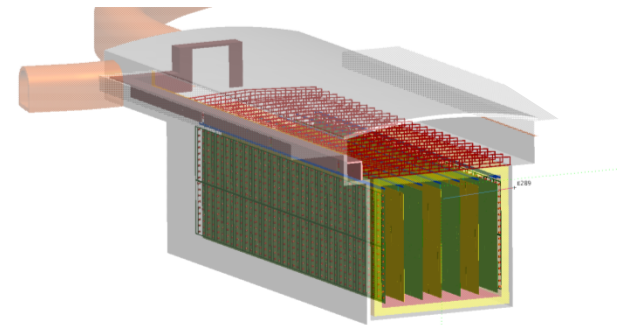
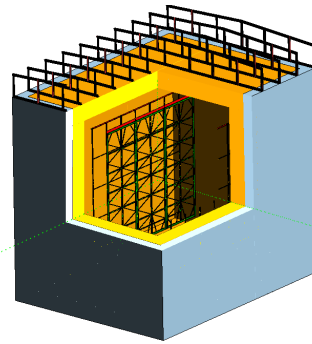
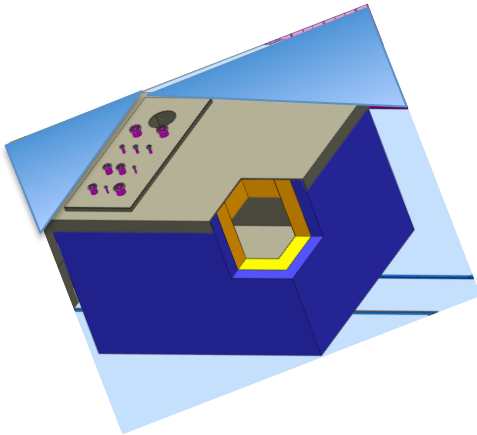


Some Work on

# Liquid Argon Purity at Fermilab



# Introduction

## ***Our approach/program for Liquid Argon TPC technical development:***

Learn, and keep learning, as much as we can from existing work – particularly ICARUS

Develop hands-on experience and our own infrastructure:

**filters**, cryogenics, pumps, electronics

Look at technical topics which may not have been fully explored

**material tests**, in-liquid electronics (*H. Chen*)

Put a detector in a neutrino beam to exercise a complete system (ArgoNeuT)

(*M. Soderberg*)

**Build a demonstration of good drift-lifetime in an unevacuated vessel.**

**Demonstrate good lifetime in an unevacuated membrane cryostat (35 ton liquid)**

(*R. Rucinski*)

Design, build and run the MicroBooNE Experiment in Booster Neutrino Beam

Construct a 1 ktonne prototype of the LBNE Detector Module ..

*All done in collaboration with other national laboratories and Universities*

# Introduction

## Some Relevant Numbers for electron drift-lifetime in Liquid Argon :

100 parts per trillion (ppt) Oxygen equivalent = lifetime of 3 milliseconds  
(contamination x lifetime = constant; 10 milliseconds = 30 ppt Oxygen equivalent)

Drift velocity at 500 V/cm = 1.6 mm/microsecond

=> 3.75 m drift = 2.35 milliseconds drift time;

Effect of poor lifetime is to reduce dynamic range and put more demands on electronics;

would like drift lifetime > two maximum drift-times (40% loss of signal)

# Introduction

Argon `purity' is a major emphasis of program of liquid Argon work at Fermilab

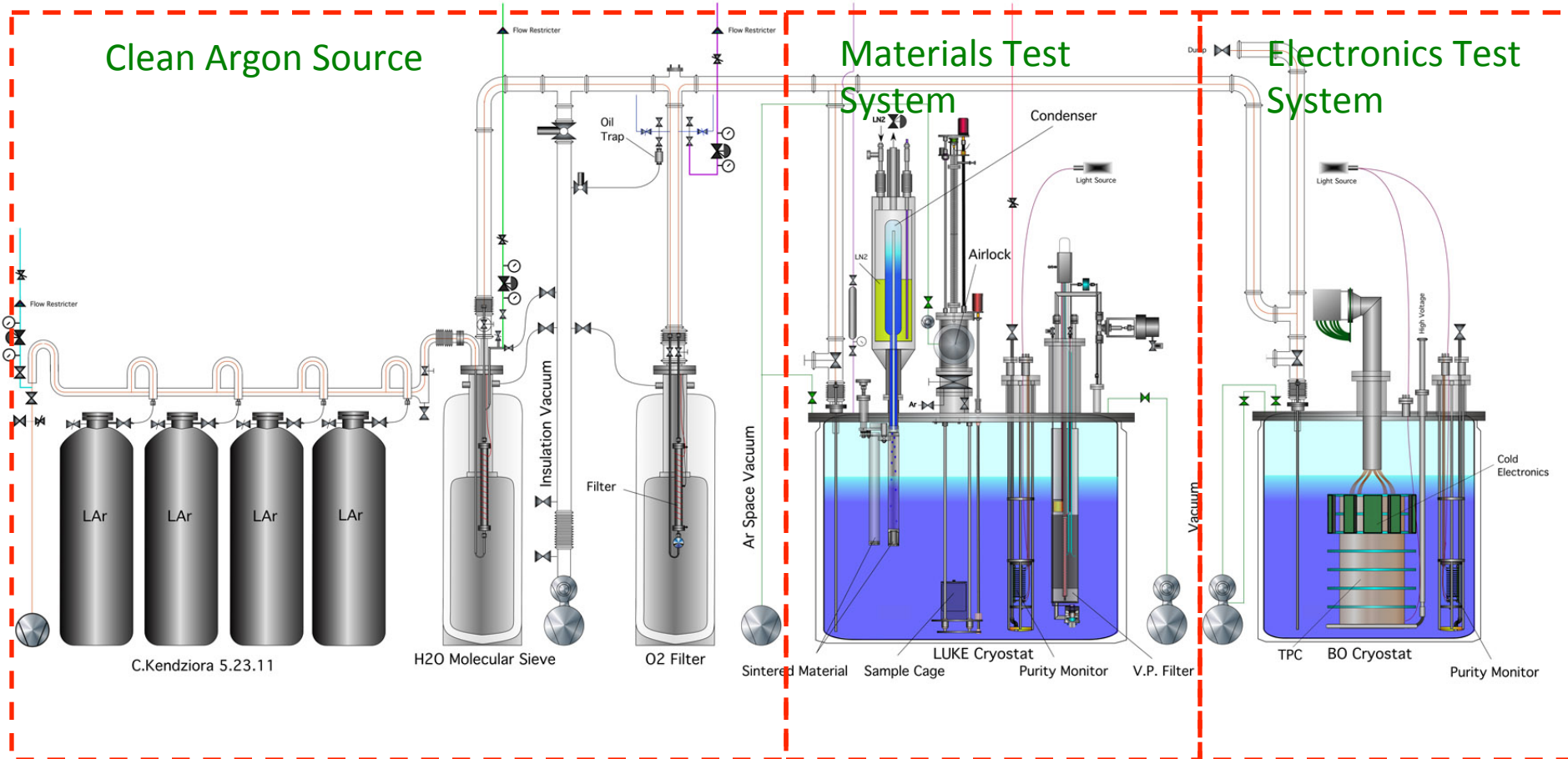
- **Materials Test Stand**
- **Liquid Argon Purity Demonstration (LAPD)**
- **LAr35t**

# Liquid Argon Materials and Electronics Test System Area



# Materials Test System

## Schematic of Argon Source, Materials Test System and Electronics Test System



Commercial Argon, Home-made Filters for H2O (Zeolite) and Oxygen (Engelhard Cu-0226)

N2 Condenser, Insertion Cage, 50 cm drift TPC, 3 readout planes, in-liquid electronics

# Materials Test System

Test Candidate Detector Materials for contamination of Argon:

## Features

Home-made Filters - regenerated in place

Can insert materials into known clean argon

Can insert materials after purging only or after pumping on them

Can position materials into liquid and into ullage giving range of temperatures

Can insert known amounts of contaminant gases

Nitrogen-based condenser can maintain liquid for long (weeks) studies

Internal filter-pump can remove contamination introduced by materials – 2hr cycle

Argon sample points at source, after single-pass filters, and in cryostat gas and liquid.



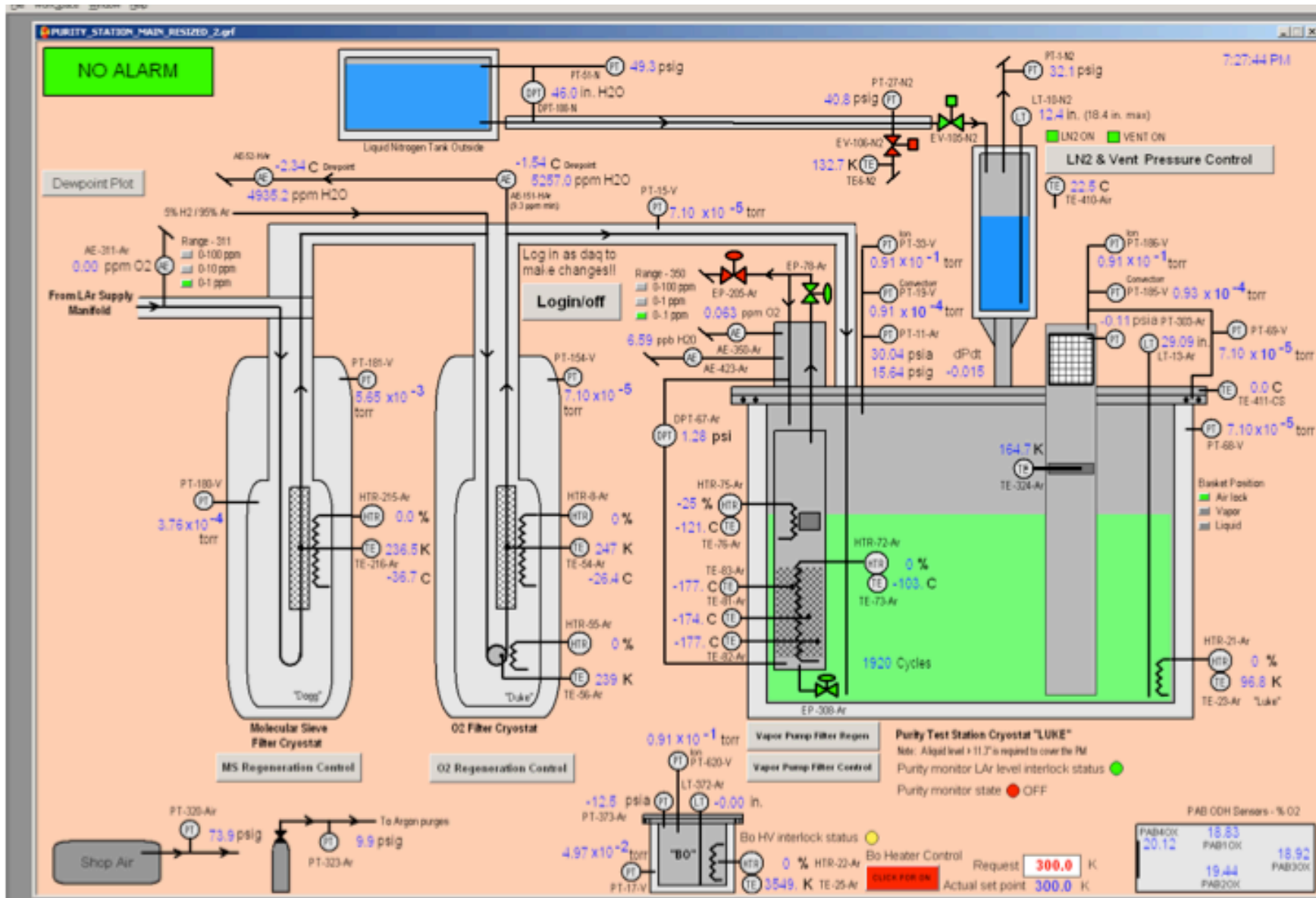
H2O Meter



O2 Meter

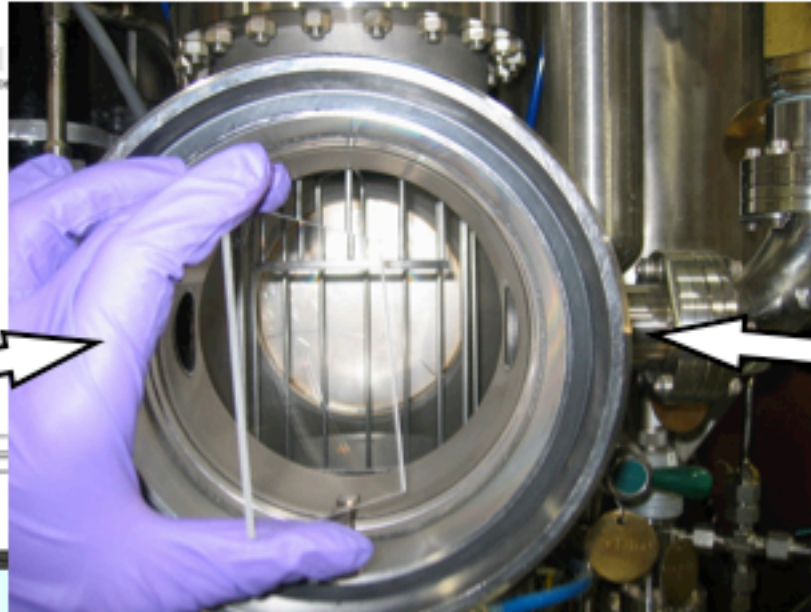
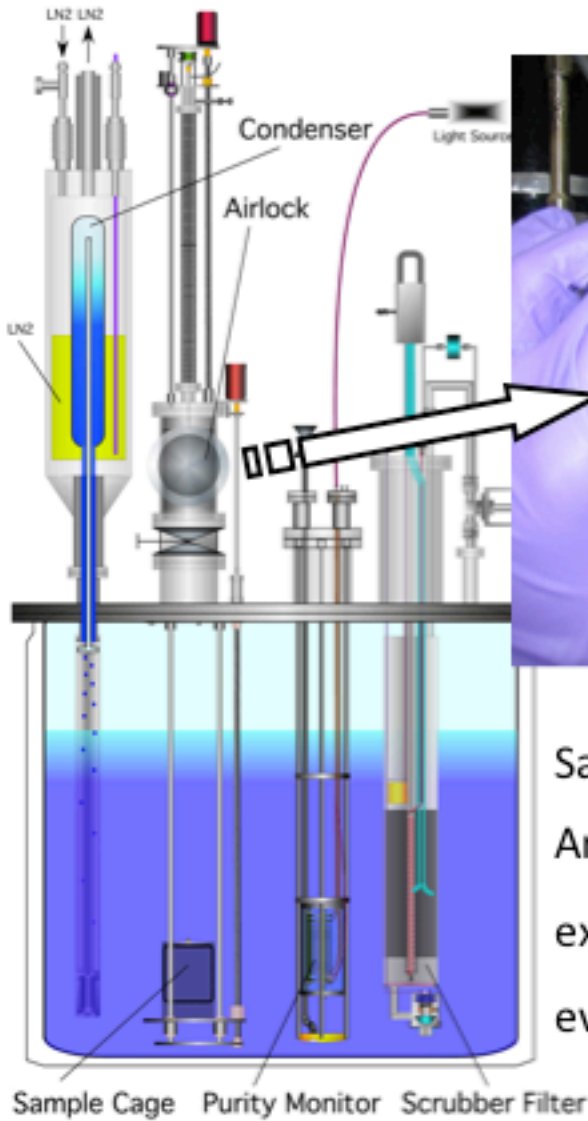
# Materials Test System

## Cryogenics Control Panel





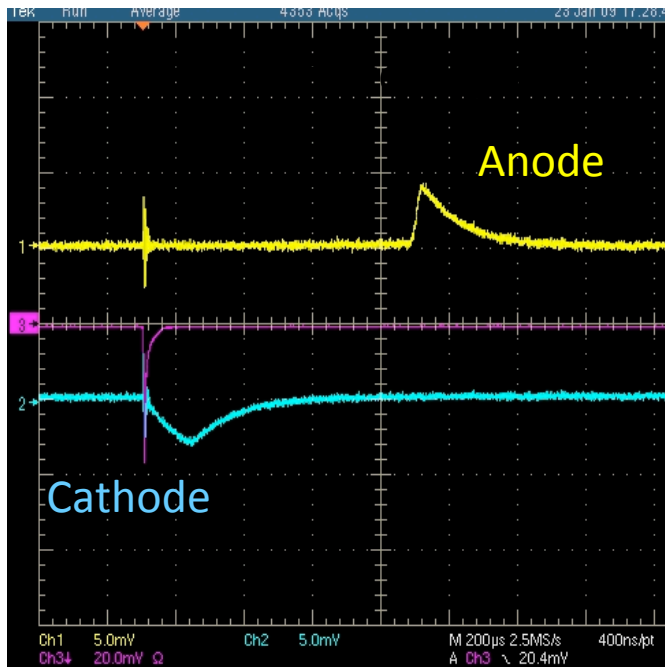
# Materials Test System



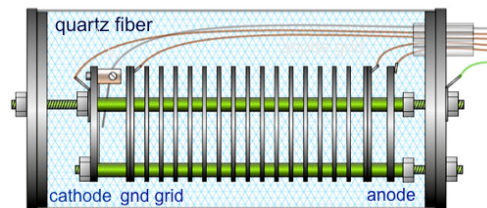
Sample volume 10 cm x 10 cm x 10 cm  
Argonlock can be purged with  
external argon, cryostat argon, and/or  
evacuated

# Materials Test System

- Measure electron drift lifetime (0.3 milliseconds to 10 milliseconds)
- Measure Oxygen (0.5 ppb sensitivity) with oxygen meter (Delta-F & Tiger Optics)
- Measure H2O in gas (0.5 ppb sensitivity) with water meter (Tiger Optics)
- Cryogenic data, Lifetime Data, analytic instrumentation data in single data-base
- Runs 24/7 unattended - except for filter regeneration and argon refills



Lifetime Monitor  
Analysis →



Lifetime Monitor  
a la ICARUS

Interval (Min) 120  
Remaining 85:55  
Sets 1  
Liquid Status

Waiting for Next Interval

Smoothing = 40  
RMS Cut = 10

Stop DAQ

Run Number 3146  
Run FileName C:\PrM Data\Run\_03146.txt  
Log File Path E:\

Results

1/23/2009 4:51:05 PM Run = 3146 Pass = 1 Diode Peak = -3.680e-02 Diode Time = 6.000e-06 Diode Baseline = -8.640e-04 Cathode Peak = -2.659e-03 Cathode Time = 1.380e-04 Cathode Baseline = 2.599e-04	Anode Peak = 3.746e-03 Anode Time = 8.228e-04 Anode Baseline = 1.073e-04 Anode Rise = 2.654e-05 Cath Factor = 1.724e00 Anode Factor = 1.138e00 Anode True = 4.350e-03 Cathode True = 5.032e-03 LifeTime = 5.647e-03
-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------	---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

O-Scope

Print Form

CH 1  CH 2  CH 3  CH 4

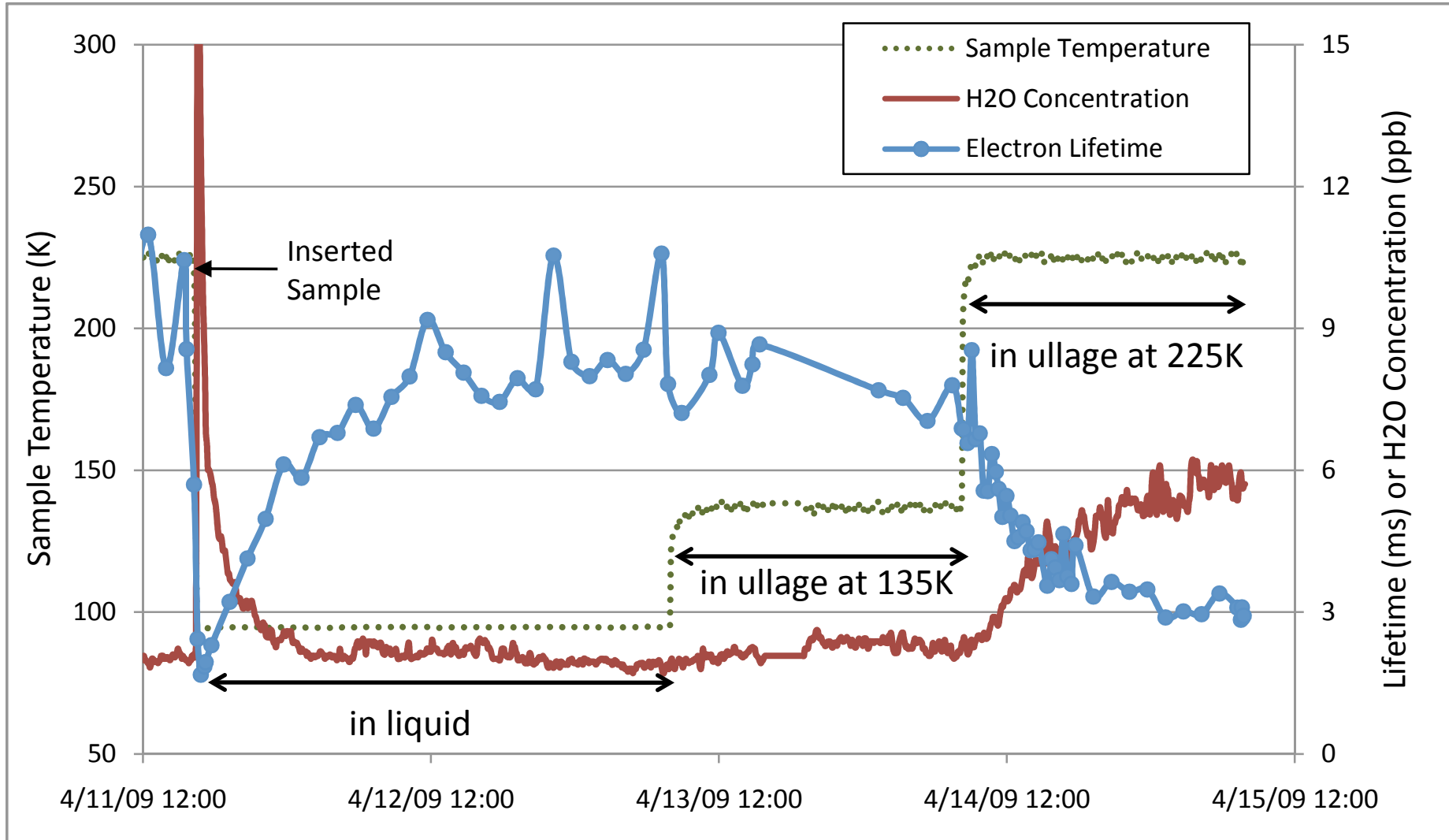
Analysis Wave Choice

Ch 1  Smooth  Raw  
Ch 2  Smooth  Raw  
Ch 3  Smooth  Raw

Display

# Materials Test System

FR-4 Sample in Materials Test System showing effects in liquid, in cold ullage and in warm ullage



# Materials Test System



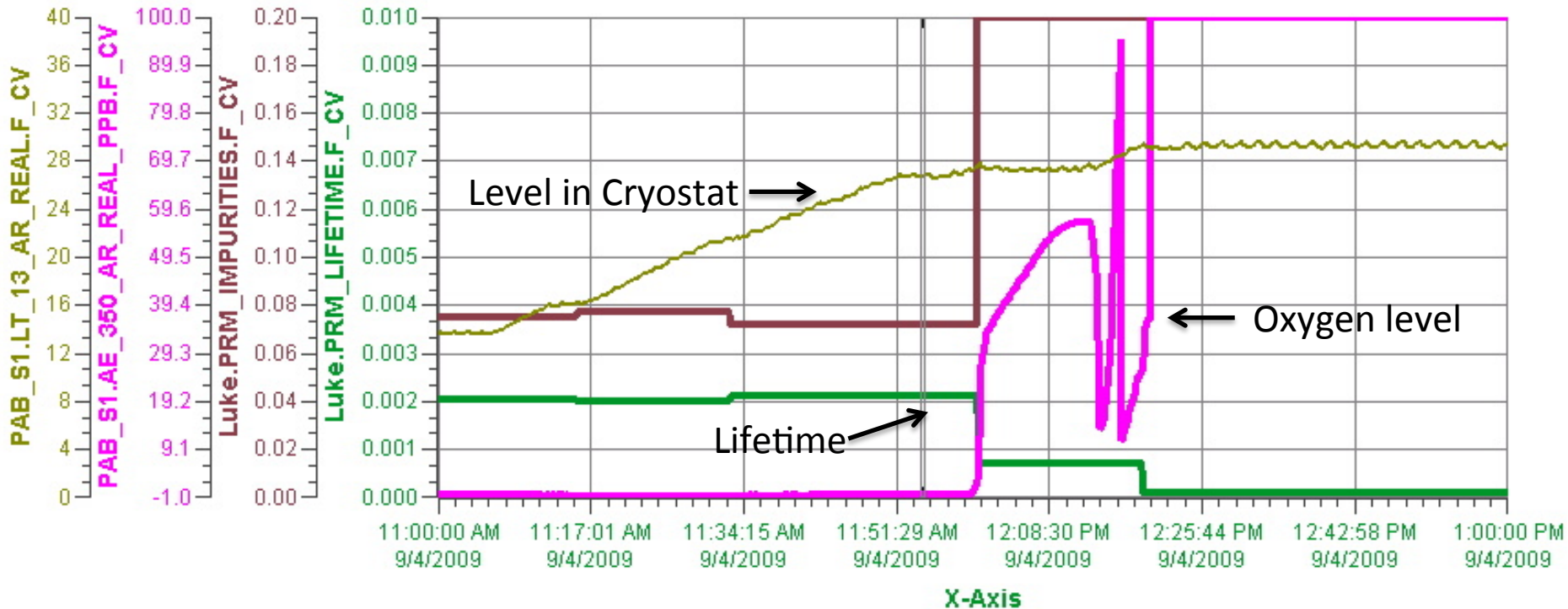
Sample of data on different materials

Material	Date test started	Preparation	Tests	Water [ppb]	Lifetime [ms]	LogBook #
Cleaning Solution	6/29/09	evac. 24 h	vapor/liquid	4	5	946
Vespel	7/9/09	evac. overnite	liquid/vapor	5-7	2-5, 4-6	960
MasterBond glue	7/16/09	purged 18 h	vapor/liquid	1.6	1.3- 2.9	974
LEDs	7/31/09	purged 38 h	vapor	3.5	5	993
Carbon filter material	8/12/09	evac. 24 h	liquid/vapor	2	4-9	1000
962 FeedTru Board V2	10/12/09	evac. 24 h	vapor/warm	85	1-5	1062
Teflon cable	1/9/10	purged 28 h	warm/liquid/vapor	8-20	2-5	1175
3M "Hans" connectors	1/29/10	purged 46 h	warm/liquid/vapor	5-12	3	1198
962 capacitors	3/2/10	evac. 24 h	warm/liquid/vapor	6-14	3-6	1228
962 polyolefin cable	4/12/10	evac. 16 days	warm	25-60	2	1237
Rigaku feedthrough	4/20/10	purged 7.5 h	warm	15	3	1250
Rogers board (Teppej)	4/23/10	purged 26 h	warm/liquid/vapor	40	2, 6-10	1254
Arlon Board (Teppej)	5/14/10	evac. 0.5 h, pur.2 days	warm/vapor	300, 80	1.3, 3.5	1263
Polyethylene tubing	5/24/10	evac. 6 h, pur. 66 h	warm	300-500	1	1278
Teflon tubing	5/27/10	evac. 1 h, pur.17 h	warm	9-13	4-5	1283
Jonghee board	5/28/10	evac. 6 h, pur. 1.5 h	warm/vapor	100,28	1.2, 5-8	1285
Jonghee connectors	6/4/10	evac. 3.5 h, pur. 16 h	warm/vapor	50	2-3	1290
PVC cable	6/14/10	evac. 29 h, pur.1 h	warm	120	1-2	1296
Teppej TPB samples	8/3/10	purged 26 h	warm	600-1600	0.7	1342
Teppej TPB samples	9/4/10	purged 37 h	liquid /vapor	15, 300	6	
PrM feed tru (baked)	10/5/10	purged 25 h	warm/vapor	35, 20	3, 2	1396
Copper foil on mylar film	10/14/10	purged 26 h	warm/liquid/vapor	15, 10, 9	3, 8, 7	1409
Teppej SHV connector	10/25/10	purged 25 h	warm/vapor/liquid	35, 11, 0	2, 6, 6	1415
FR4	11/16/10	purged 25 h	warm/liquid/vapor	180, 20, 65	1.5, 6, 2.5	1429
Gaskets	3/11/11	purged 24 h	warm/liquid/vapor	8, 10	2.5, 8, 7	1521
LBNE AP-219 Color. Developer	4/13/11	purged 25 h	warm/vapor	65, 15	4, >6	1722
LBNE RPUF Foam	4/22/11	evac. 26 h, pur.1 h.	warm	800	0.2	1729
LAPD LEDs	5/12/11	purged 49 h	vapor	0.6 ppb	10	1769

Good correlation with data in water-content data-base at [outgassing.nasa.gov](http://outgassing.nasa.gov)

# Materials Test System

Filter capacity & example of Filter 'Break Through'



Pen Name	Description	Value	Eng Units	High Over Range	Low Over Range
— Luke.PRM_LIFETIME....	Luke.PRM_LIFETIME.F_CV	0.00210	sec	0.00210	0.00005
— Luke.PRM_IMPURITIE...	Luke.PRM_IMPURITIES.F_...	0.0715	imps	2.7750	0.0715
— PAB_S1.AE_350_AR_...	ppb version for plotting (F_...	-0.80	ppb	463.06	-1.01
— PAB_S1.LT_13_AR_R...	Luke Argon Level Probe	26.6	inches	29.7	13.5

9/4/2009 11:00:00 AM

9/4/2009 1:00:00 PM

# Materials Test System - Summary

## Filter Materials:

Industrial filter materials are capable of removing all electronegative materials (water, oxygen) and producing liquid with >10 milliseconds lifetime.

Data on filter capacity at our requirements are sparse;

Filters can be regenerated many times; we know how - using non-flammable Ar-H mix

## Detector Materials:

Materials immersed in the liquid have no effect on lifetime;

Materials immersed in warm gas volume above the argon have:

- no effect on lifetime if the argon is venting at a certain rate;

- significant effect if the warm gas mixes with the liquid argon directly;

- metal surfaces outgas water vapor at significant rate and ... see above

## Contaminants:

Absent leaks, water is the main concern; it comes from detector materials and the cryostat walls in the warm gas.

'A system to test the effects of materials on the electron drift lifetime in liquid argon and observations on the effect of water' R. Andrews *et al.*, Nucl.Instrum.Meth.A608:251-258,2009.

# Materials Test System - Summary

## Design Considerations for any LAr TPC:

eliminate any cold surfaces in the ullage that will condense the contaminant-laden gas directly into the liquid;

select materials for the ullage that have minimum water content

=> no - nylon, yes - teflon;

minimize the amount of material in the warm ullage;

=> reduce cable plant by multiplexing TPC signals

arrange an adequate flow of argon gas away from the liquid; (ICARUS exploits this)

pass all return Argon through filters before allowing re-entry to liquid

## New Feature for multi-kiloton cryostats:

Cryostat not evacuable

=> LAPD (Liquid Argon Purity Demonstration)

# Challenges for multi-kiloton scale detectors

## Liquid Argon Purity Demonstration

*All previous LArTPC detectors have been evacuated before filling. Remove this constraint on the cryostat for large detectors.*

Demonstrate good life-time in an industrial vessel without evacuation.

First multi-ton purification system designed and built at Fermilab.

Expect to start commissioning early June using *Argon Piston™* to remove atmosphere and then circulate through filters (seen in foreground)

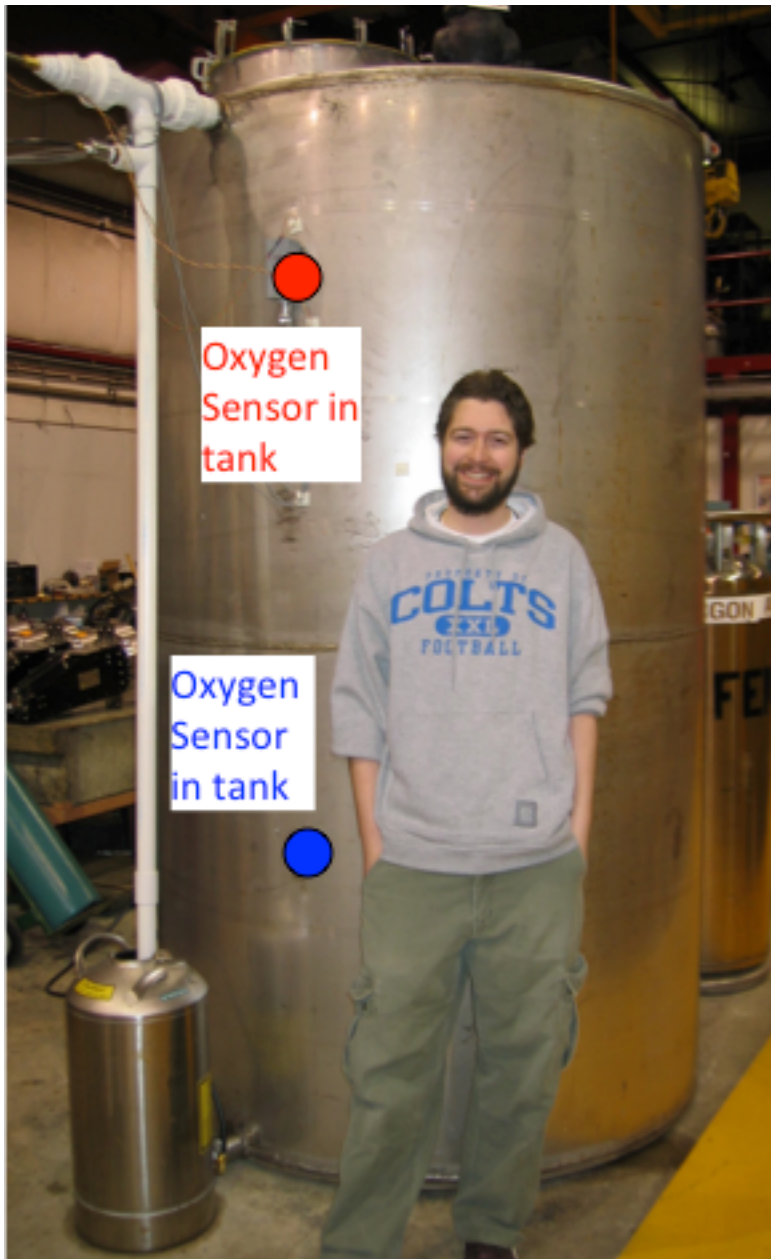
Stage 1 – bare tank

Stage 2 – with detector materials

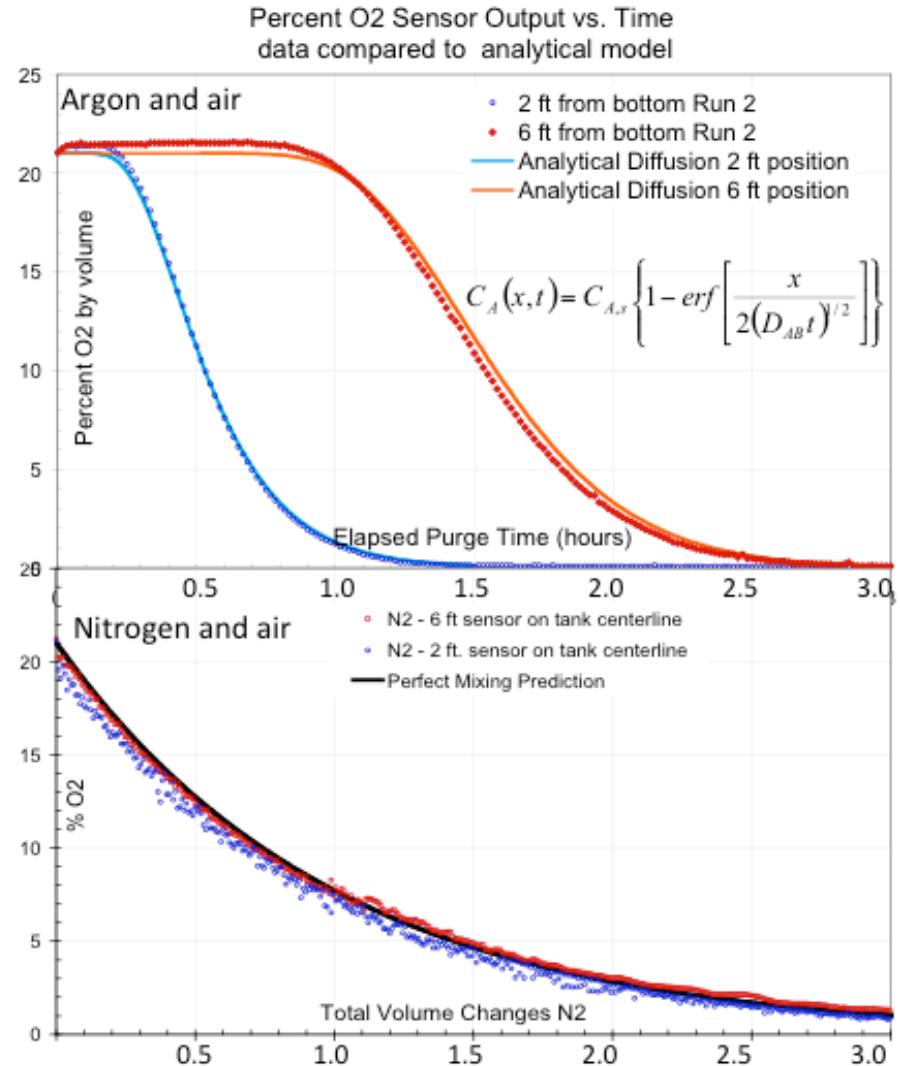




# Challenges for multi-kiloton scale detectors



## Argon Piston Demonstration



# Challenges for multi-kiloton scale detectors

## LAPD System Flow

LAr In

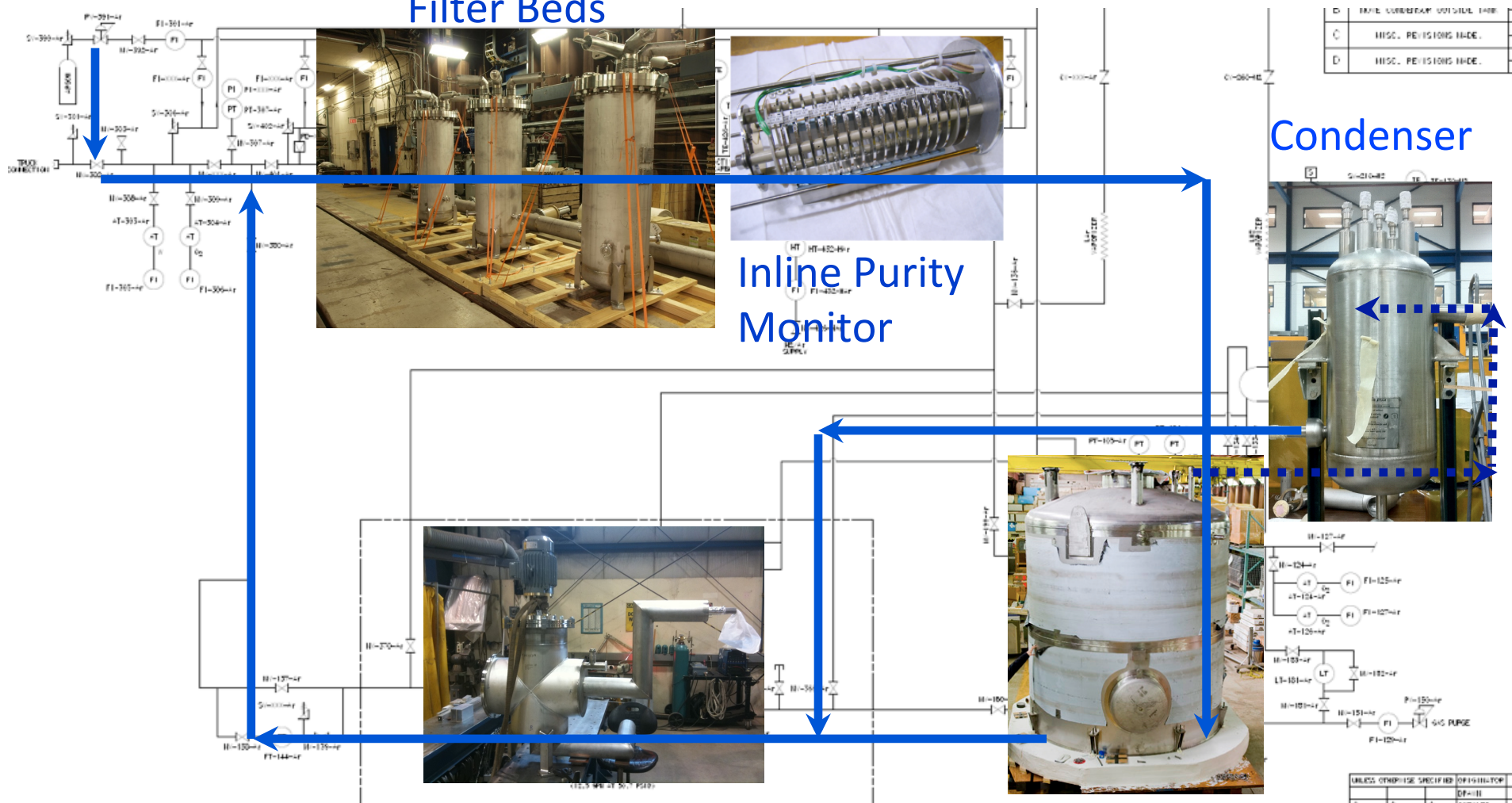
Filter Beds

Condenser

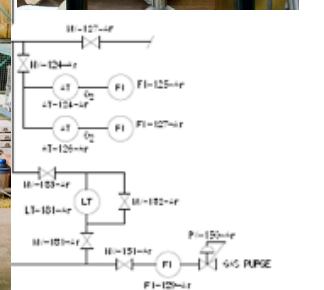
Inline Purity Monitor

Liquid Recirculation Pump

Tank



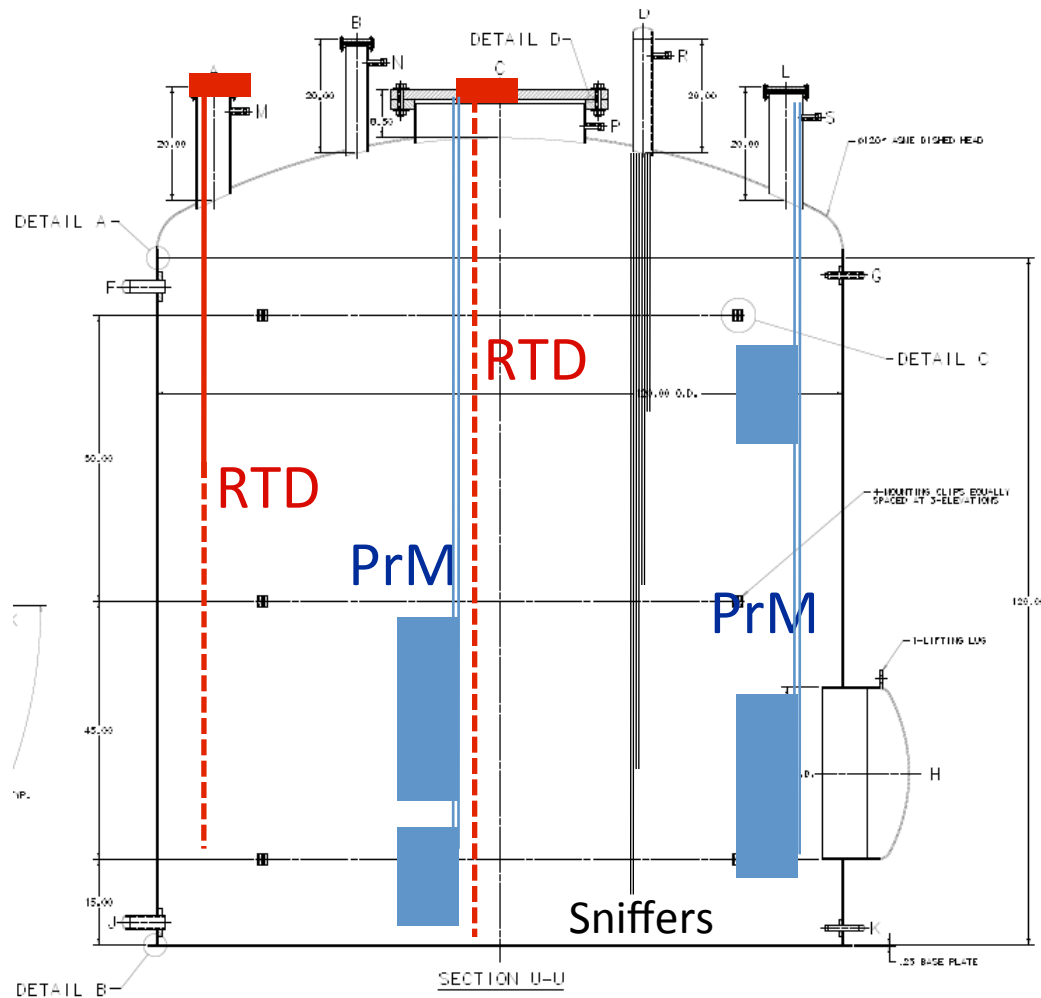
D	MISC. REVISIONS MADE.
C	MISC. REVISIONS MADE.
D	MISC. REVISIONS MADE.



UNLESS OTHERWISE SPECIFIED (OFFSHOOTER)	
±	DP=II
±	CHECKED

# Challenges for multi-kiloton scale detectors

## Instrumentation for LAPD:



### Analytic Equipment

Oxygen meters (0.4 ppb sensitivity)

H2O meters (0.5 ppb sensitivity)

N2 meter (20 ppb sensitivity)

*can sample multiple points*

### In the Tank

2 sets of 2 PrM (20 cm / 60 cm)

2 sets of 3 translating RTDs

Sniffer set to measure purge evolution

### Inline

Purity Monitor

# Challenges for multi-kiloton scale detectors

## LAPD Program

### Phase 1:

*Demonstrate good electron lifetime in an unevacuated vessel;*

Measure the effectiveness of the first purge at removing oxygen;

Measure how long it takes to remove water and oxygen from vessel to ppm level;

Measure temperature distribution in the liquid and in the ullage and compare with simulations -> input for convective flow calculations

Measure lifetime as a function of liquid recirculation rate;

Measure contamination rate;

Measure filter capacity;

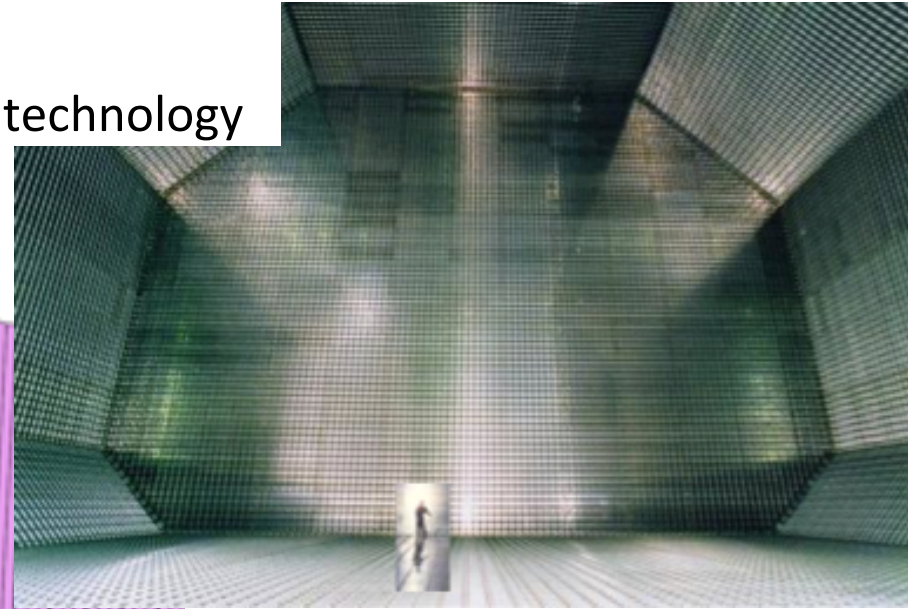
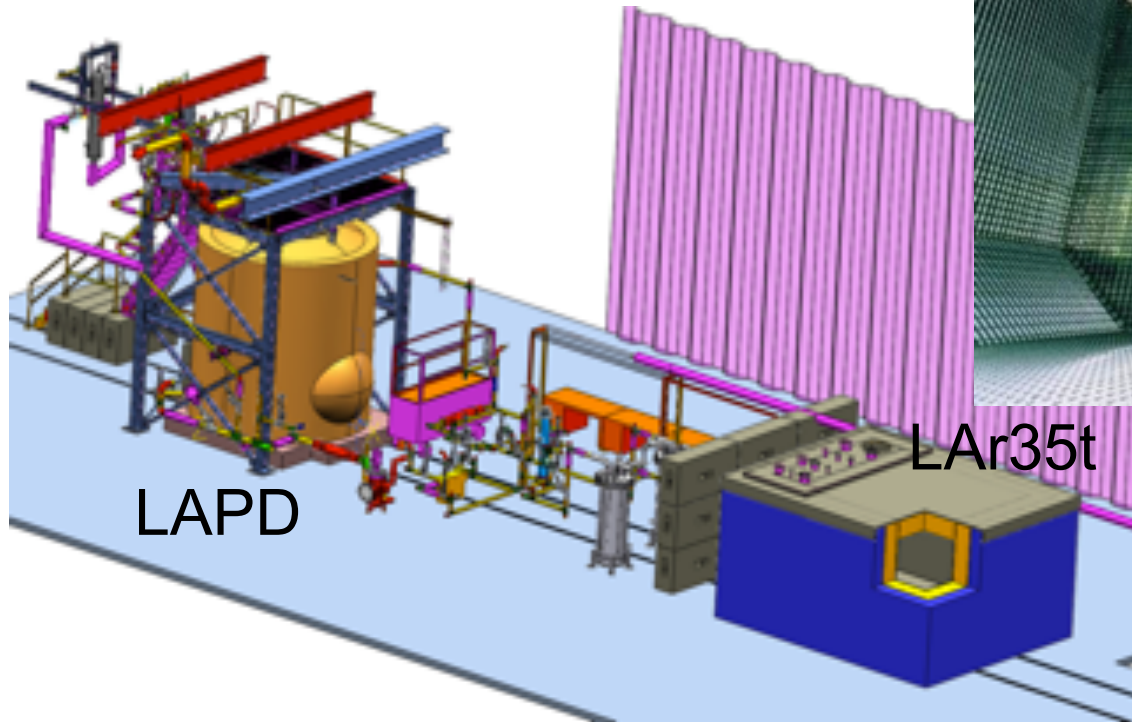
### Phase 2:

Install Detector Material (cables, G-10 frames) in the ullage and back to list for Phase 1

# Challenges for multi-kiloton scale detectors

## Membrane Cryostat Technology

based on modern LNG transport and storage technology



LNG Tanker Hull  
(see talk of R. Rucinski)

The LAr35t cryostat is our first foray into the use of membrane cryostats. It will use the same cryogenics and purification system as developed for the Liquid Argon Purity Demonstration and go through the same program. (We have tested a sample of the membrane wall in the MTS and it is fine)

# Dark Matter – Depleted Argon Distillation

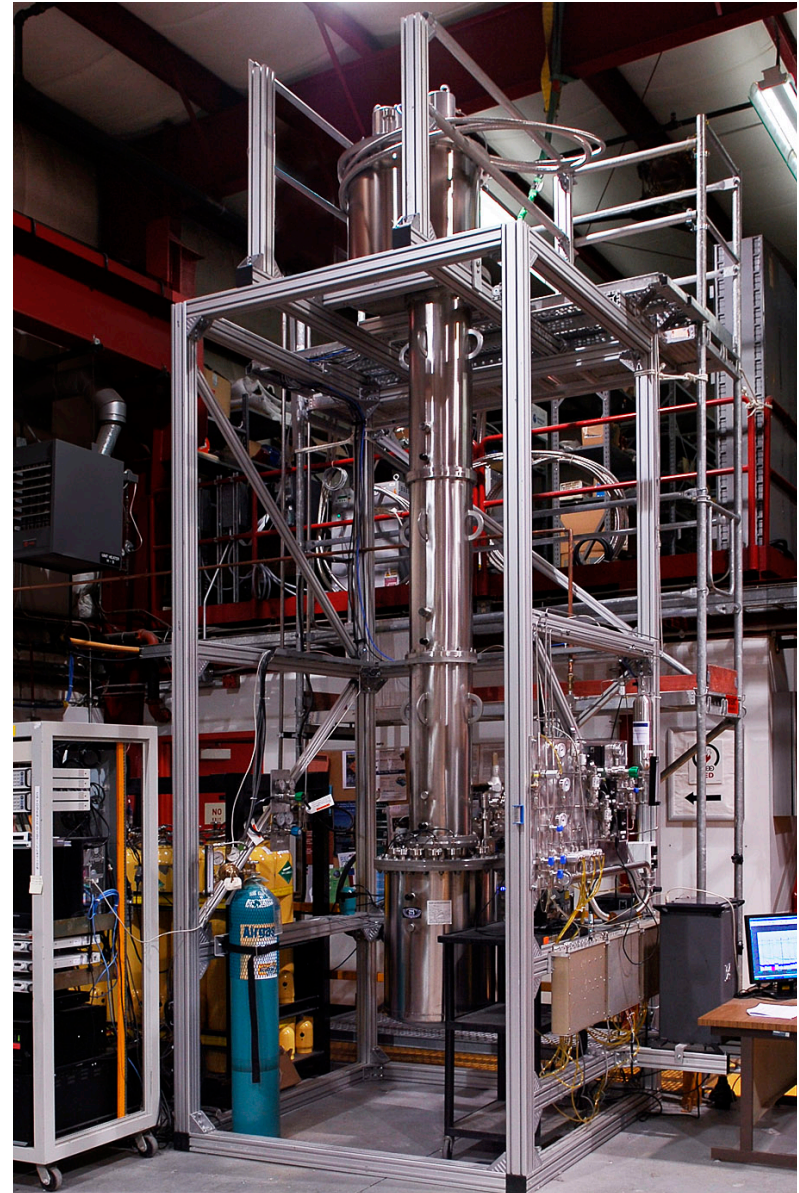
Atmospheric Argon contains  $^{39}\text{Ar}$  which produces a background rate of 1 Bq/kg limiting the size of LAr Dark Matter 2-phase TPCs to  $\sim 1$  ton because of dead-time.

Commercial  $\text{CO}_2$  wells have been found \* which contain 400 ppm Argon with  $<1/25$  (measurement limit) the atmospheric concentration.

**This Argon represents an unique resource for the Dark Matter program.**

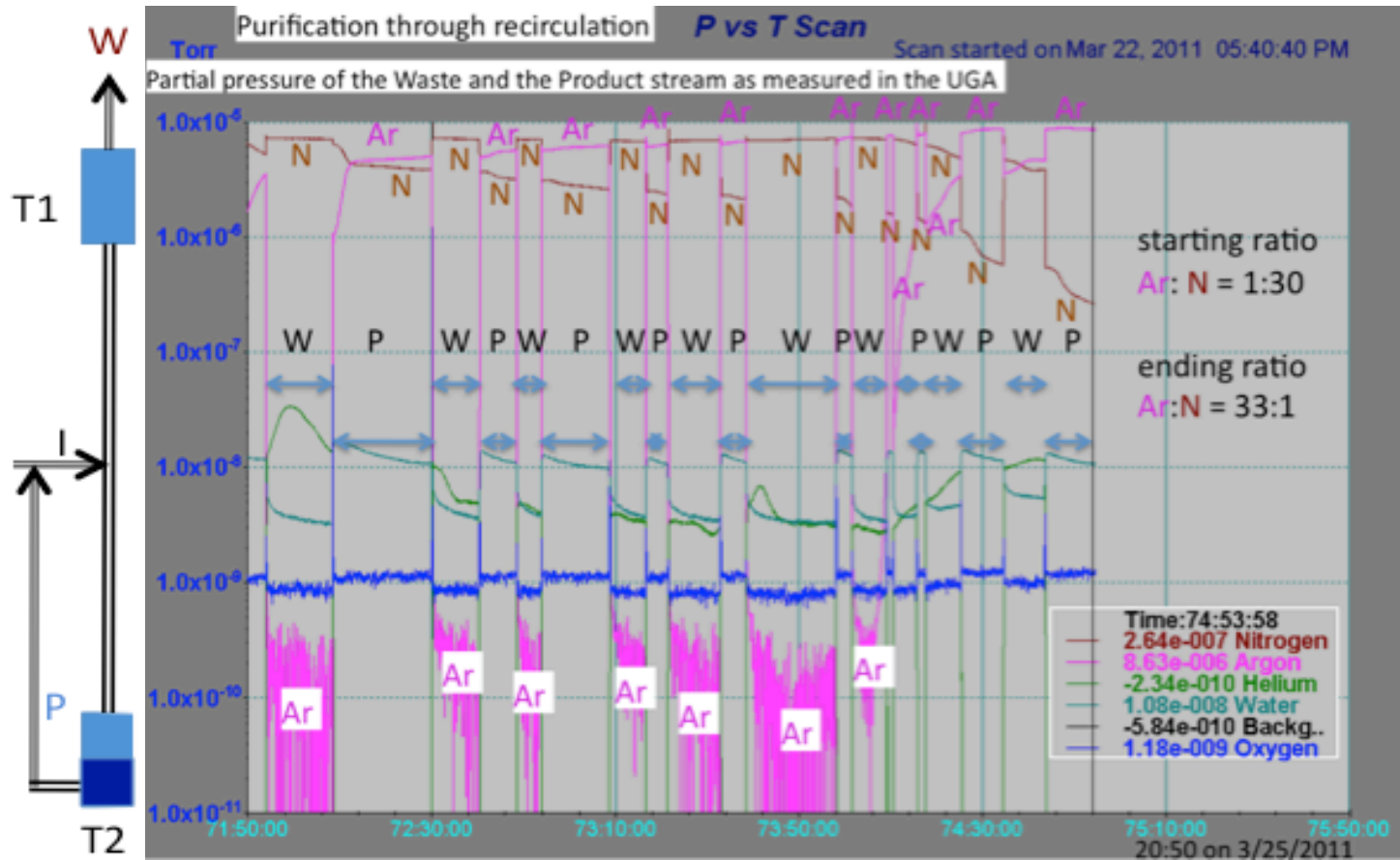
After removal of the  $\text{CO}_2$  a mixture of 3% Ar, 27% He and 74%  $\text{N}_2$  is obtained. Fermilab has agreed to host the distillation column designed to separate the Argon.

The basic column was designed at Princeton. It was assembled, safety reviewed, and has had its first run at FNAL. A series of upgrades to increase throughput is underway.  
(see talk of H. Back)



# Dark Matter – Depleted Argon Distillation

## First Distillation



Can distill efficiently but so far only in batch mode

Upgrades planned to improve cooling at input to achieve continuous distillation

Condenser-Booster (to put Argon into high pressure cylinders) is to be brought online

# Conclusion

**There is a broad and vigorous program at Fermilab to address issues of Argon purity.**

The **Materials Test System** is a unique facility for measuring the effect of materials on drift-lifetime. It has guided/is guiding us in identifying appropriate materials and developing the strategy for obtaining good lifetime.

The **LAPD** is aimed to demonstrate that we can indeed achieve good electron drift-lifetime in a large system, and to validate the techniques to get there and the models used to simulate liquid temperature distributions.

It will serve as a prime data source for understanding 'purging to purity'. We expect to start commissioning in late June.

**LAr35t** will repeat this using the specific material and design of the cryostat proposed for LBNE.

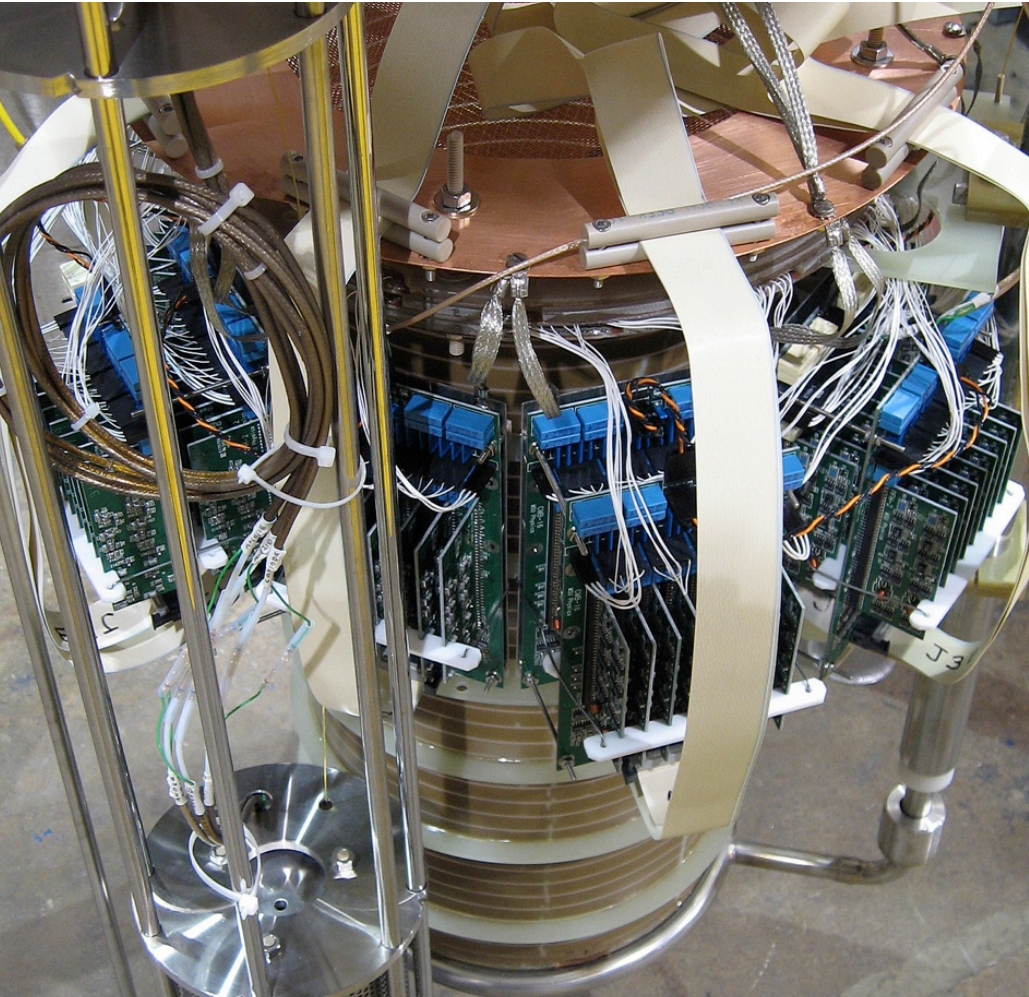
**The Distillation Column** will provide Argon that is pure in both a chemical and radiologic sense.



fin....

# Electronics Test System

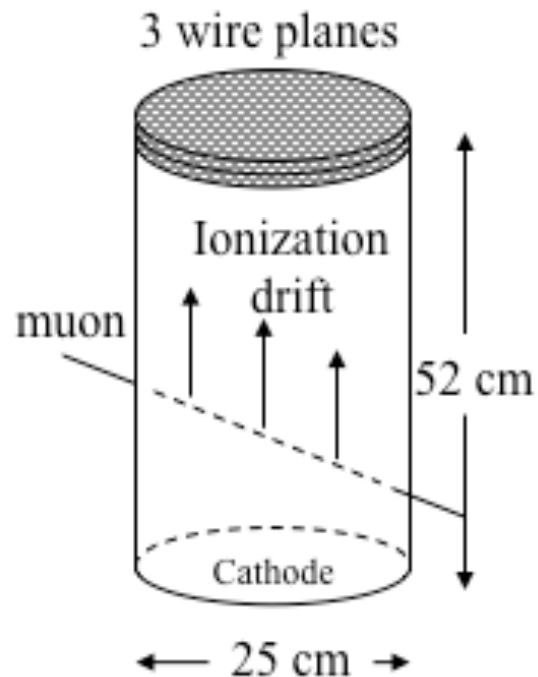
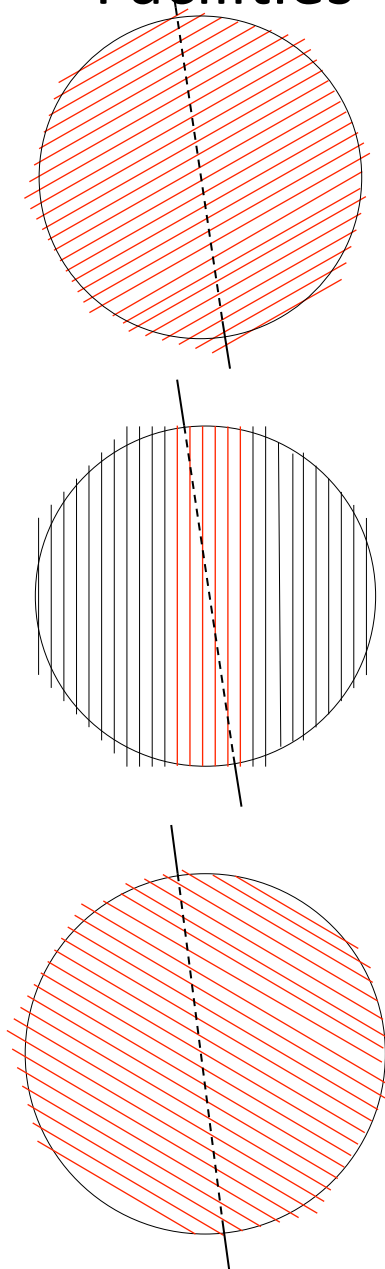
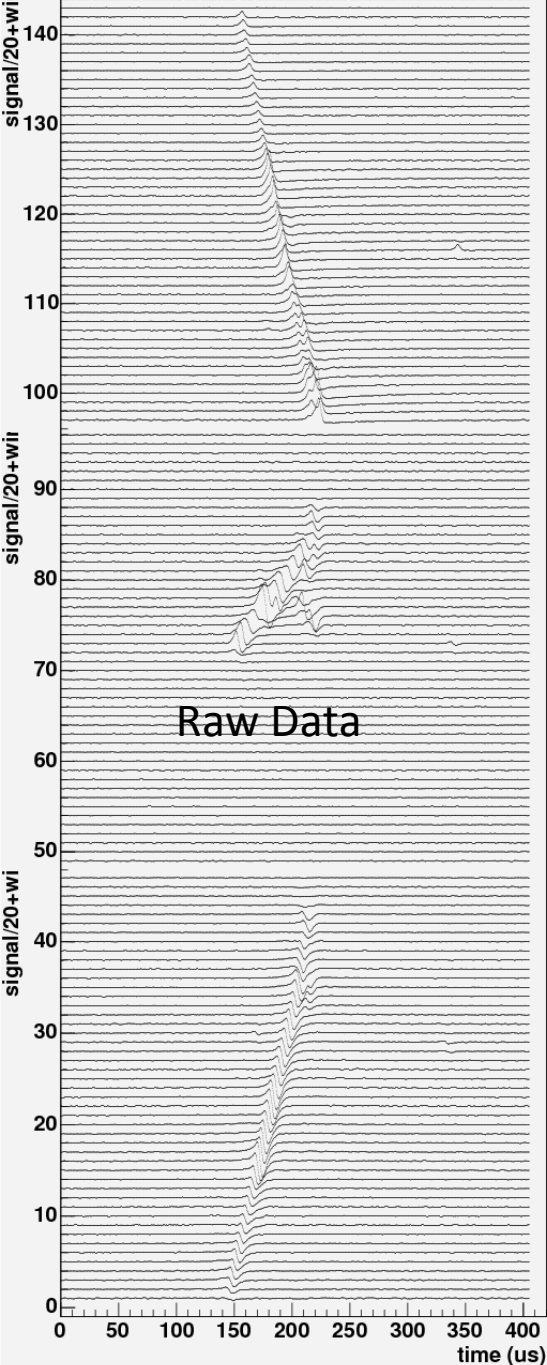
1<sup>st</sup> in-liquid 'CMOS' electronics on a LArTPC  
– built at Michigan State Univ.



CMOS based design.

- Common technology.
- Operates well at 90K.
- Can be converted to ASIC.
- Capacitors and Inductors require careful selection.
- Connectors and cabling need careful testing.
- All connections need robust mechanics.

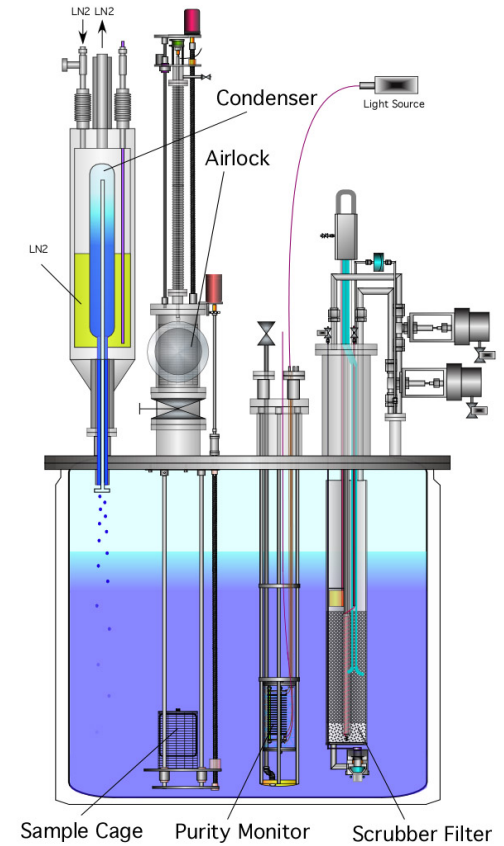
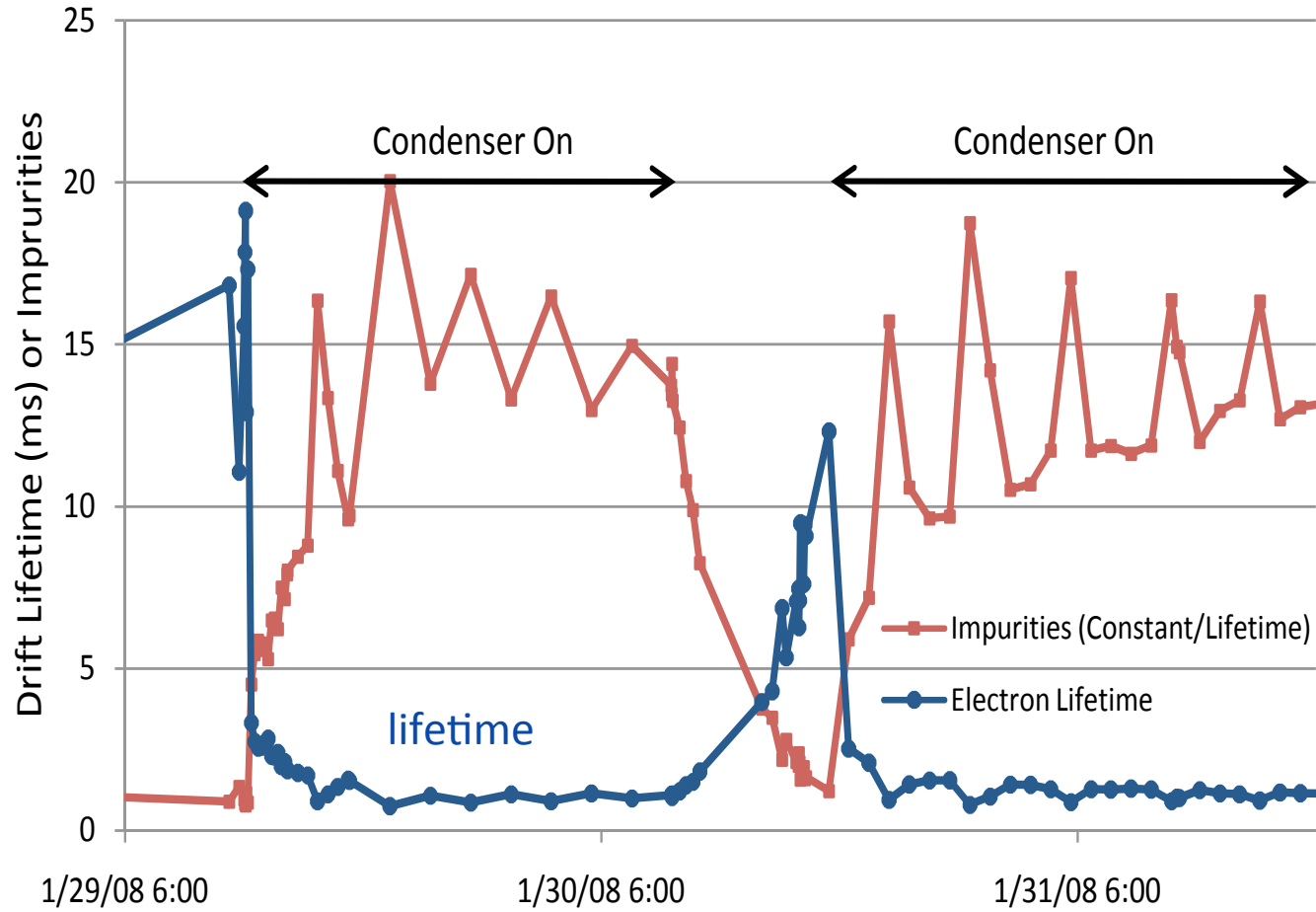
# Facilities – Electronics Test System



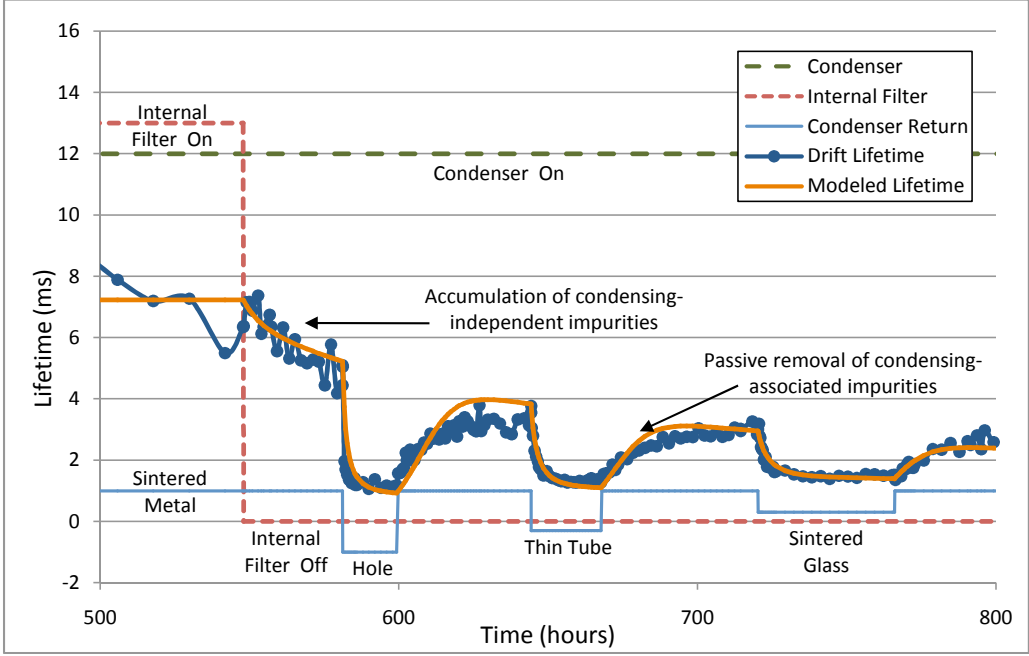
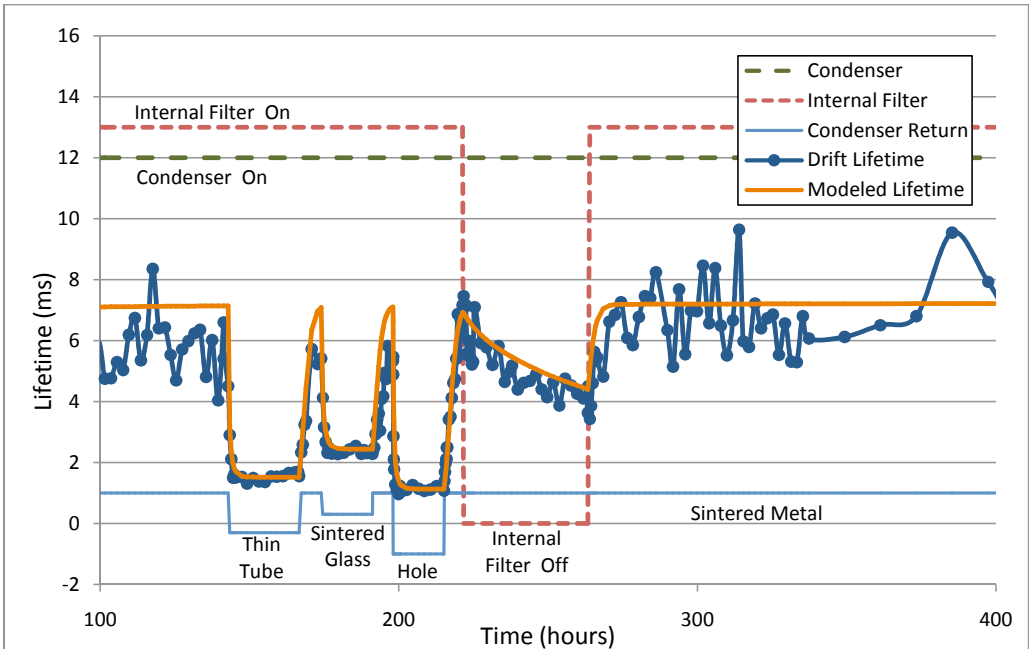
In-liquid electronics

S/N looks very promising

# Our first experience of a closed system – returning re-condensed argon directly to liquid



# Drift Lifetime Study with different returns



Condensed argon exits condenser from 1 inch tube concentrically above 1.5 inch filter housings

1.5 inch tube with no filter material

3/8 inch stainless steel tube, slightly spiraled

Sintered materials housed in 1.5 inch stainless steel tube

Sintered glass, 10-15 um pores

Sintered metal, 10 um pores

Stainless steel wool

