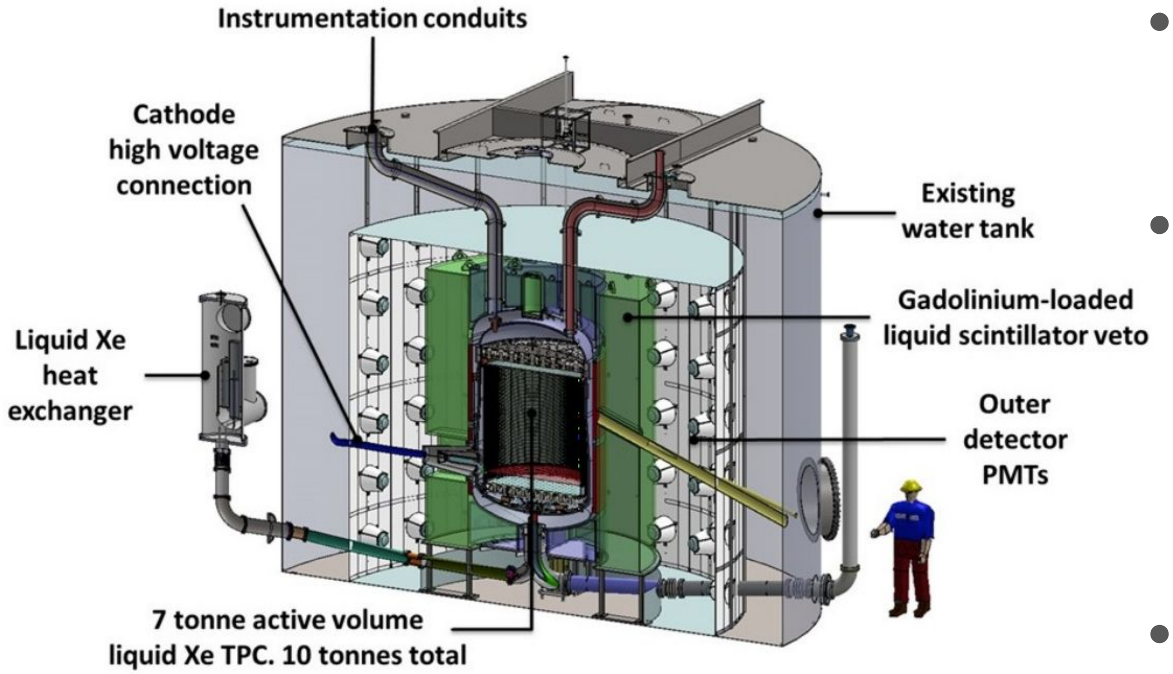
Krypton Removal via Gas Chromatography for LZ

Drew Ames, on behalf of the LZ collaboration

LRT 2022

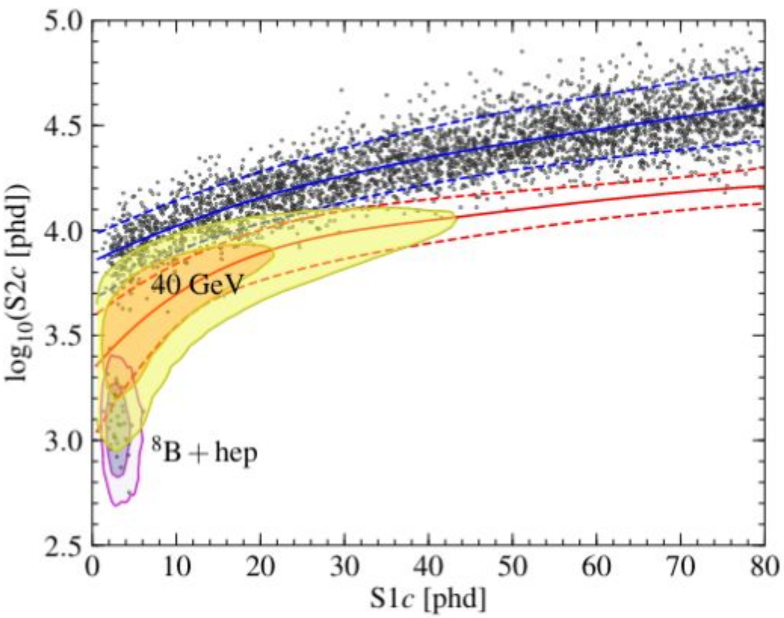
The LUX-Zeplin (LZ) Detector



- Located at Sanford Underground Research Facility in Lead, SD
- Dual-phase xenon time projection chamber with 7 tonne active volume (10 tonnes total xenon)
- Energy depositions in the TPC result in light (S1) and charge (S2) signals
    - 3D position reconstruction using PMTs (x-y) and drift time (z)
    - Discriminate nuclear vs electron recoils with S1/S2 ratio
- Outer detector system (Gd-loaded liquid scintillator + instrumented xenon skin) for rejection of external backgrounds

Backgrounds in LZ

Simulated LZ background data for 1000 live days
Blue: ER band, Red: NR band



(Projected WIMP sensitivity of the LUX-ZEPLIN (LZ) dark matter experiment, https://arxiv.org/pdf/1802.06039.pdf)

Background contributions for 1000 live days

Source	Nuclear recoils	Electron recoils
Detector components	0.07	9
Xenon contaminants	0	819
Laboratory and cosmogenics	0.06	5
Surface contaminants	0.39	40
Physics (neutrinos, ^{136}Xe $2\nu\beta\beta$)	0.51	258
Total (before ER discrimination & NR efficiency)	**1.03**	**1195**

Xenon contaminants: radioactive nobles

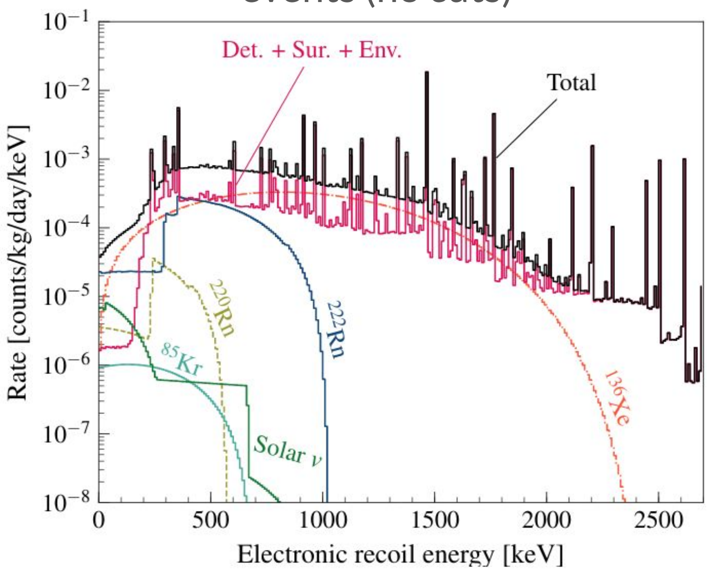
Isotope	ER cts
^{222}Rn (1.8 μBq/kg)	681
^{220}Rn (0.09 μBq/kg)	111
^{nat}Kr (0.015 ppt g/g)	24.5
^{nat}Ar (0.45 ppb g/g)	2.5

Krypton-85 ER backgrounds

^{85}Kr β decay

$$^{85}_{36}\mathrm{Kr} \xrightarrow[10.8\mathrm{y}]{} {}^{85}_{37}\mathrm{Rb} + e^- + \bar{\nu}_e$$

ER background spectra in the fiducial volume for single scatter events (no cuts)

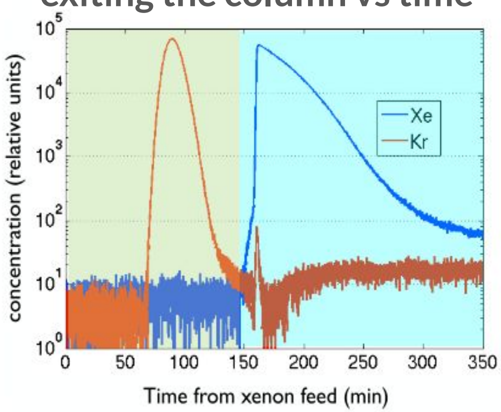


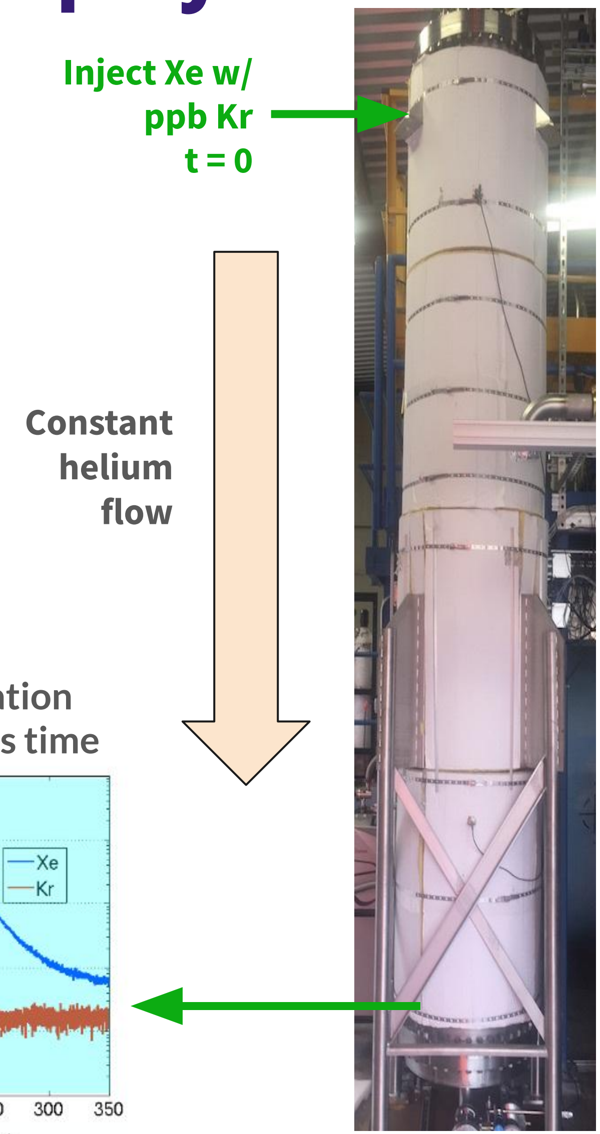
- Krypton naturally occurs in atmosphere at about 1 part per million (ppm, 10^{-6}) by volume
- About 1 part in 10^{-11} ^{nat}Kr is beta emitter ^{85}Kr
- Xenon extracted from the atmosphere contains trace krypton
  - Commercial research grade xenon contains about 1,000 - 10,000 parts per trillion (ppt = 10^{-12}) ^{nat}Kr (by mass)
- LZ goal: ^{85}Kr beta decay background comparable to irreducible solar neutrino background
- Requires purity of less than **0.3 parts per trillion (3×10^{-13})** g/g ^{nat}Kr in Xe

Gas Charcoal Chromatography

- **Chromatography**: separation of a mixture based on differing transit times through a stationary medium (charcoal)
- Xenon & krypton atoms adsorb onto charcoal due to van der Waals forces caused by random fluctuations in polarization
- Xenon is more polarizable → longer transit time
- Helium carrier gas maintains flow through column
- We use this technique to separate krypton from xenon at SLAC before transporting xenon to SURF

Relative concentration exiting the column vs time



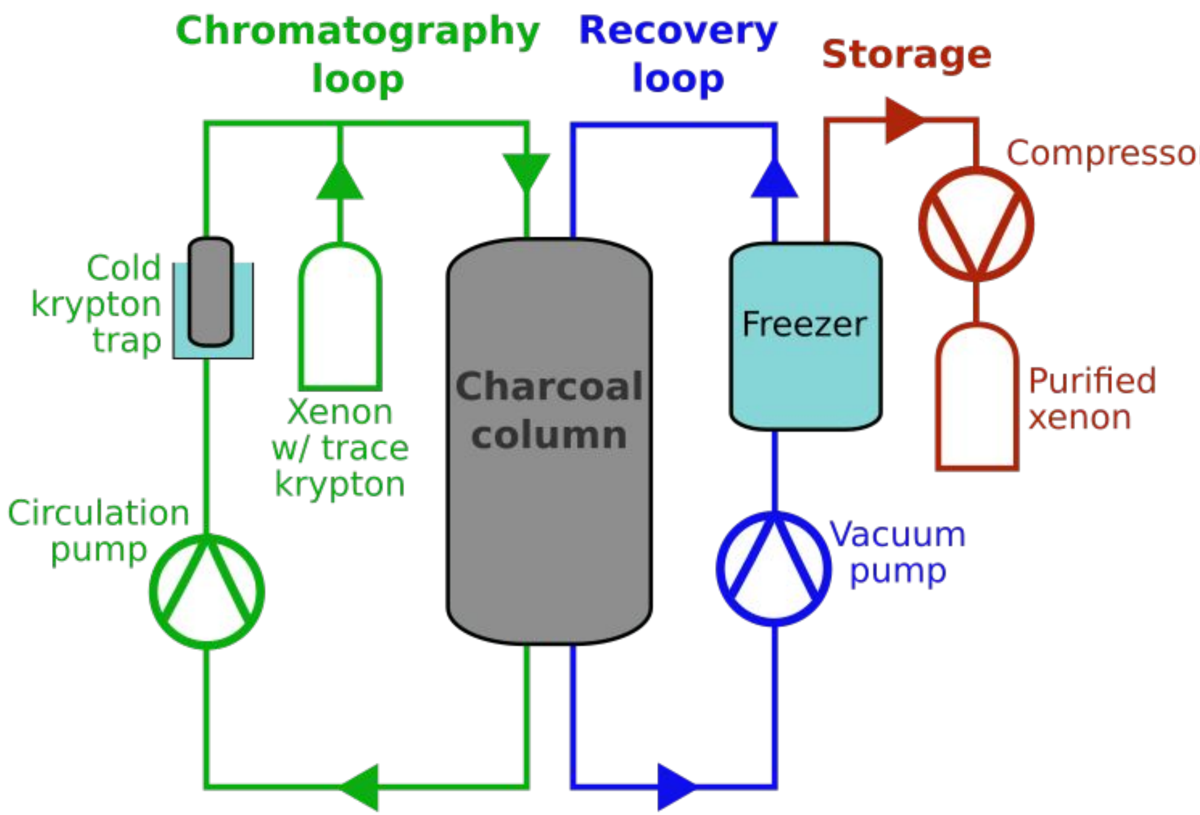


Krypton removal system overview

1. **Chromatography:**
   Xe gas injected into column; Kr separated and discarded

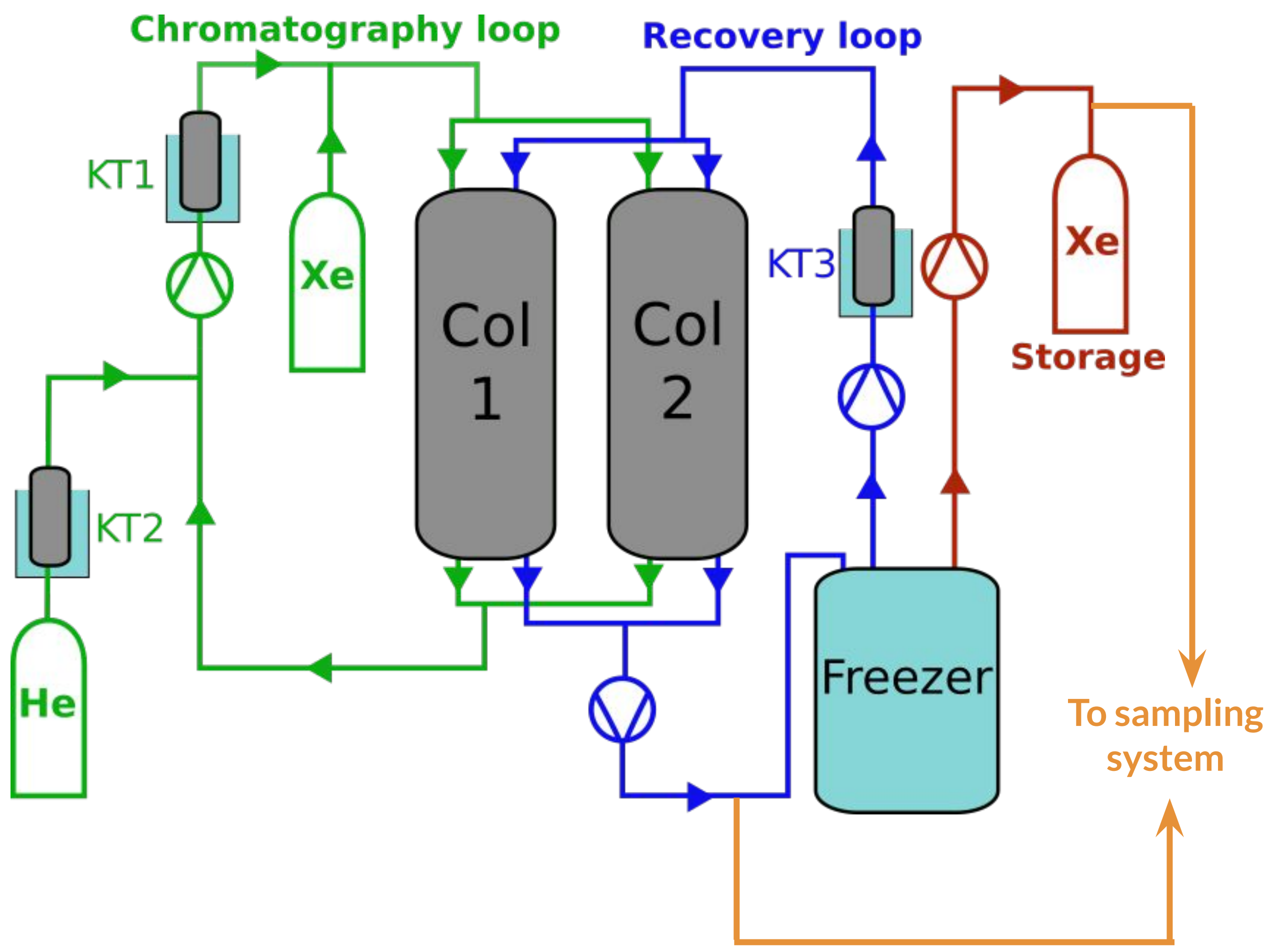
2. **Recovery:**
   Purified Xe transferred to freezer

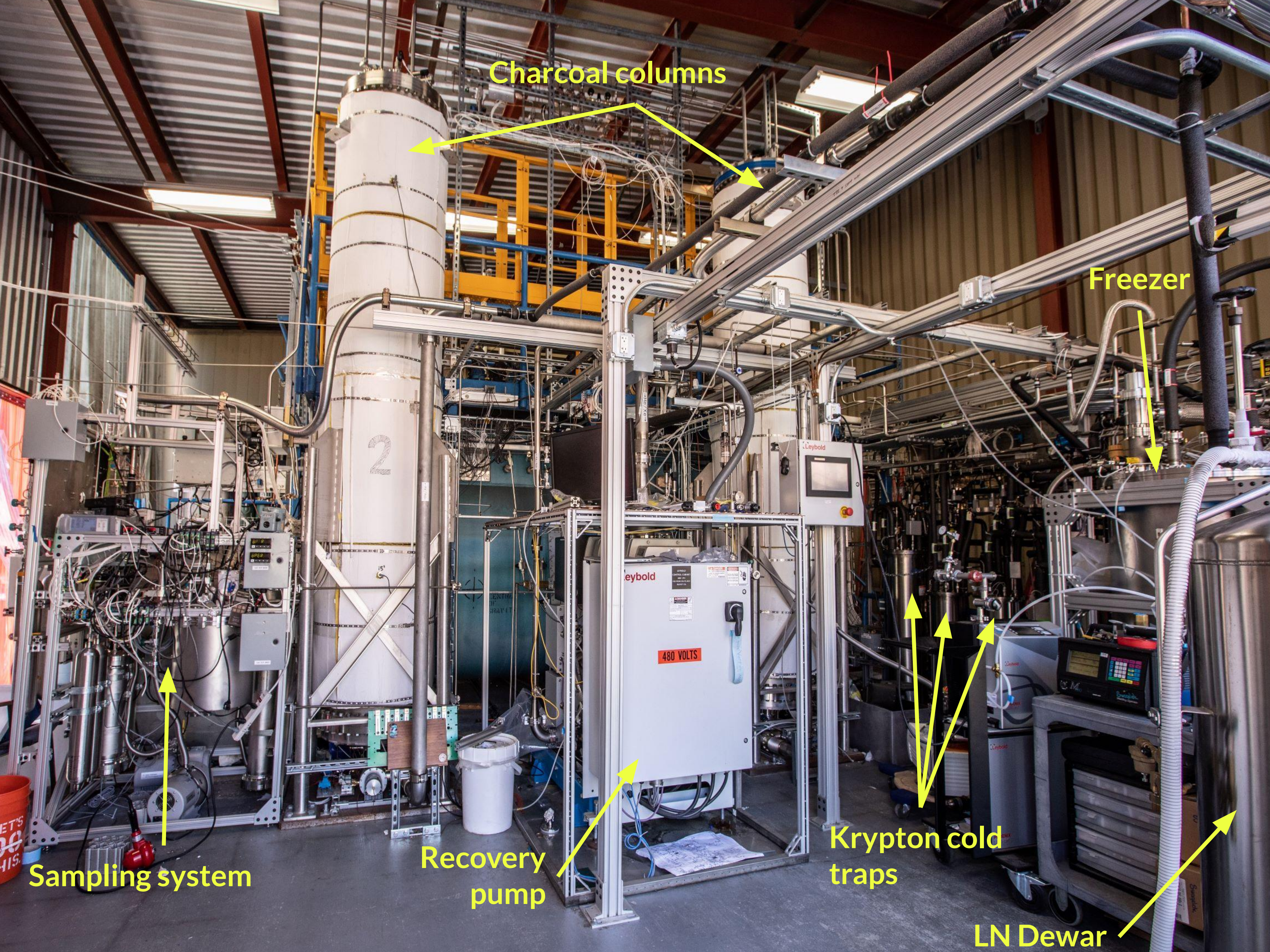
3. **Storage:**
   Xe compressed into cylinders for transport



Expanded system diagram

- 2 charcoal columns → parallel processing: One column in chromatography phase, one in recovery
- Multiple Kr traps
- Connections to sampling system at recovery pump and storage line




Charcoal columns
Freezer
Sampling system
Recovery
pump
Krypton cold
traps
LN Dewar
2
480 VOLTS
Leybold

Chromatography loop

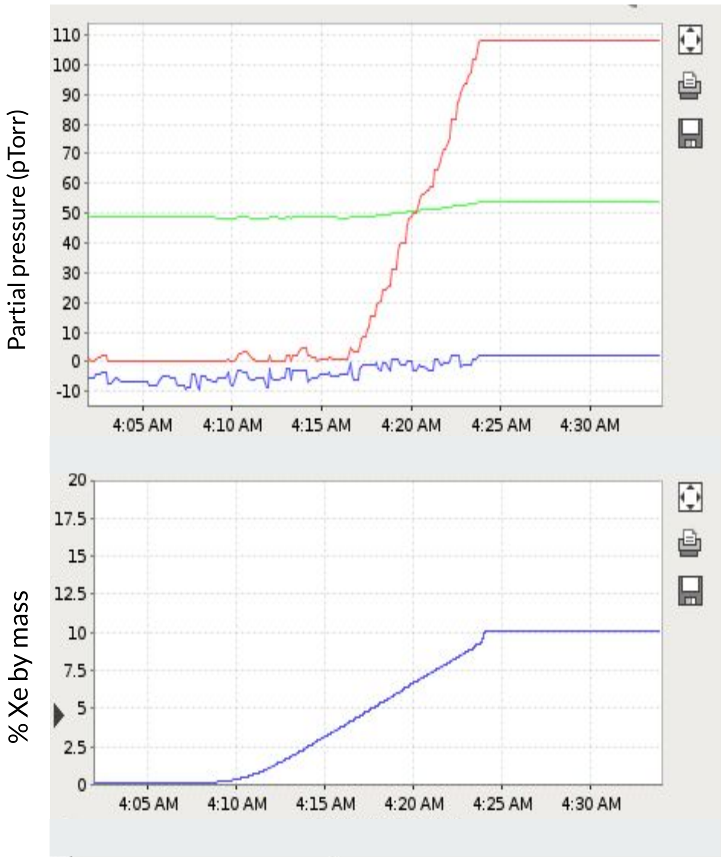
Charcoal columns

Total cycle duration: ~3 hours
Charcoal mass: ~500 kg/column

1. ~16 kg xenon is fed into the column
2. Helium is circulated through the column as a carrier gas
   - ~600 - 1200 SLPM helium flow, adjusted based on ambient temperature to maintain 3h chromatography duration
   - Column pressure is maintained at 1.4 bar
3. A cold trap captures the krypton as it exits the column
4. When the first xenon is detected emerging from the column, circulation is stopped.

Detecting the end of chromatography

The composition of the gas exiting the column is monitored by 2 devices:

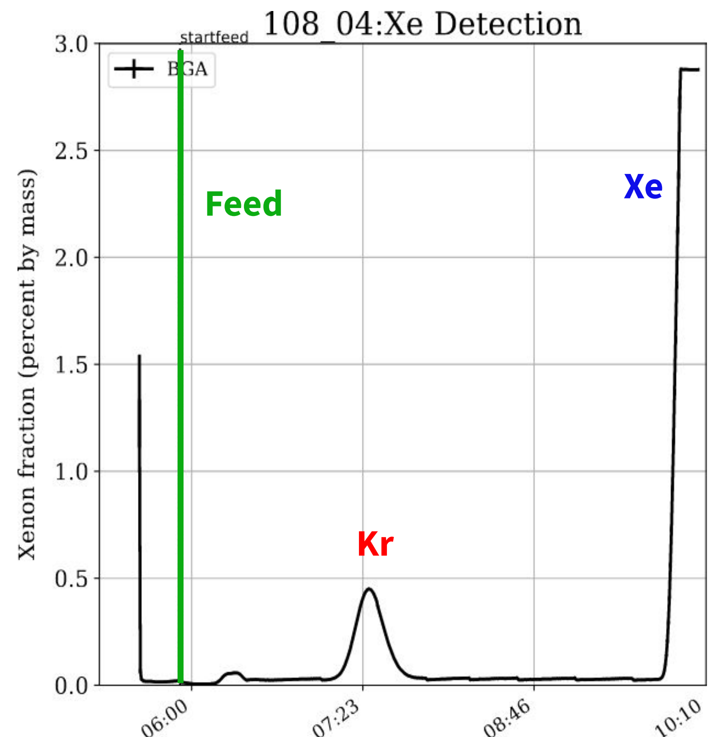


Residual Gas Analyzer (RGA)

- Mass spectrometer
- Sensitive to multiple gases
- Noisy, requires change detection algorithm

Binary Gas Analyzer (BGA)

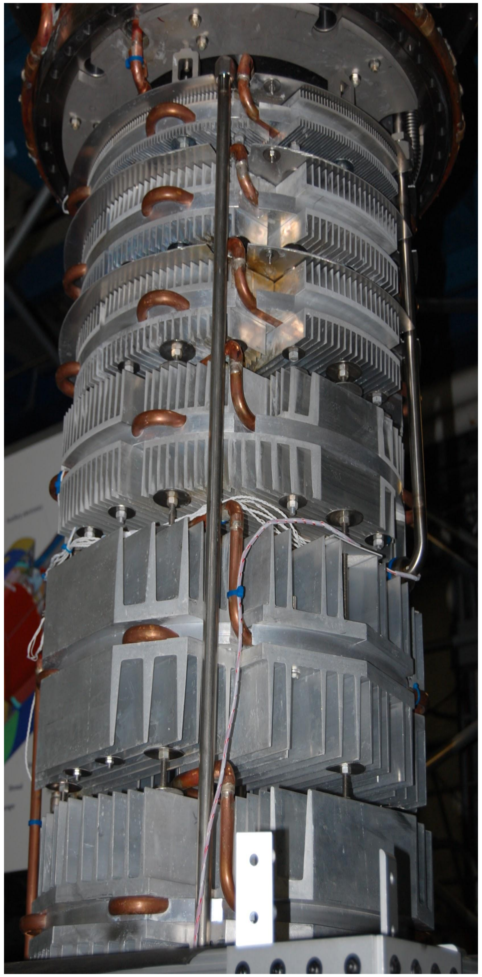
- Uses sound speed to analyze mixtures of 2 gases
- Much lower noise



BGA trace for one full chromatography cycle, showing Kr peak and rising Xe edge (600 ppm calibration xenon - Kr peak is not normally visible)

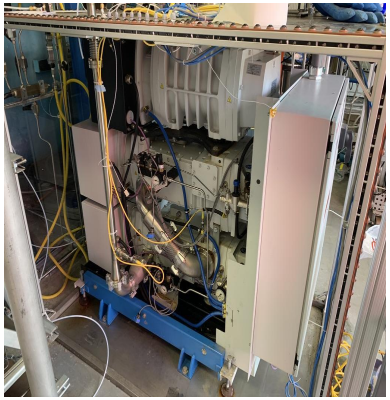
Recovering purified xenon

1. Column is pumped down from ~1.4 bar to 10 mbar - most efficient pressure for recovery due to high volume flow
2. Helium circulates in recovery loop; vacuum pump maintains column at 10 mbar, freezer at 800 - 1000 mbar
3. Recovered xenon freezes onto the collection plates of the freezer
4. Recovery ends when xenon content of gas at the column outlet falls below threshold



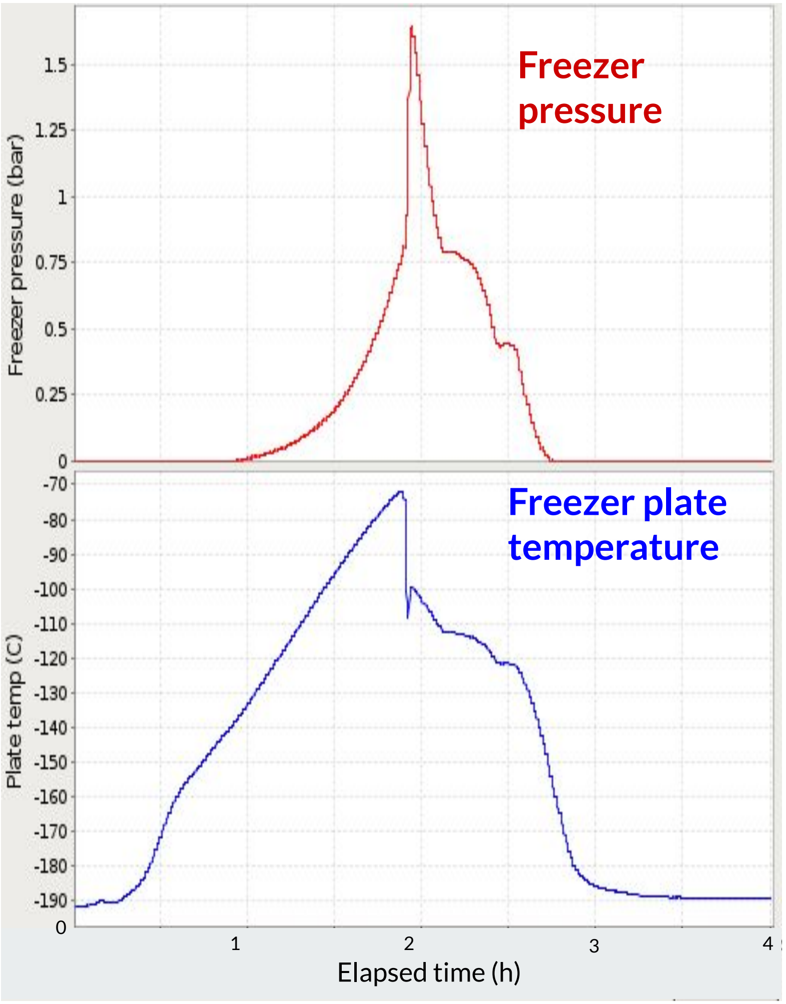
LN lines (copper) run through collection plates (aluminum) in the freezer interior

Cycle duration: 2.5-3h
Freezer capacity: ~200 kg
(~12 recovery cycles)



3 stage vacuum pump maintains columns at 10 mbar

Helium reduction



- When freezer is at capacity, helium must be pumped out prior to storing the xenon
- He can damage PMTs -- require **< 200 ppb** for LZ PMT lifetime
- Pumping on Xe ice for hours leaves O(1ppm) residual helium
- Solution: liquefy the xenon to release entrained helium, then refreeze and pump it away
- Result: O(10 ppb) He
- Requires precise temperature and pressure control to avoid overpressure of freezer

Storage

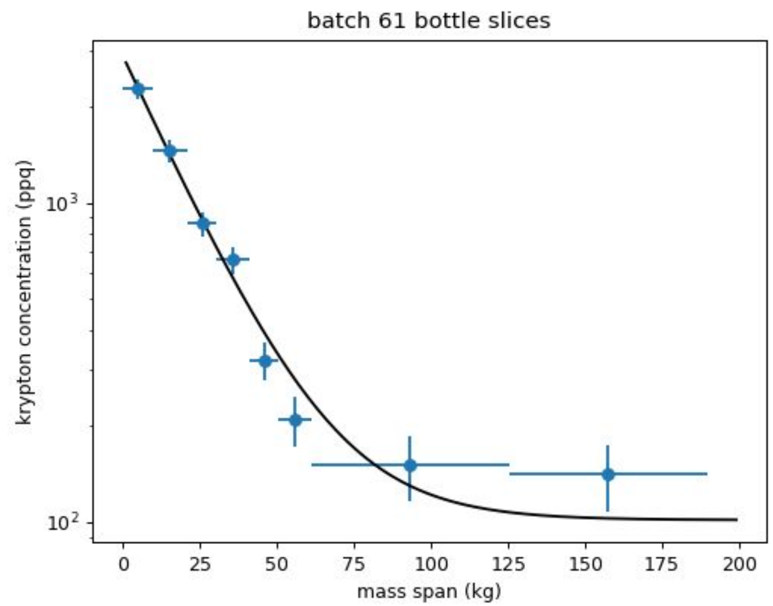
After helium pumpout process, xenon is warmed and compressed into cylinders for transport

Xe storage pack

Storage compressor

Distillation during storage: the first xenon out of the freezer consistently had the highest krypton concentration

We used this to selectively reprocess the “dirtiest” xenon by segregating the first ~50 kg of a batch

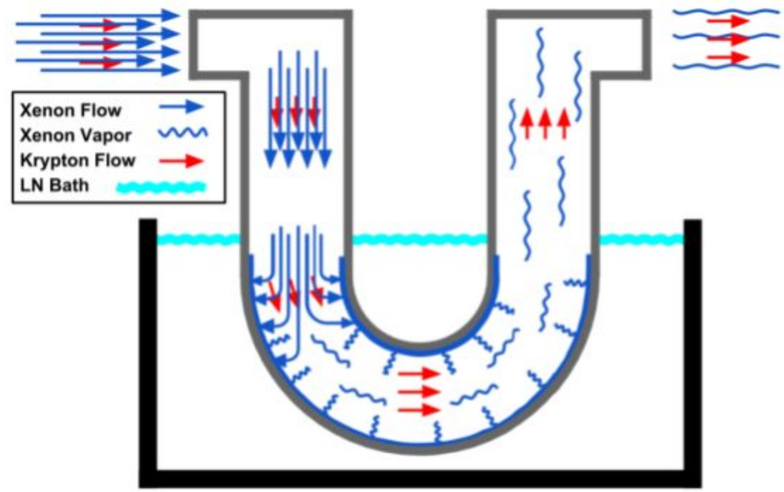


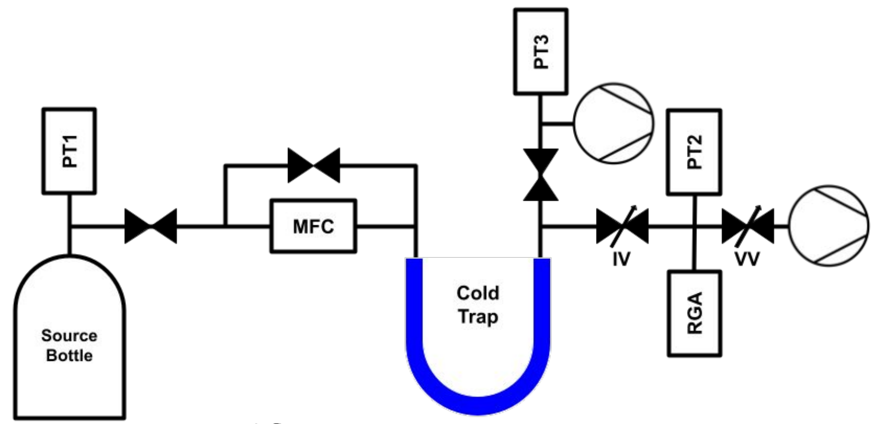
Krypton concentration vs cumulative mass stored for one freezer batch

Sampling System

Measurement technique produces ~10^9 gain in sensitivity to krypton

- Xenon samples (100g) are taken from recovery loop or storage bottles
- Pass through LN temperature cold trap
- Xenon vapor is enriched with impurities
- RGA monitors cold trap output
    - Off the shelf has ~1 ppm (10^{-6}) sensitivity
    - This technique: sensitive to tens of ppq, 10^{-15}





Automated processing

- Fully automated xenon feed, chromatography and recovery cycles
- Runs without human intervention for a full freezer batch (~200 kg, 3-4 days), swapping between chromatography and recovery cycles every 3 hours
- Automatically prepares freezer for storage once capacity is reached, including warmup and refreeze to release helium
- Storage (operation of the compressor) was the only process that required human control during normal operations

Kr removal system PLC

Automated processing

Built on a combination of PLC (programmable logic controller) routines and slow control scripting:

PLC

- Low level controls (operation of valves, instruments, sensor readout)
- Reliable - handled all interlocks related to xenon and equipment safety
- Individual, discrete operations (e.g. xenon feed, chromatography & recovery circulation, transitions between states) are handled by PLC routines

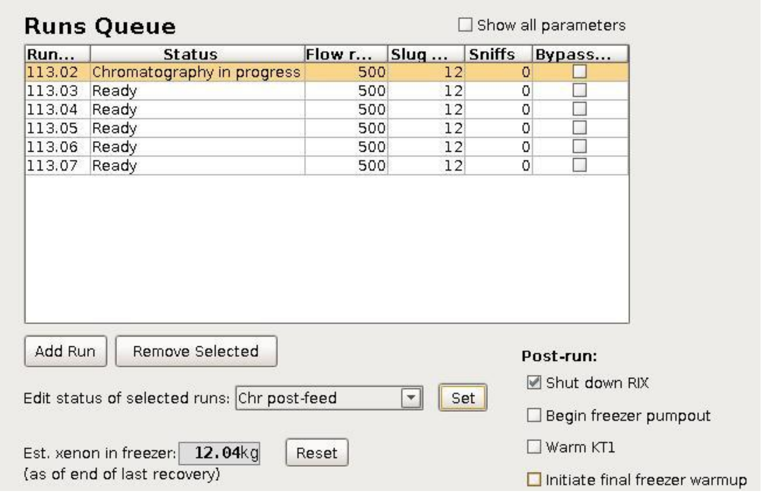
Slow control

- Human interface to PLC, view long term trends in sensor data
- Python scripting in Ignition (slow control software) coordinates PLC routines for longer-term operation
- Alarms alert operators to sensors deviating from normal ranges

Ignition run control infrastructure

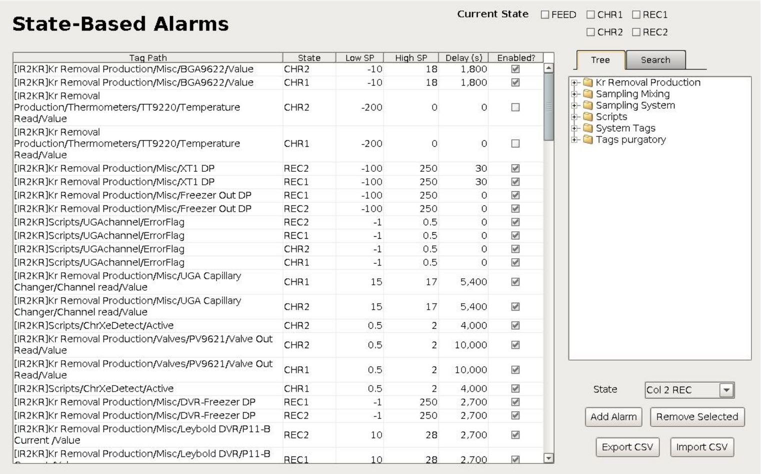
Run control

- Coordinates parallel operations in both columns
- Interface with automation of sampling system for automatic sample taking and processing
- Tracks location and status of each “slug” of Xe; monitors freezer contents
- Triggers preparation for storage when freezer capacity is reached



State-based alarms

- Dynamic alarm levels based on current system status - extends Ignition’s “static” alarm capabilities
- Customizable delays to avoid tripping on transients at the beginning of an operation



Results

10,379 kg total delivered to SURF

Recall: <0.3 ppt g/g required

Final (mass averaged) purity measured at SLAC:

Purity measured after condensing into LZ:

Purified Xe underground at SURF

Results

10,379 kg total delivered to SURF

Recall: <0.3 ppt g/g required

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0.12 ppt

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Purified Xe underground at SURF

Results

10,379 kg total delivered to SURF

Recall: <0.3 ppt g/g required

Final (mass averaged) purity measured at SLAC:

0.12 ppt

Purity measured after condensing into LZ:

0.10 ppt 🎉

Purified Xe underground at SURF

Conclusions

- To meet requirements on ER backgrounds, LZ needs xenon with <0.3 ppt (3×10^{-10}) g/g ^{nat}Kr
- Kr removal system at SLAC employed gas charcoal chromatography to purify 10 tonne LZ xenon payload
- Fully automated chromatography and recovery allowed operation for multiple days without human intervention
- 10.3 tonnes of xenon delivered at an ultimate purity of 0.12 ppt, confirmed by in-situ measurements after filling detector

LZ (LUX-ZEPLIN) Collaboration

35 Institutions: 250 scientists, engineers, and technical staff

@lzdarkmatter

https://lz.lbl.gov/

- Black Hills State University
- Brandeis University
- Brookhaven National Laboratory
- Brown University
- Center for Underground Physics
- Edinburgh University
- Fermi National Accelerator Lab.
- Imperial College London
- Lawrence Berkeley National Lab.
- Lawrence Livermore National Lab.
- LIP Coimbra
- Northwestern University
- Pennsylvania State University
- Royal Holloway University of London
- SLAC National Accelerator Lab.
- South Dakota School of Mines & Tech
- South Dakota Science & Technology Authority
- STFC Rutherford Appleton Lab.
- Texas A&M University
- University of Albany, SUNY
- University of Alabama
- University of Bristol
- University College London
- University of California Berkeley
- University of California Davis
- University of California Los Angeles
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- University of Massachusetts, Amherst
- University of Michigan
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- University of Rochester
- University of Sheffield
- University of Wisconsin, Madison

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Thanks to our sponsors and participating institutions!

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Office of Science

UKRI Science and Technology Facilities Council

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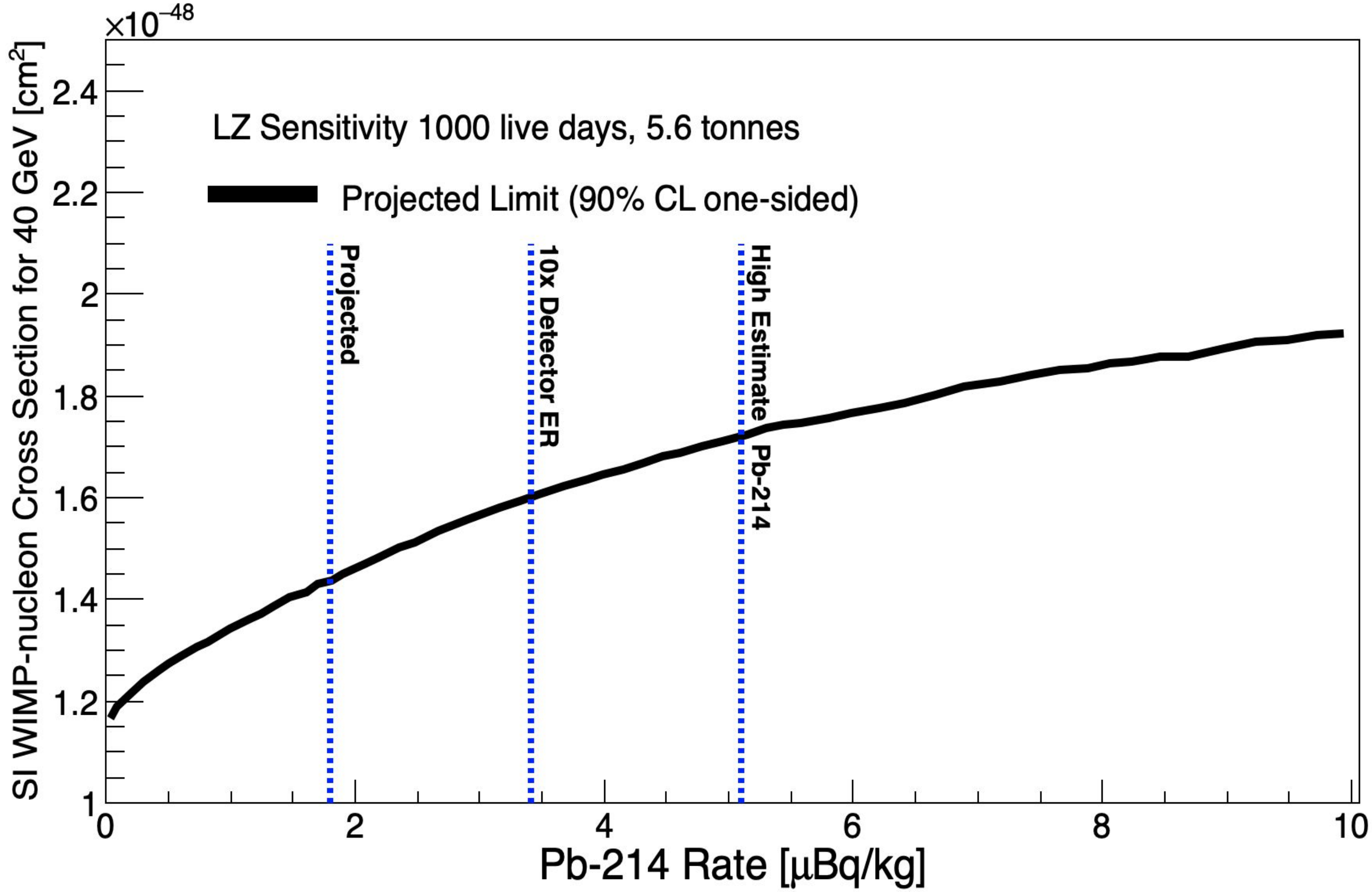
South Dakota Science and Technology Authority

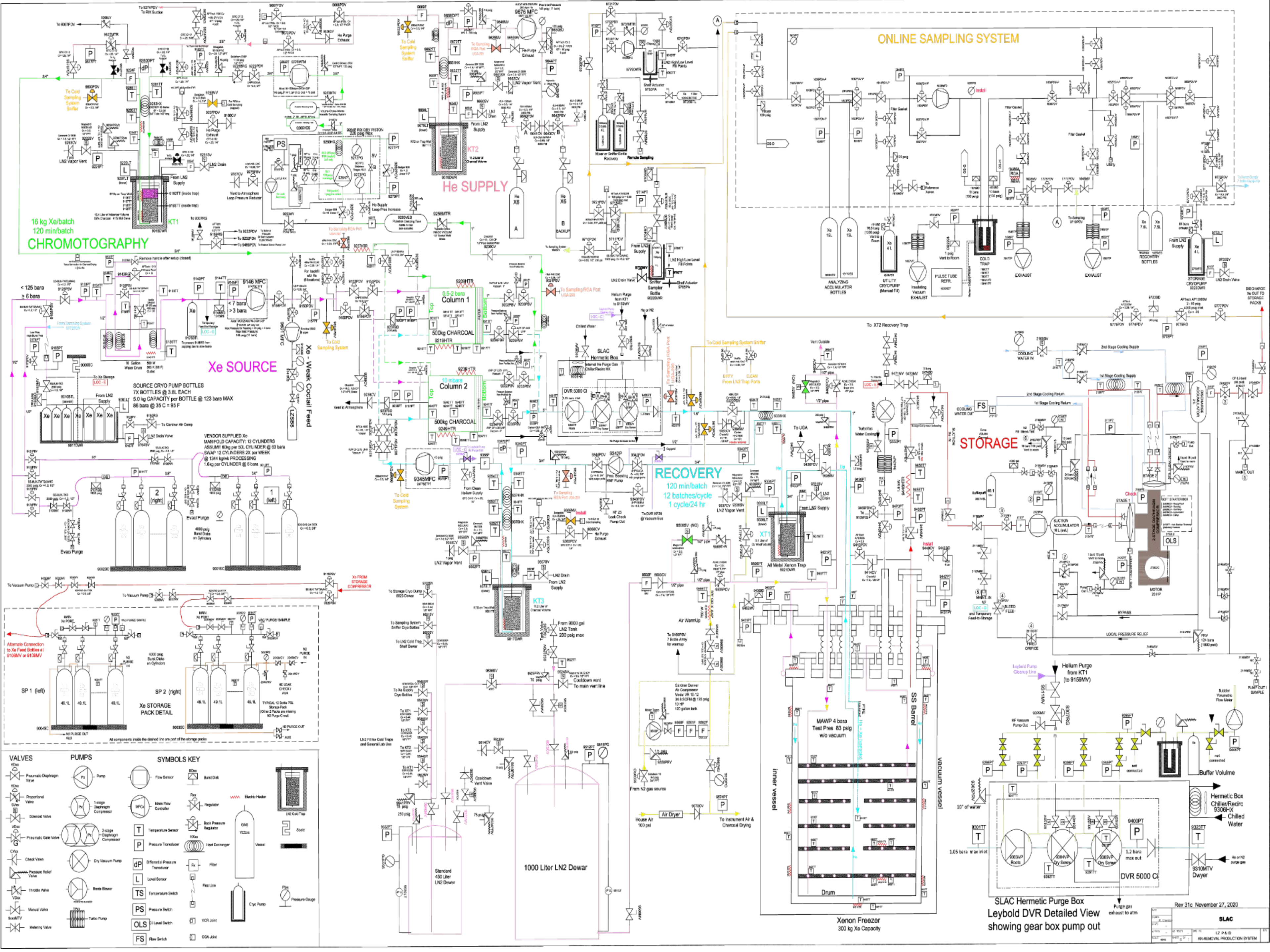
FCT Fundação para a Ciência e a Tecnologia

ibs Institute for Basic Science

Thanks!

Effect on sensitivity of increasing Rn activity




ONLINE SAMPLING SYSTEM
He SUPPLY
CHROMOTOGRAPHY
16 kg Xe/batch
120 min/batch
Xe SOURCE
SOURCE CRYO PUMP BOTTLES
7X BOTTLES @ 3.8L EACH
5.0 kg CAPACITY per BOTTLE @ 123 bara MAX
86 bara @ 35 C = 95 F
VENDOR SUPPLIED Xe
MANIFOLD CAPACITY: 12 CYLINDERS
ASSUME 60kg per 50L CYLINDER @ 63 bara
SWAP 12 CYLINDERS 2X per WEEK
@ 1344 kg/wk PROCESSING
1.6kg per CYLINDER @ 6 bara
Xe - Weak Coaxial Feed
9146 MFC
9676 MFC
9345MFC
Column 1
500kg CHARCOAL
Column 2
500kg CHARCOAL
0.5-2 bara
10 mbara
KT1
KT2
KT3
XT1
RECOVERY
120 min/batch
12 batches/cycle
1 cycle/24 hr
STORAGE
SLAC Hermetic Box
DVR 5000 CI
1000 Liter LN2 Dewar
Standard
450 Liter
LN2 Dewar
MAWP 4 bara
Test Pres 83 psig
w/o vacuum
SS Barrel
inner vessel
vacuum vessel
Drum
Xenon Freezer
300 kg Xe Capacity
SP 1 (left)
SP 2 (right)
Xe STORAGE
PACK DETAIL
Evac/Purge
VALVES
PUMPS
SYMBOLS KEY
Buffer Volume
Hermetic Box
Chiller/Recirc
9306HX
Chilled
Water
Helium Purge
from KT1
(to 9159MV)
SLAC Hermetic Purge Box
Leybold DVR Detailed View
showing gear box pump out
1.05 bara max inlet
1.2 bara max out
Rev 31c November 27, 2020
SLAC
LZ P&ID