottom T	91.5-95K	**±0.7K**

Commissioning First Results

- Flown: ~ 5,800 l of Ar gas
- Temperature control: tested and functioning
- Control system: complete (fine tuning in progress)
- When cryocoolers are running:
  - Pressure stays constant
  - Temperatures reach quickly the set point
- When turning cryocoolers off:
  - Pressure stays constant
  - Temperatures rise in the top part
  - Gas boils off and it is vented from the top
  - Reboiler stays cold (LAr temperature)

What's next

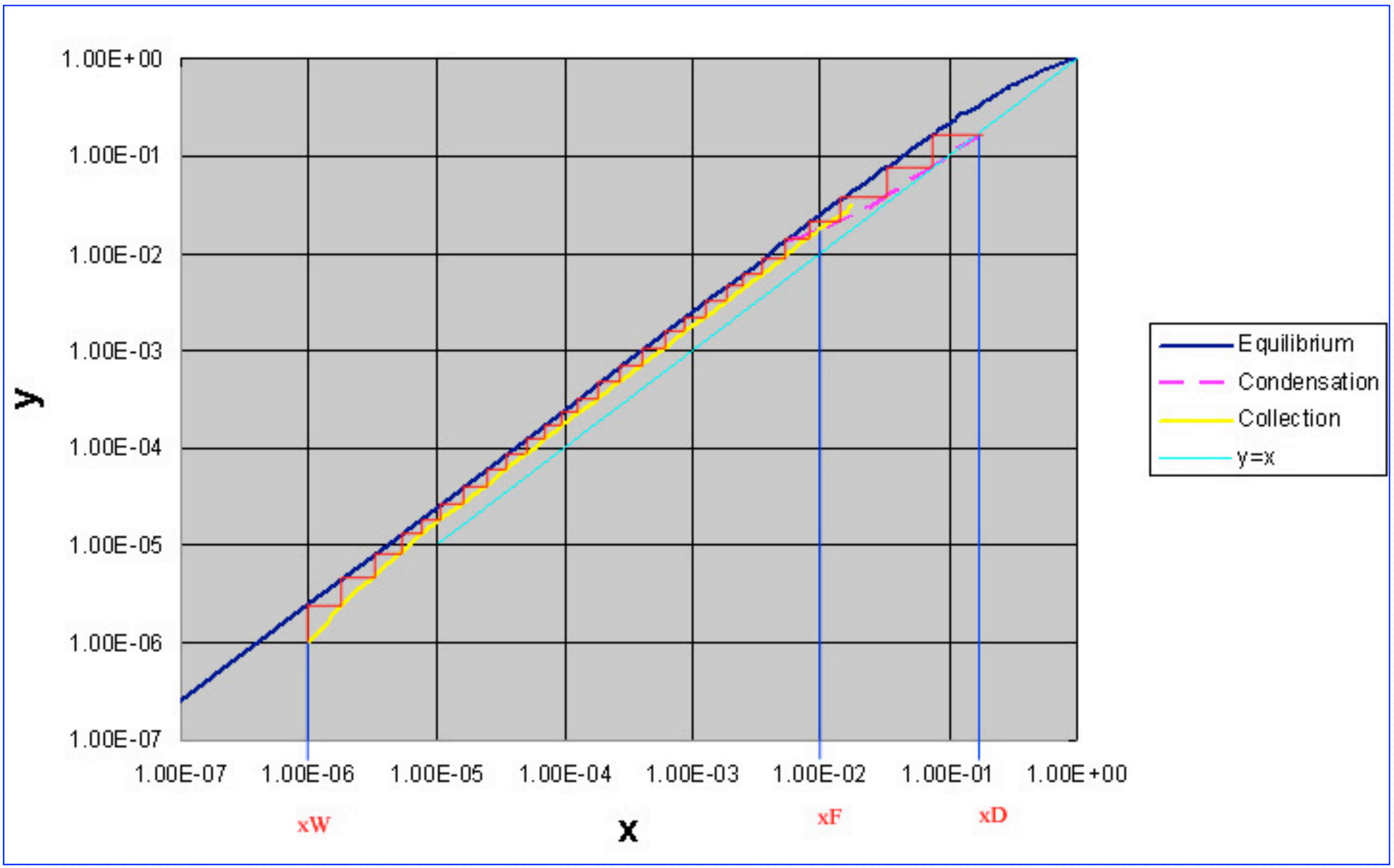
- Fine tuning of PID loops
- Control system fine tuning
- Cold flow control with Mass flow controller
- Commissioning with artificial mixture of regular gases
- Purification of 120 kg of Depleted Argon from the Argon Extraction Plant

Cryogenic distillation plant is already sized to purify Depleted Argon for ton-scale detectors

最终

Other slides

Design - McCabe-Thiele Method



Design - UNISIM

- Same Design Parameters
- Confirmed Results
- Optimal feed: middle height of the packing

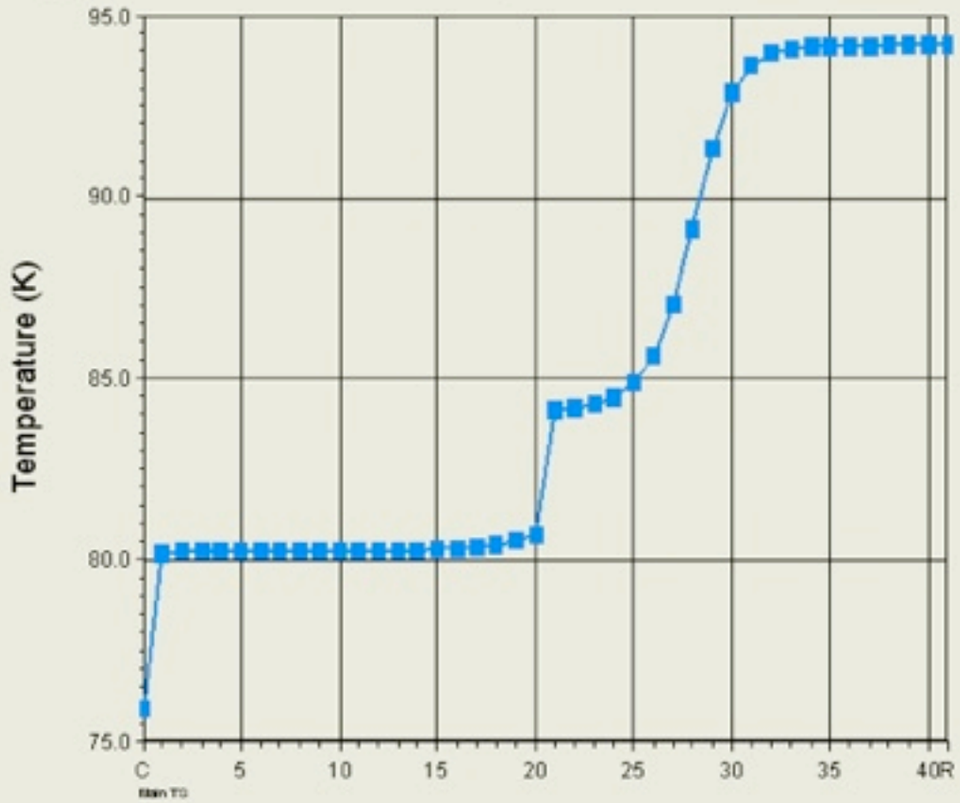
Ar+He+N2-2010-4-Mix.usc - UniSim Design R370.6

File Edit Simulation Flowsheet Tools Window Help

Environment: Case (Main)
Mode: Steady State

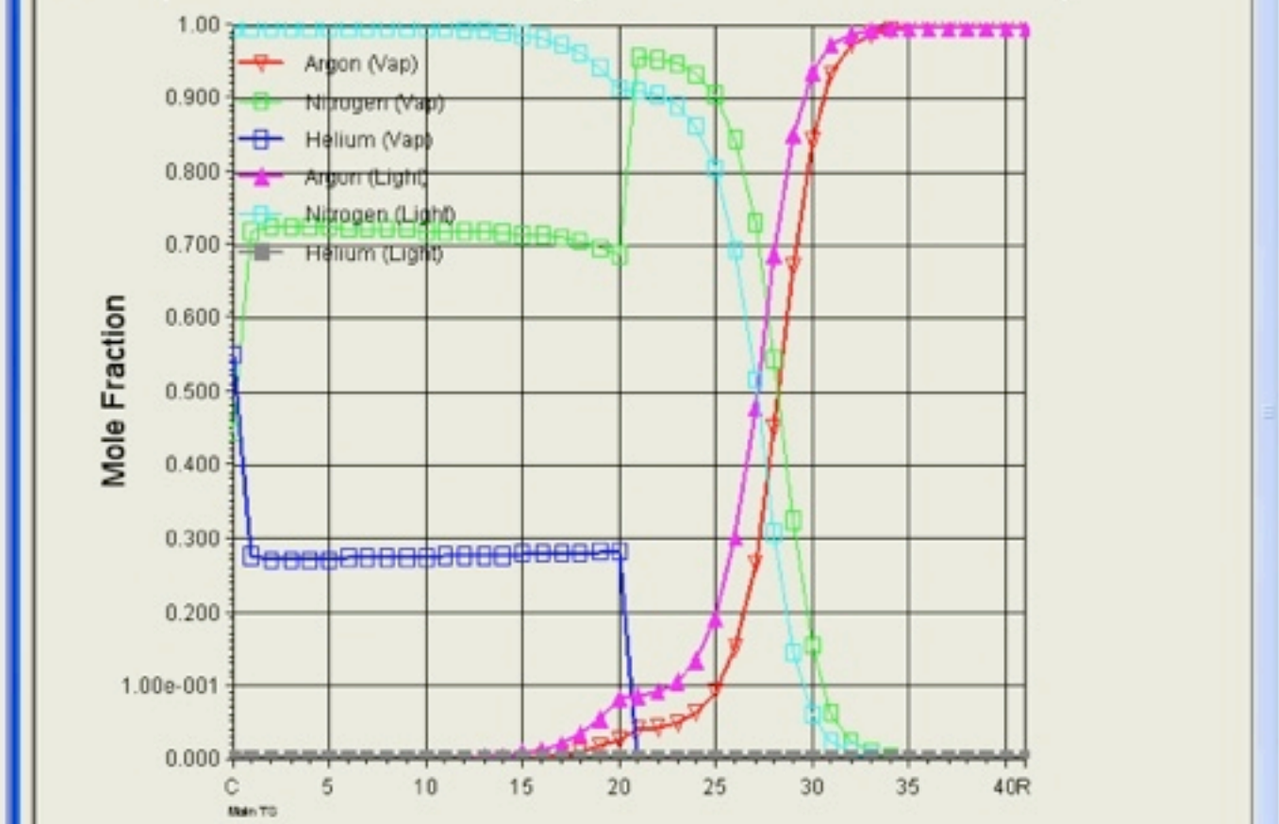
Temperature Profile

Temperature vs. Tray Position from Top



Composition Profile

Composition vs. Tray Position from Top



Properties...

Optional Info : 8 -- Unknown Flow Rate
Optional Info : 8 -- Unknown Compositions
Optional Info : 8 -- Not Solved

Saving case C:\DOCUME~1\usl028\Local Settings\Temp\AutoRecovery save of Ar+He+N2-2010-4-Mix (0x60864).abc...

Completed.

Warnings

Technical specs

- Designed following **ASME Sect. VIII Div. 1-2 (F_s = 5)**
- Seamless 304-316 stainless steel electropolished
- Custom made pressure and vacuum vessels
  - Condensing Volume and Condenser: 7.25” OD x 5” high
  - Reboiler: 10” OD x 15” high
- Custom made flanges (copper and stainless steel)
- Tube size:
  - 1/4” OD process
  - 1/16” OD peak capillary
- VCR Fittings
- ASME stamped Safety Pressure Relieving Devices
- Helium leak check
- Double wall vacuum jacket with MLI

The End