Darkside: The cryogenic distillation of underground argon (UAr)

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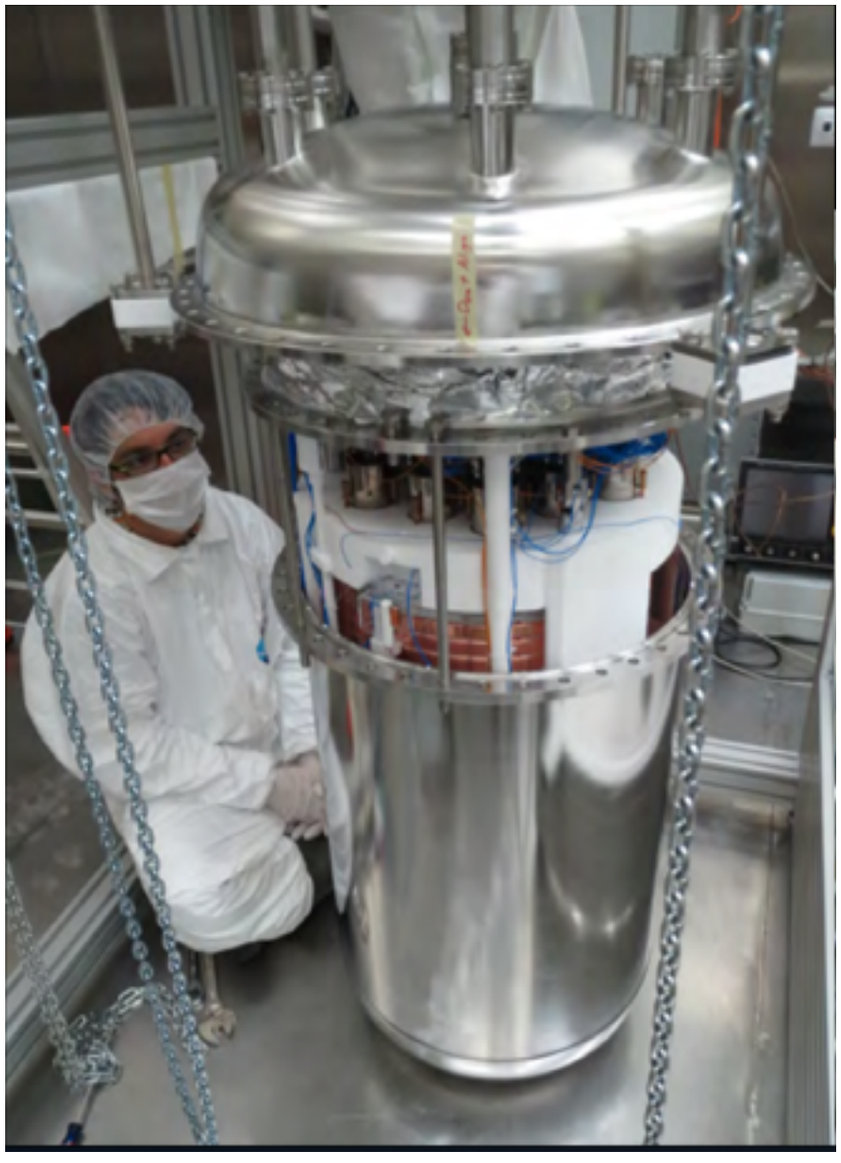
Outline

- Darkside experiment brief description
- Why underground argon?
- Underground gas extraction
- Purification plant overview
- Helium separation
- Argon recovery from waste stream
- Distillation column
- Argon nitrogen distillation
- Purification results
- Future plans and goals

DARKSIDE EXPERIMENT

- Direct dark matter searches
- 152 kg UAr total mass with 50 kg active mass
- Dual phase time projection chamber (TPC)
- currently taking data using atmospheric argon
- Gran Sasso National Laboratory



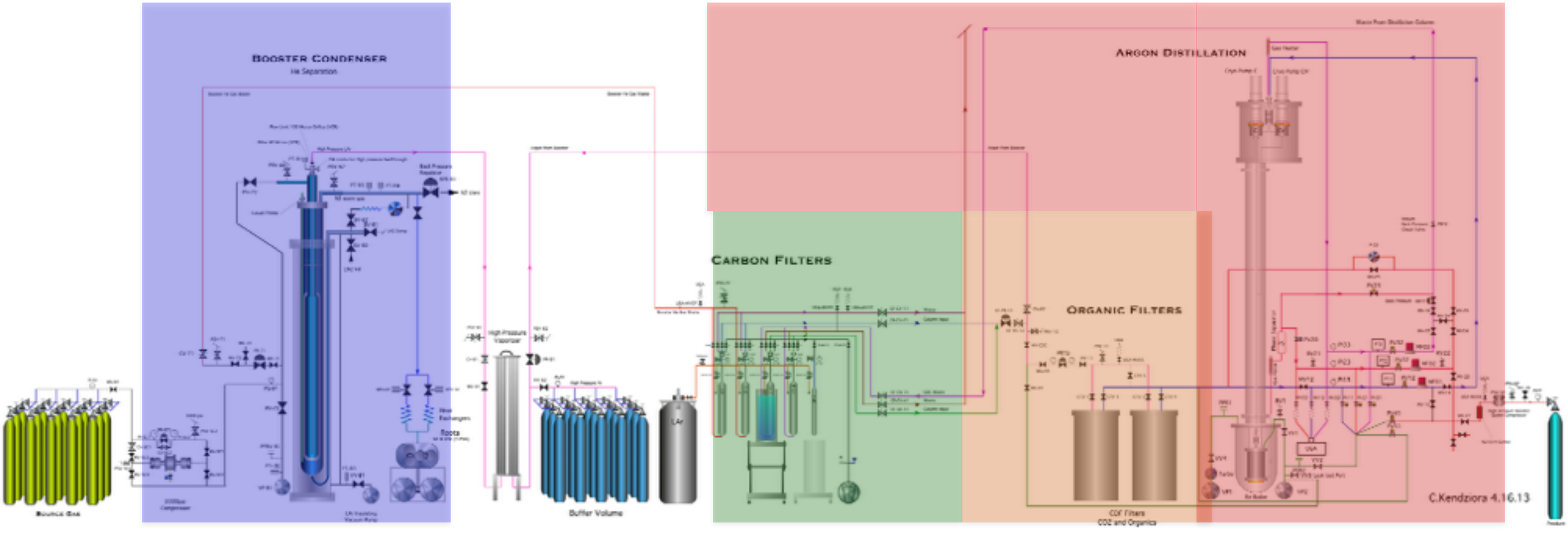
Why Underground Argon?

- Atmospheric argon contains ^{39}Ar at 1 part in 10^{15}
- ^{39}Ar undergoes beta decay
- Specific activity of 1 Bq/kg (Bq = 1 decay/sec)
- ^{39}Ar is the limiting factor for large multi-ton Ar detectors
- UAr is shielded from cosmic rays
- Naturally low in ^{39}Ar
- UAr contains at least 150x less ^{39}Ar

Underground gas extraction

- Rate of extraction ~0.3 kg/day
- Raw gas: ~96% CO_2 , N_2, He
- Contains ~300 ppm Ar
- 3-5% Argon after VPSA processing
- Ar, He, N_2 Shipped to Fermilab for purification

PURIFICATION PLANT



Helium separator

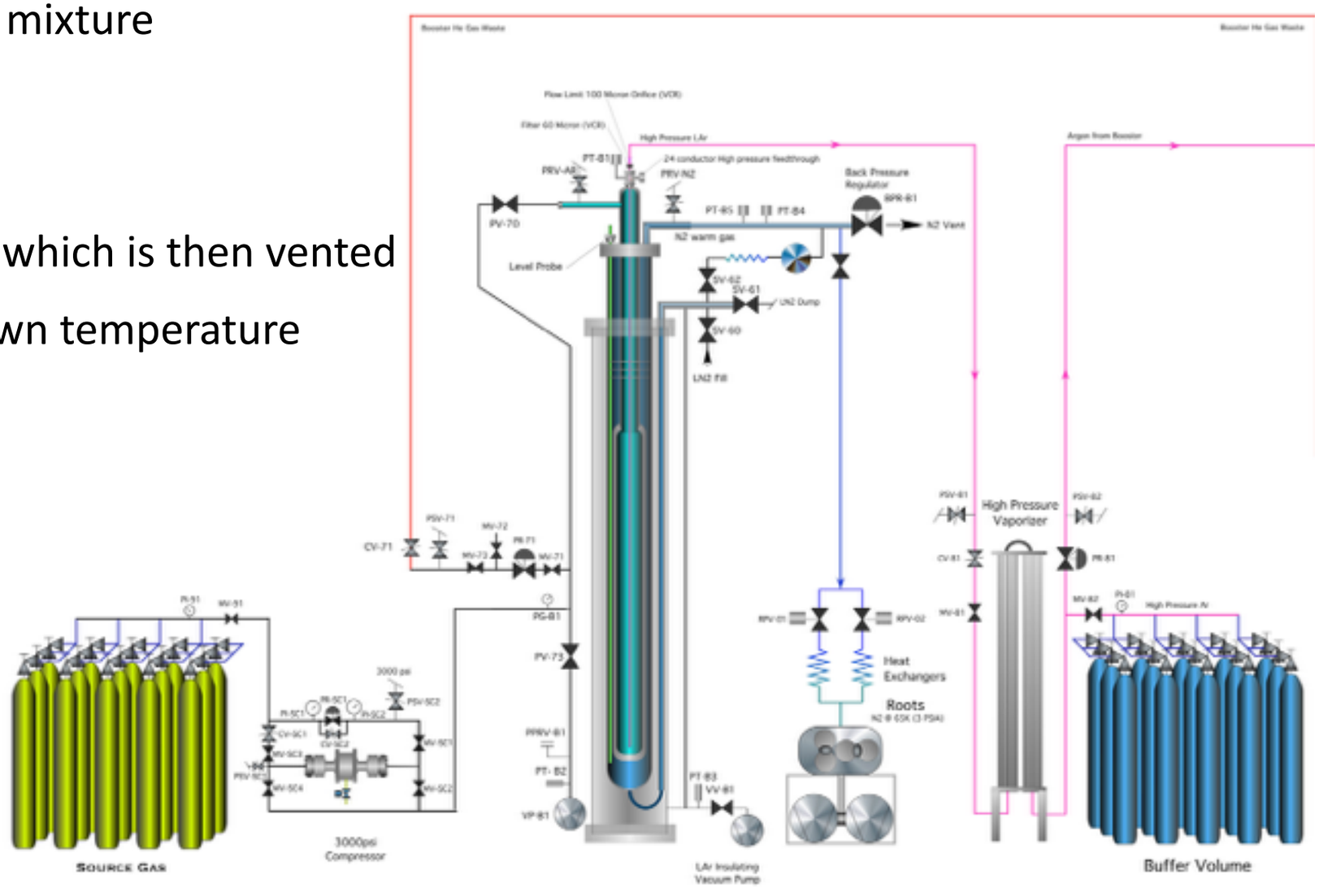
Argon recovery from waste

Residual contaminant freeze out

Distillation

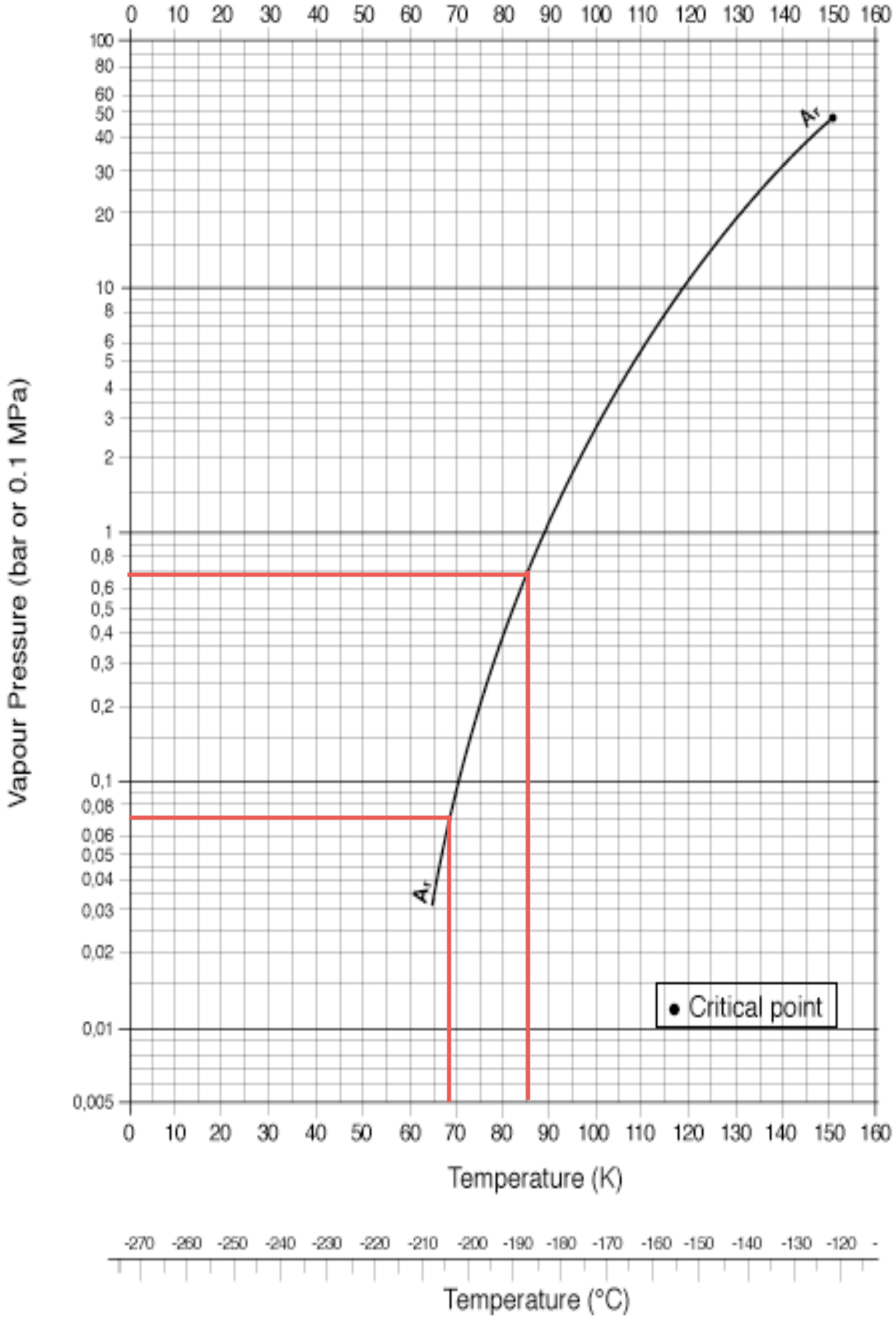
CONDENSER BOOSTER

- Vacuum insulated 20 Liter volume
- Pressurized with He-N_2-Ar mixture
- Cooled with LN_2
- Ar and N_2 condense
- Remaining pressure is He, which is then vented
- Pumping on LN_2 drives down temperature below 77 K



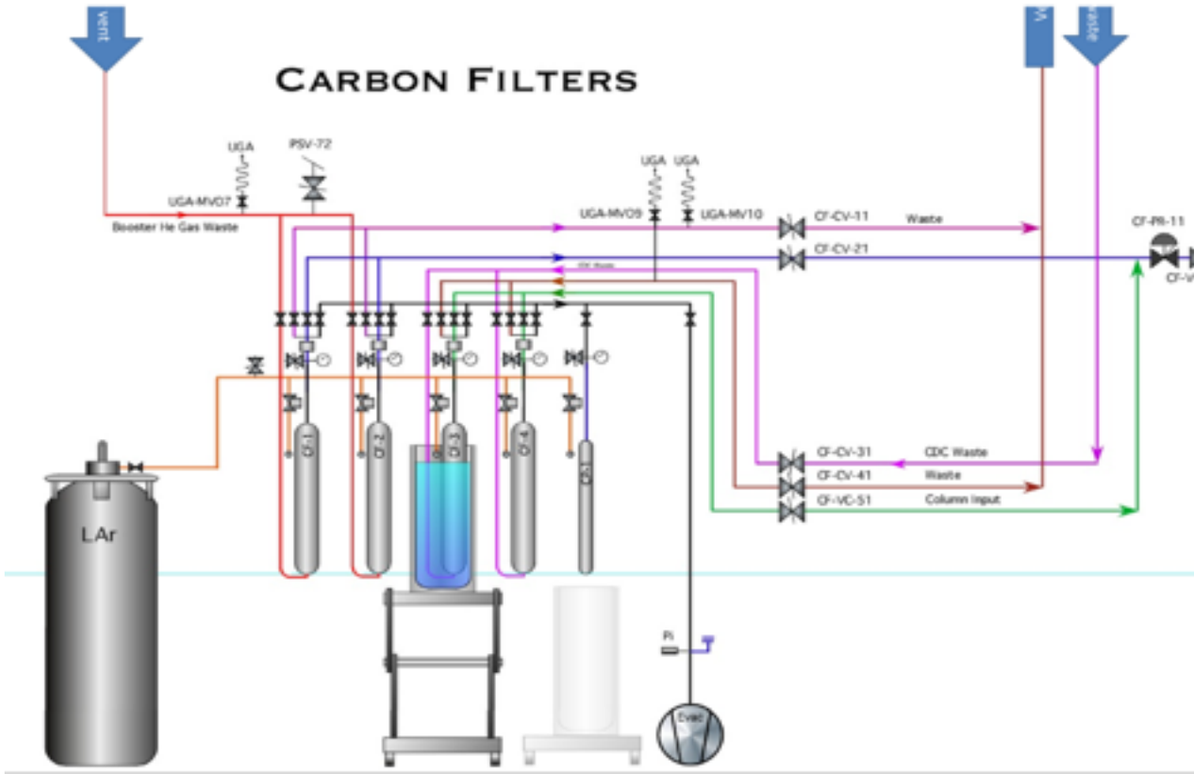
CONDENSING EFFICIENCY

- Condenser booster pressurized to 2700 psi (186 bar) = ~ 3,640 L @ stp
- LN_2 boils @77K at atm. pressure
- Operation at 85 K (2 bar)
  - Ar vapor press. = 0.7 bar
  - Ar concentration in 186 bar He = 0.38%
  - Input gas = 5% argon
  - Ar not condensed = 1.04 %
- Operation at 69 K (0.3 bar LN_2)
  - Ar vapor press. @ 69 K = 0.07 bar
  - Ar concentration in 184 bar He at 69 K = 0.038 %
  - Input gas = 5% Ar
  - Ar not condensed = 0.27 %

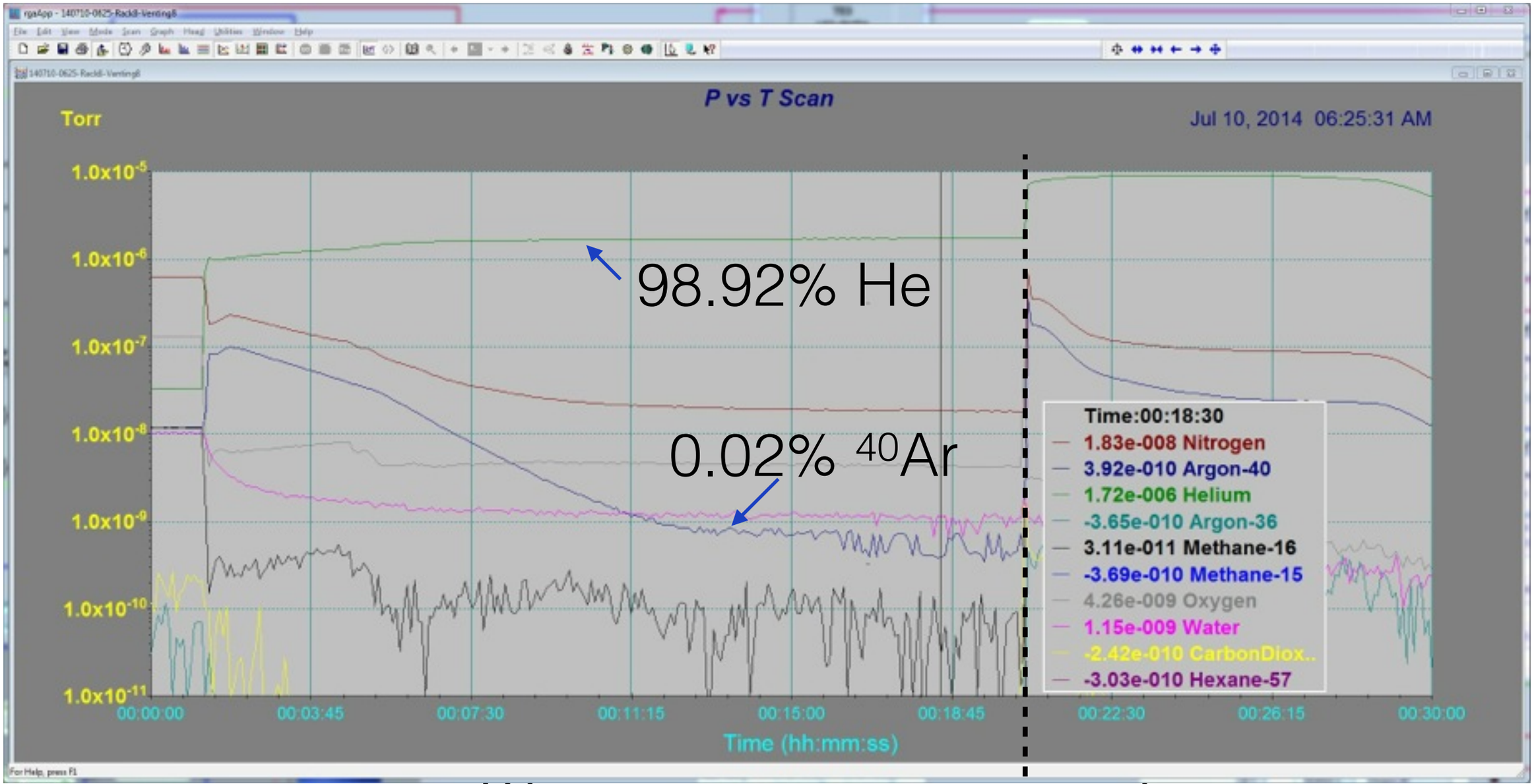


MINIMIZING LOSSES

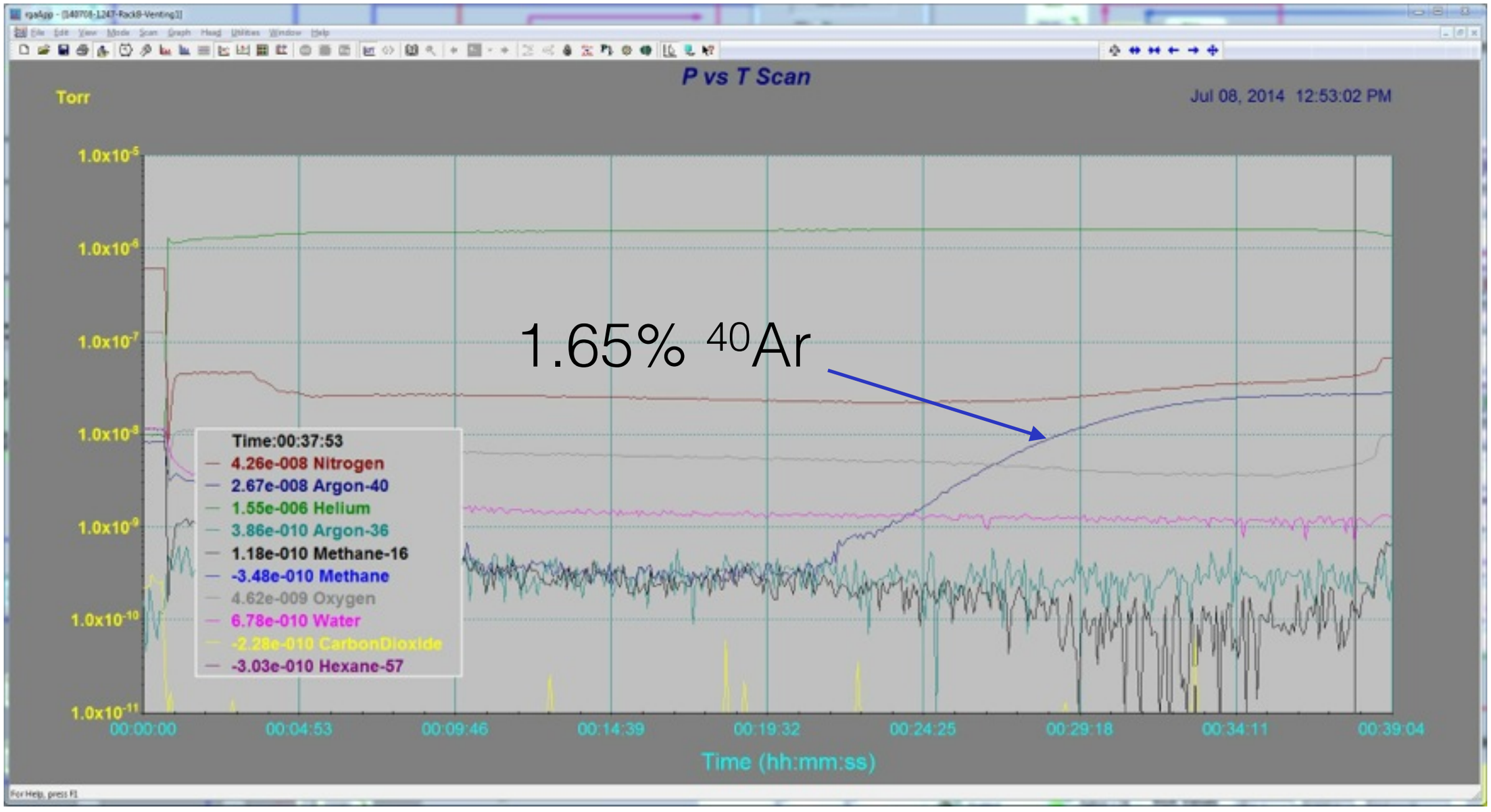
- Argon in the waste stream
- At LAr temperatures charcoal readily adsorbs argon
- Charcoal is temperature activated
- Charcoal becomes saturated



Venting the He (low Ar loss)



Venting the He (high Ar loss)



← Output from charcoal trap →

(Saturated charcoal)

Helium before and after booster

Helium concentration before the booster →

Cylinder #	fill # (1st, 2nd, ...)	Cylinder initial pressure	cylinder final pressure	pressure (psi)	Ar percentage	N2 percentage	He percentage	Ar mass (g)
714	2	0	4000	4000	4.30%	13.00%	82.70%	0.87
715	2	0	4000	4000	4.30%	12.10%	83.60%	0.87
801	2	0	3900	3900	5.30%	8.50%	86.10%	1.04
802	2	0	3900	3900	5.30%	8.50%	86.10%	1.04
803	2	0	3900	3900	5.50%	5.40%	89.10%	1.08
804	2	0	3900	3900	5.60%	6.40%	88.00%	1.10
805	2	0	3900	3900	5.80%	6.00%	88.30%	1.14
806	2	0	3900	3900	5.50%	7.60%	86.90%	1.08
807	2	0	3900	3900	5.50%	7.70%	86.80%	1.08
808	2	0	3900	3900	5.30%	8.00%	86.70%	1.04
809	2	0	3900	3900	5.40%	7.00%	87.60%	1.06
810	2	0	3900	3900	6.10%	9.10%	84.80%	1.20
811	2	0	3900	3900	5.70%	6.20%	88.20%	1.12
812	2	0	3900	3900	5.30%	6.90%	87.80%	1.04
813	2	0						0.00
814	2	0	3900	3900	6.10%	9.80%	84.00%	1.20
815	2	0	4070	4070	6.10%	9.80%	84.00%	1.25

Reduction factor ~2,167

Cylinder	N	Ar	He
#113	49.85%	49.36%	0.02%
#112	62.02%	37.90%	0.06%

Helium concentration after the booster ←

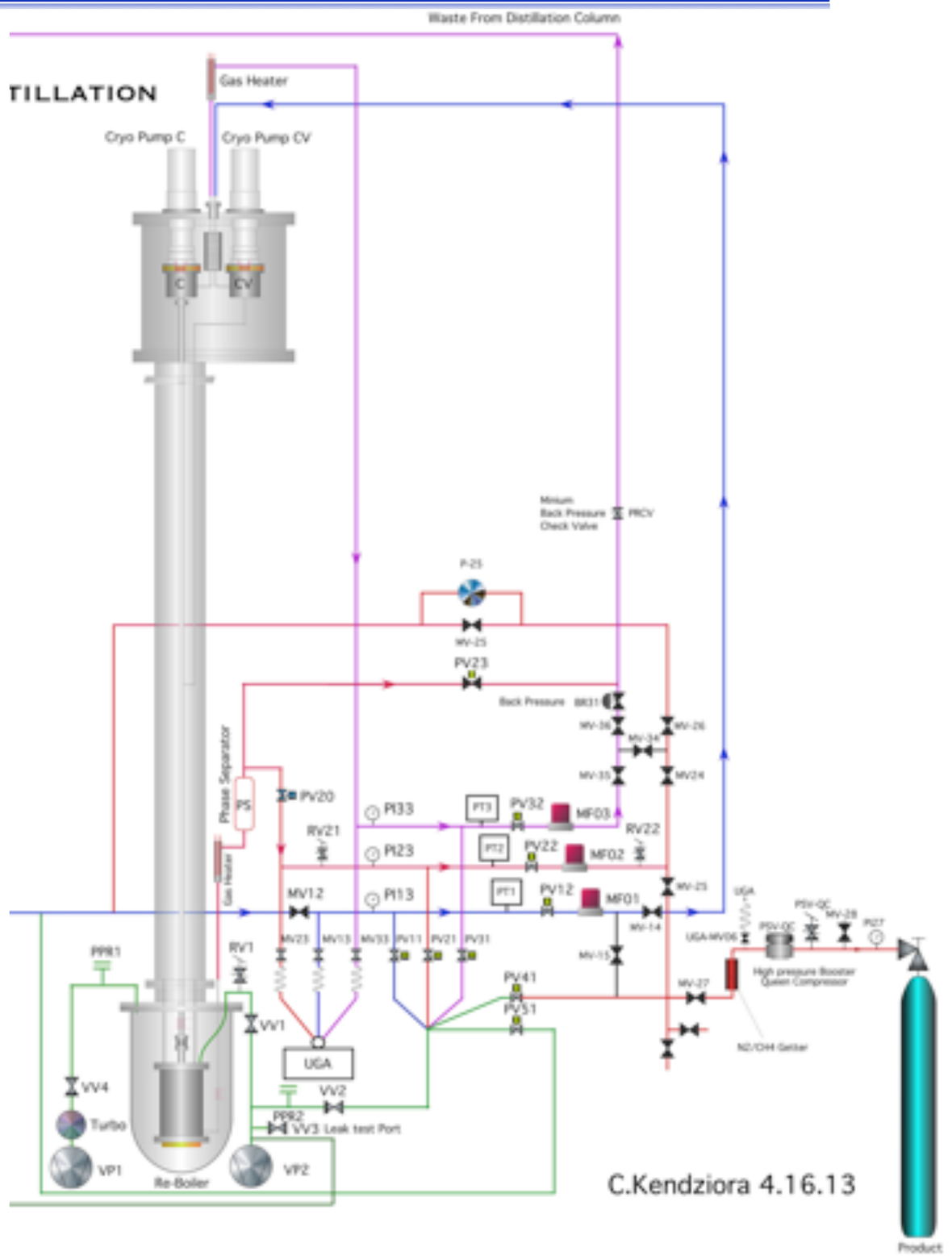
Distillation column (Ar N_2 separation)

Column Specs

- High vacuum jacket insulated
- 318 cm long stainless steel
- 56 Sulzer Chemtech packing elements
- 3 x 600 W heaters
- 2 x 600 W cryocoolers (at 77K)
- Re-boiler collects LAr
- 3 mass flow controllers (up to 20 sL/m)
- 3 UGA ports

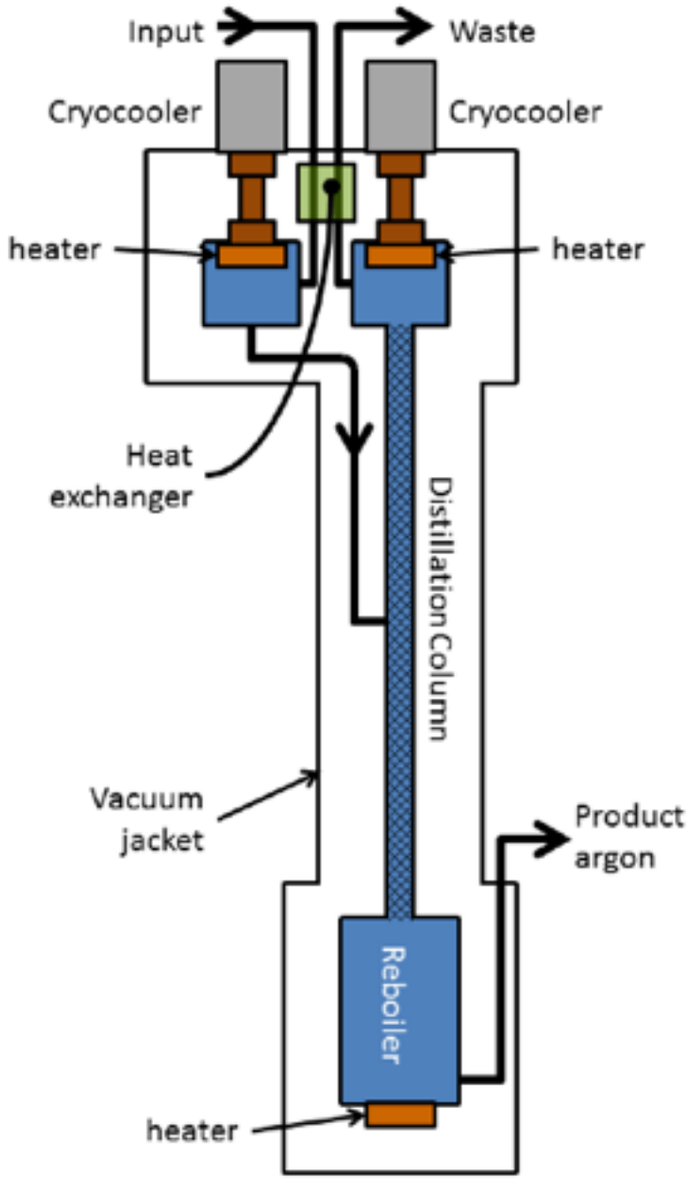
Operating Specs

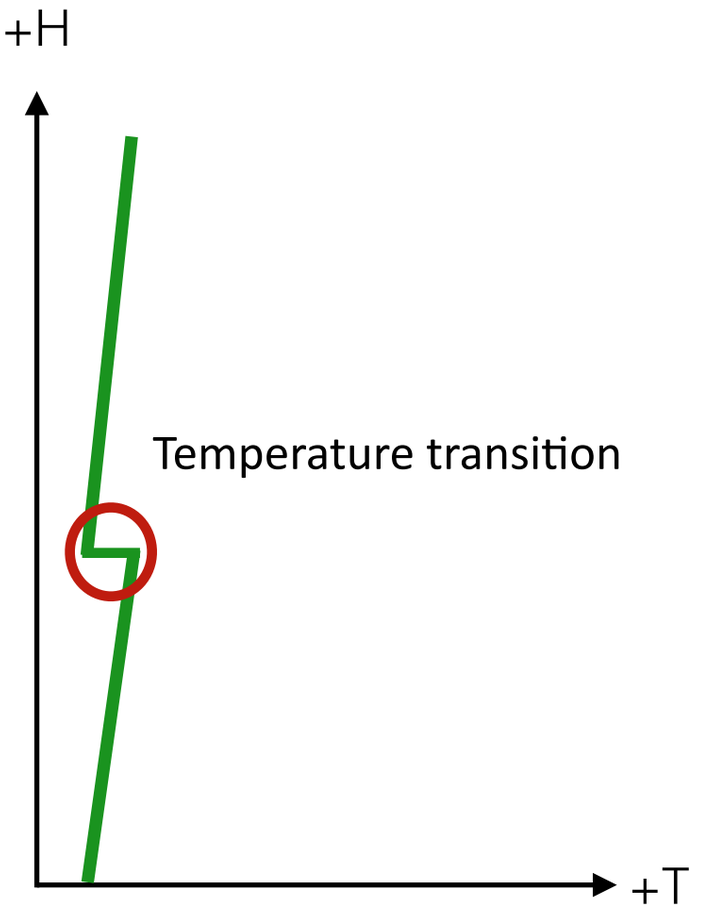
- 40-50 psia (3 - 3.5 bar)
- Condensing Volume temp. ~85 K
- Condenser temp. ~85 K
- Re-boiler temp. ~100 K



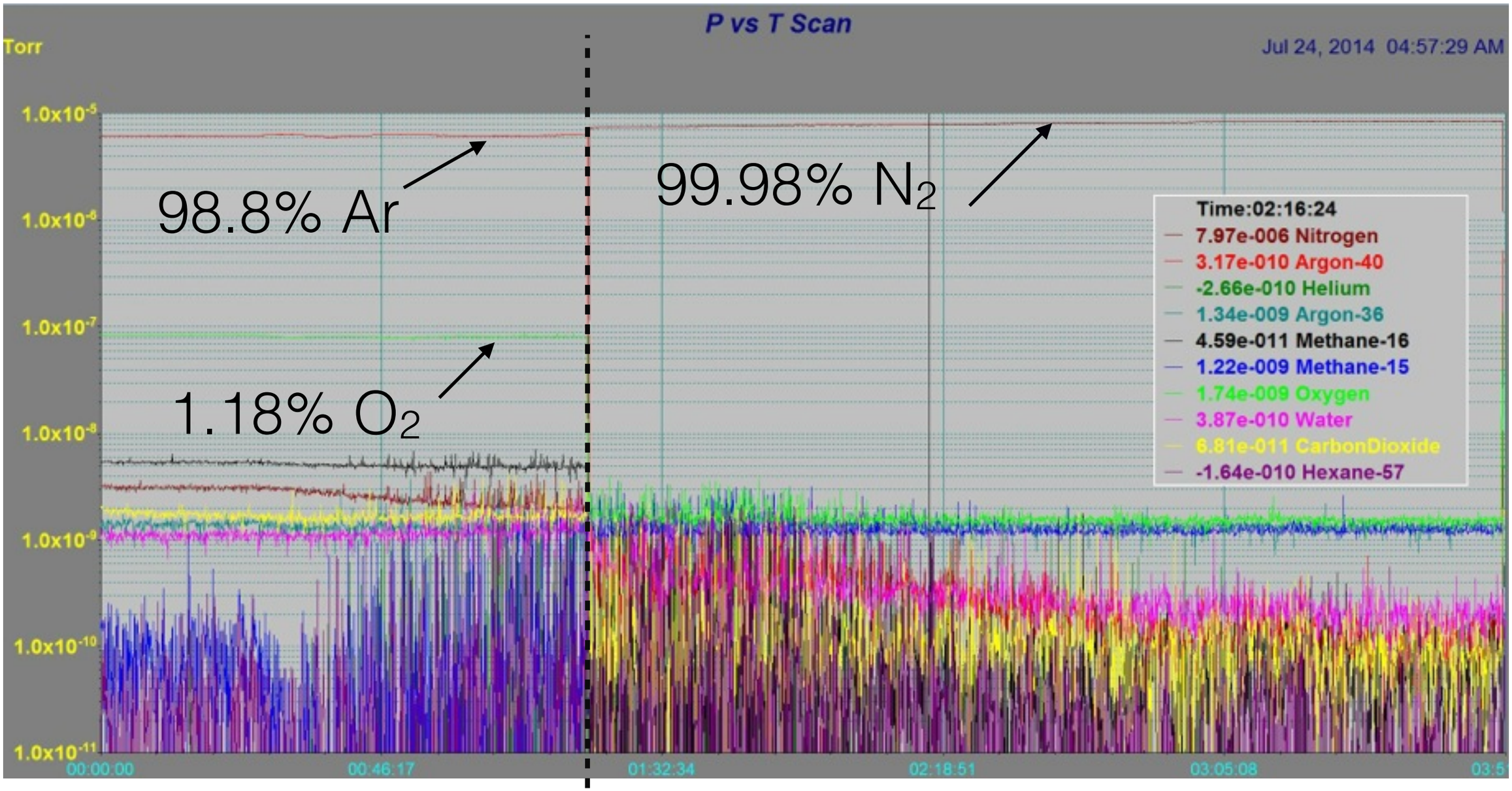
Argon nitrogen distillation

- Liquid comes in the middle of the column
- Some falls as liquid some rises as gas
- At the top nitrogen condenses while some exits
- At the bottom argon is boiling
- Heat exchange in packing elements
- Reflux = flow rate inside/ flow rate out
  - determines purity
- Temperature transition
  - behaves like a step function
  - Maintained by pressure
    - Re-boiler duty
- Equilibrium enables full reflux





PURIFICATION RESULTS



(O_2 is from underground source not from air)

RESULTS

Cylinder	Mass	Ar	N	O	CH	CO
UAr#2	*6.6 kg*	*99.97%*	*240 ppm*	*80 ppm*	*< 10 ppm*	*< 10 ppm*
UAr#3	*10.7 kg*	*99.993%*	*30 ppm*	*< 10 ppm*	*40 ppm*	*< 10 ppm*
UAr#4	*10.7 kg*	*99.917%*	*30 ppm*	*740 ppm*	*60 ppm*	*< 10 ppm*
UAr#5	*10.7 kg*	*99.944%*	*30 ppm*	*420 ppm*	*110 ppm*	*< 10 ppm*
UAr#6	*10.7 kg*	*99.956%*	*50 ppm*	*250 ppm*	*120 ppm*	*20 ppm*
UAr#7	*9.2 kg*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*
UAr#8	*8.0 kg*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*
UAr#9	*12.9 kg*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*
UAr #10	*8.1 kg*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*	*Not avail.*

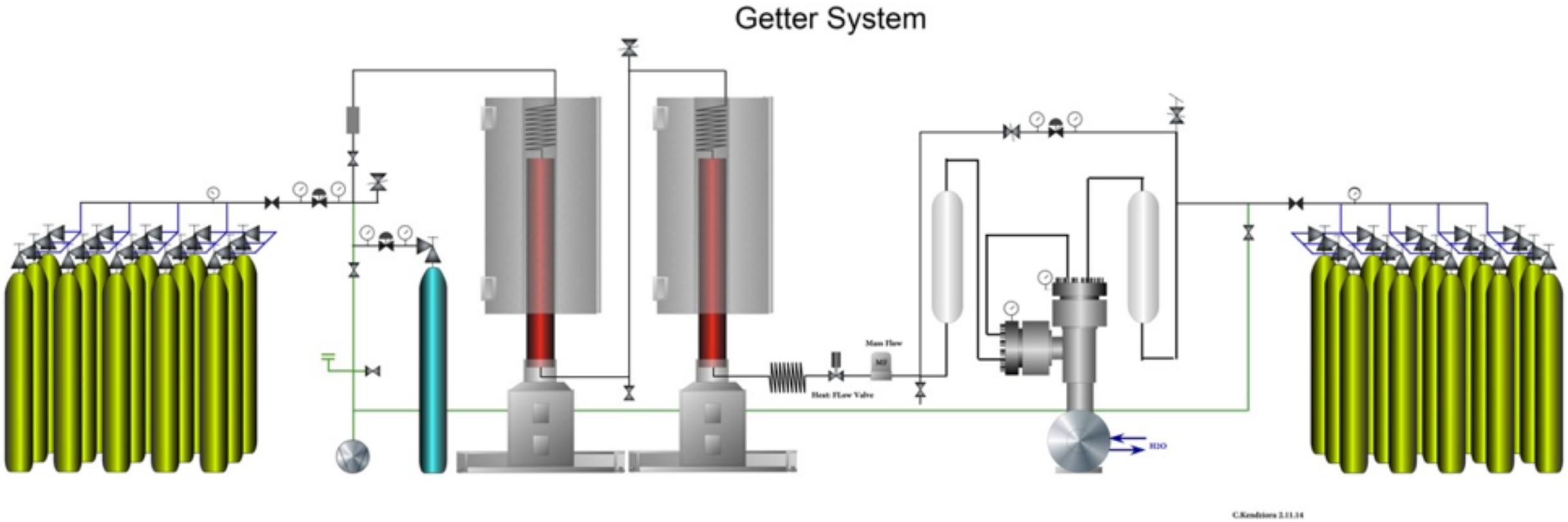
Total purified mass- 87.6 kg UAr

UAr mass with O_2 (not from air) - 21 Kg

Total mass - 136 kg

Analysis done by Pacific Northwest National Lab (PNNL)

FUTURE PLANS AND GOALS



- UAr requires final polishing to become detector grade
- Using heated Zirconium getters from SAES
- We will run all the UAr through the getters at one time
- System built, waiting for control system
- Plan to retrofit Oxygen scrubber to this plant
- Plan to reach contamination in the ppb

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A very special thank you to my mentors

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Questions?

REFERENCES

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Argon Nitrogen phase diagram

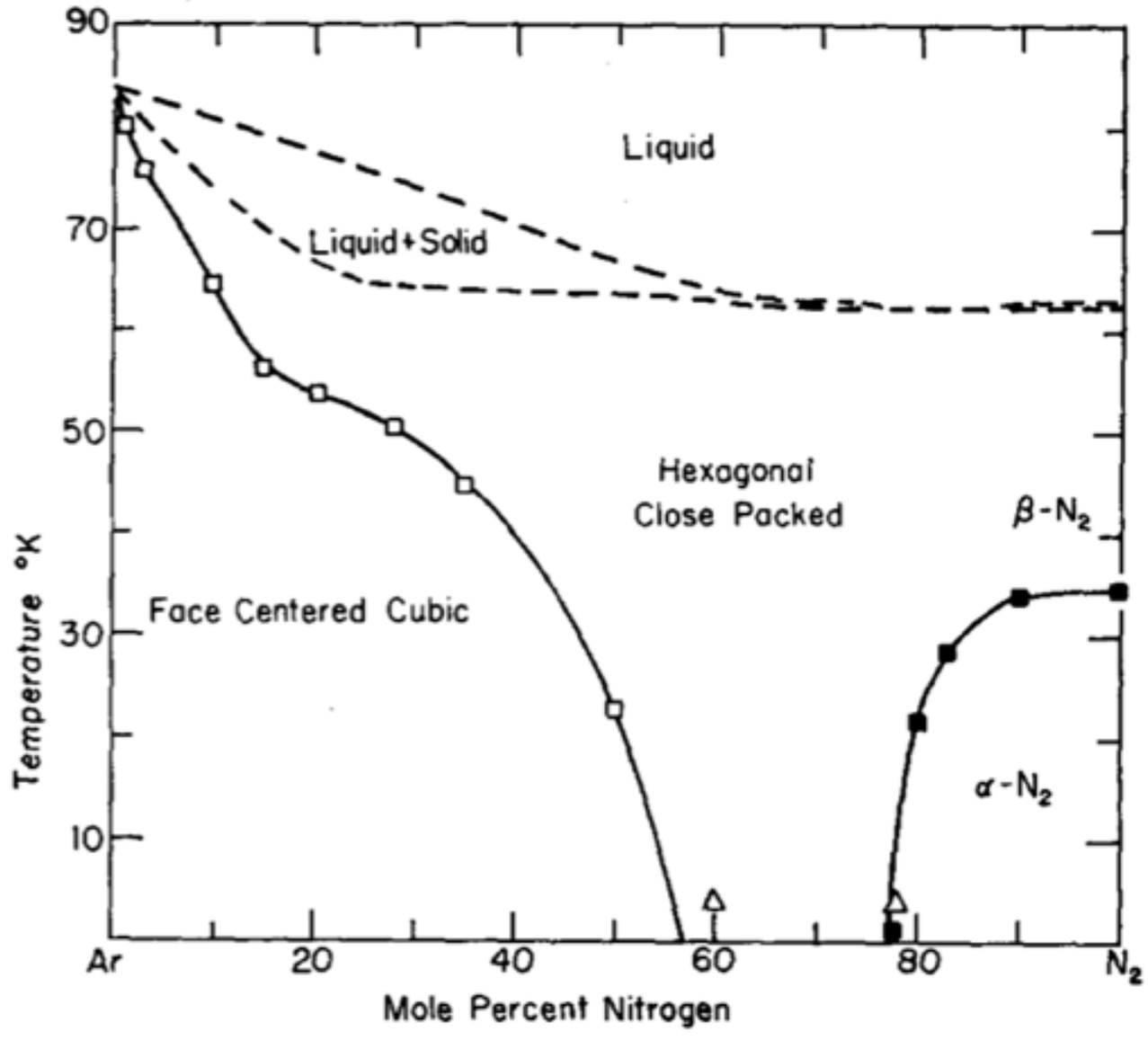


FIG. 1. Argon–nitrogen phase diagram. Open squares indicate upper limit for strain-induced martensitic transformation. Open triangles indicate no transformation detected. Filled squares indi-

Distillation column

56 x 80-mm Sulzer Chemtech packing elements

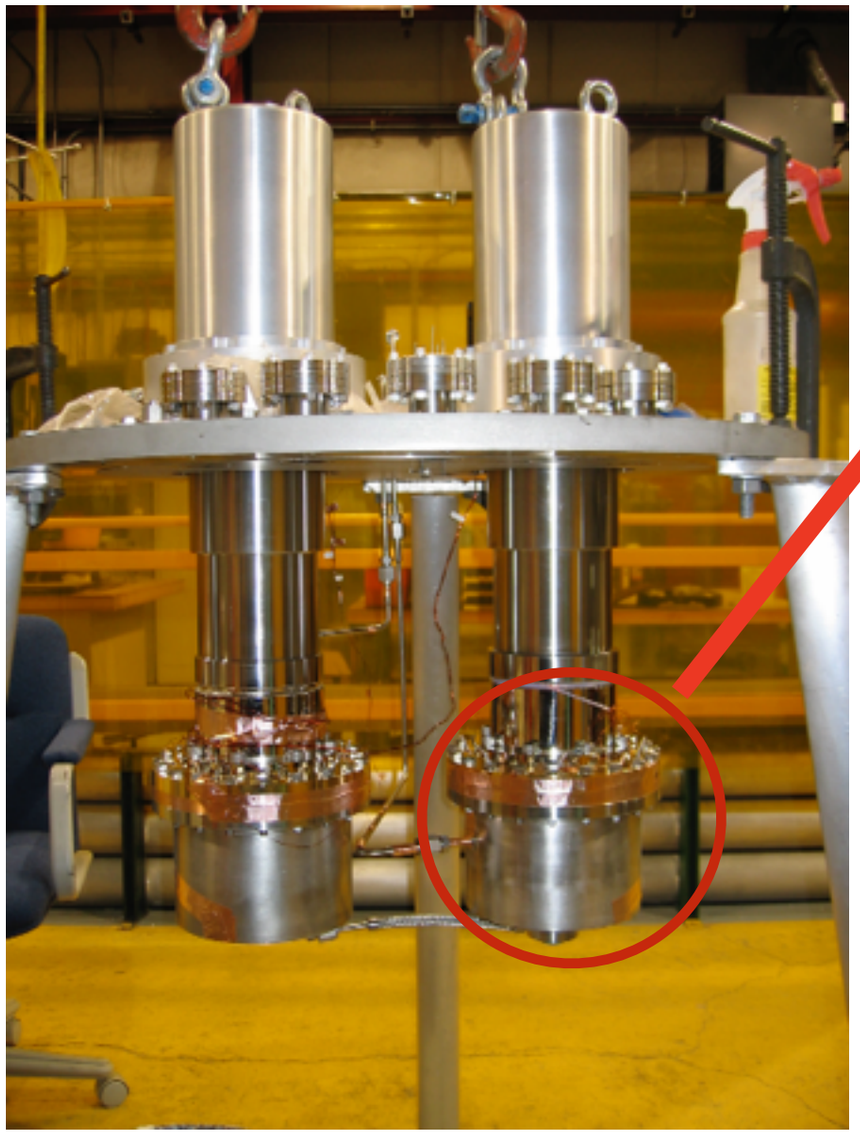
- Equivalent to 56 distillation stages

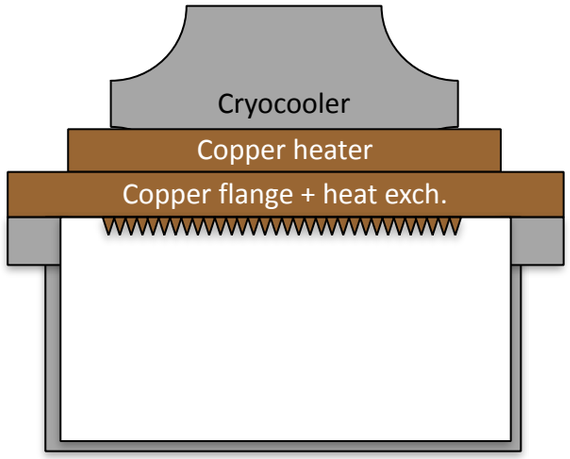
Super insulation

- minimizes heat transfer

Distillation column

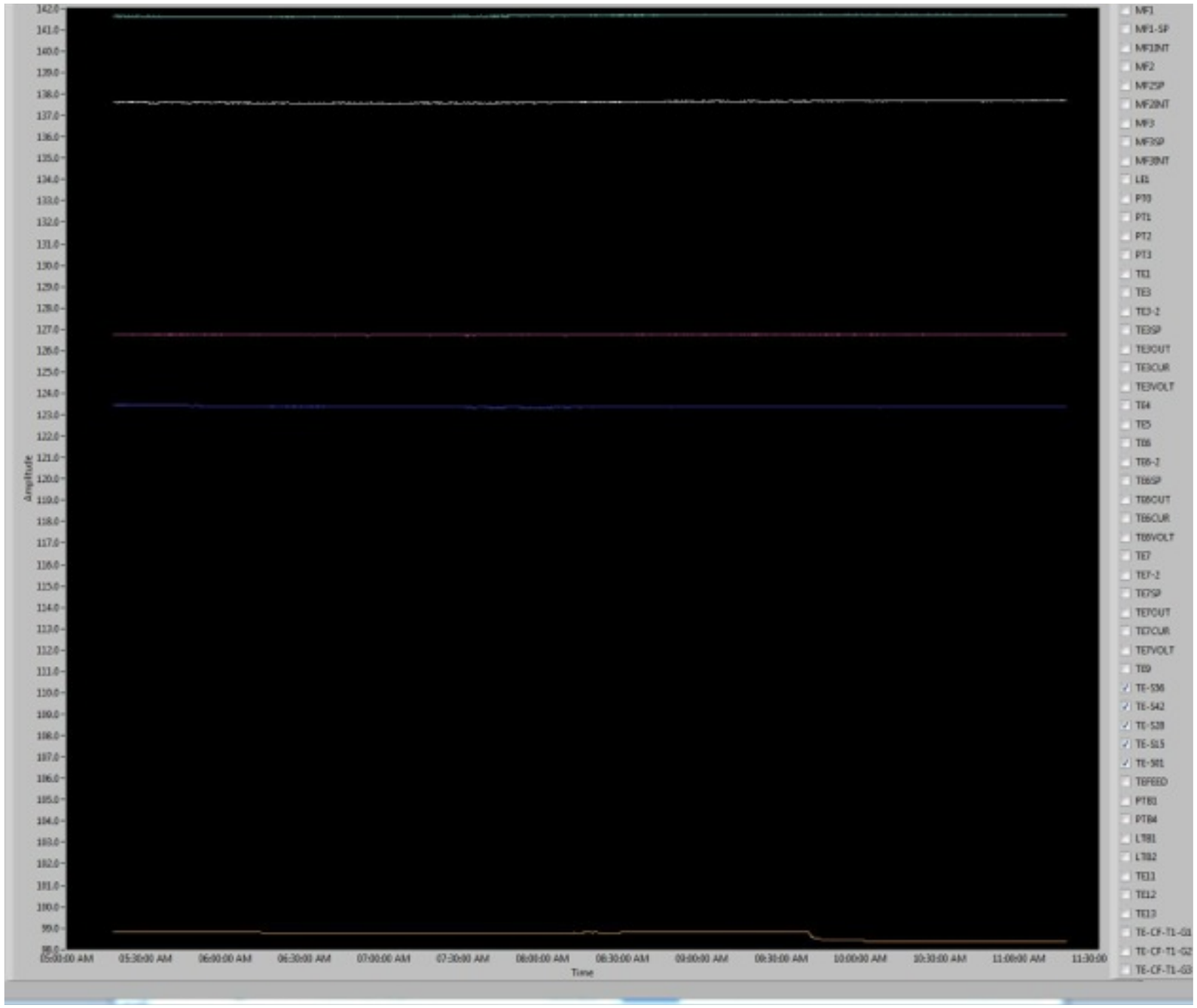
- Gas condenses on copper surface
- ridges increase surface area for more condensation
- At full reflux
  - 600 W in re-boiler heater ~130 sL/m of gas rising
  - Condenser heater does no work
  - Cryocooler is maintained at its set-point

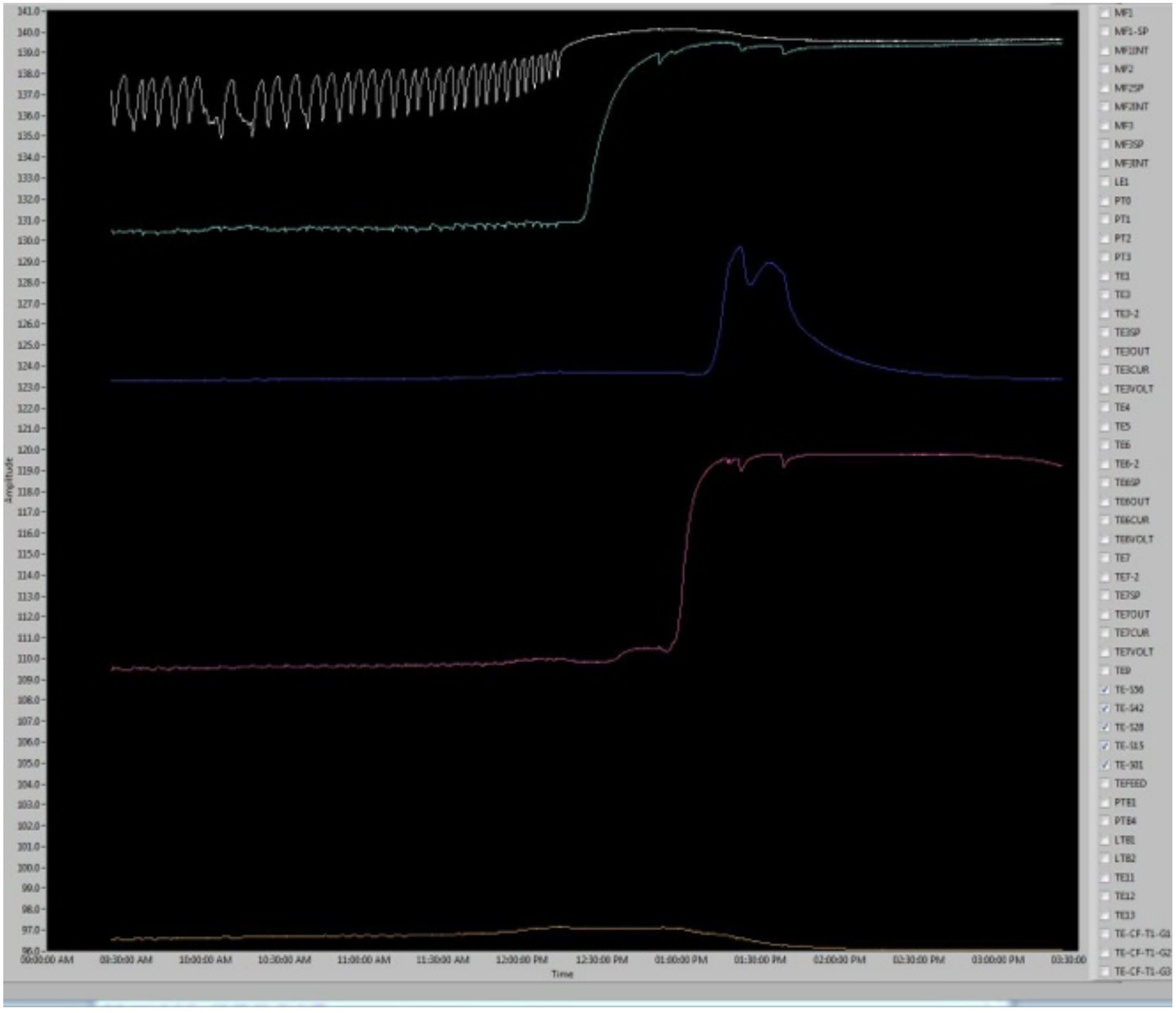




ARGON NITROGEN DISTILLATION

- Stable temperature transition



Argon nitrogen distillation

- Transition pushed down by nitrogen
- Unsure what causes oscillations