

Purifying xenon from radioactive noble gases

International Workshop on Applications of Noble Gas Xenon to Science and Technology
XeSAT2023, Nantes, June 5-9, 2023

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* member of XENON, DARWIN/XLZD, technical member of nEXO

- Introduction
- Need to purify xenon (argon) from radioactive noble gases
- Kr (Ar) removal with charcoal chromatography and online cryogenic distillation
- Rn online removal with charcoal chromatography and cryogenic distillation
- Goal of ERC Advanced Grant „LowRad“
- Conclusions

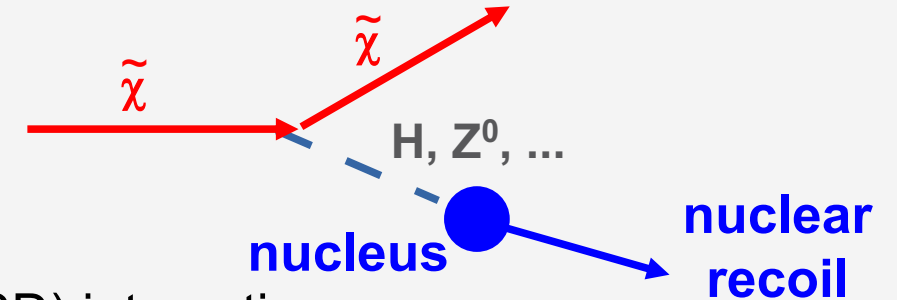


Search for nuclear recoil: energy transfer to nucleus by invisible particle

Elastic coherent scattering of the nucleus

mediated by a spin 0 (Higgs-like) or spin 1 (Z-like) propagator

Effectively a coherent spin-independent (SI) or spin-dependent (SD) interaction:



But two generic problems:

- very low rate

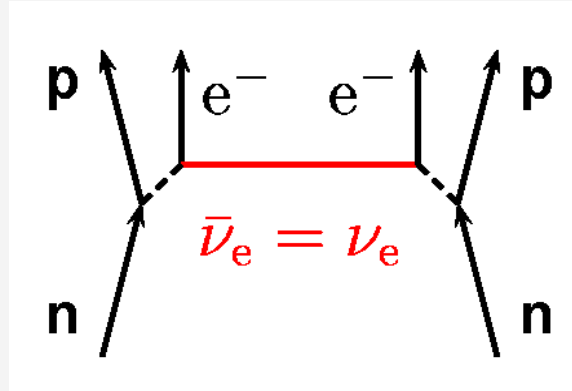
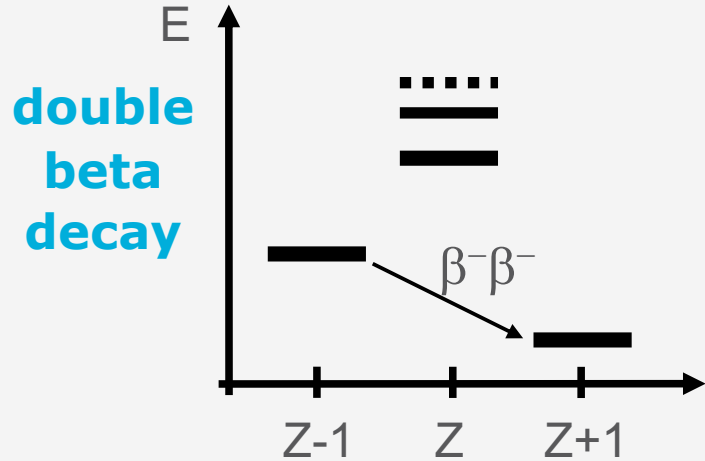
- go deep underground to avoid cosmic rays & secondary muons
& ultra-clean materials w.r.t. radioactivity
& double read-out (light+charge or heat+charge or heat+light)

- very low recoil energy

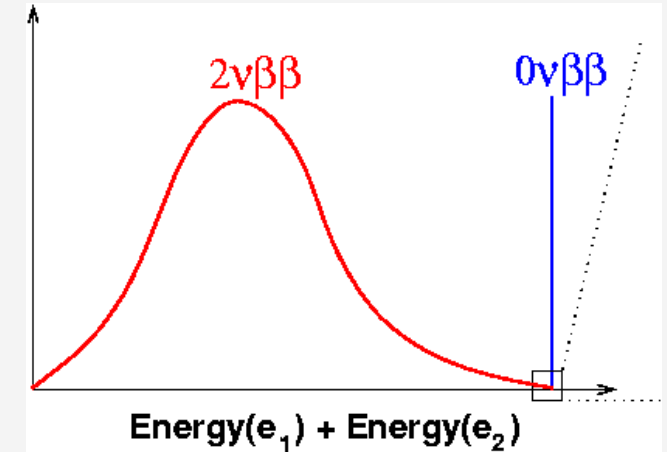
- ultra-sensitive detector



Experimental search for neutrinoless double beta decay



neutrinoless ($0\nu\beta\beta$)



Signature:

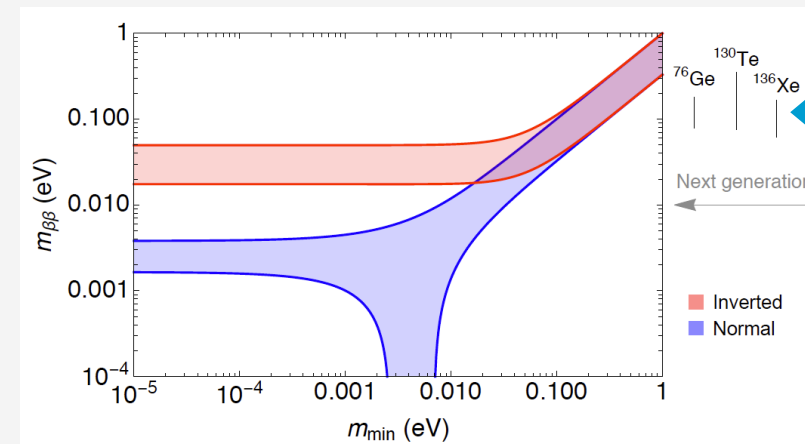
sum of both electron energies
peak sharply at Q-value

Search for $0\nu\beta\beta$: $m_{\beta\beta} := |\sum_i U_{ei}^2 \cdot m(\nu_i)|$

sensitive to Majorana neutrinos only, challenge of
uncertainties of nuclear matrix elements

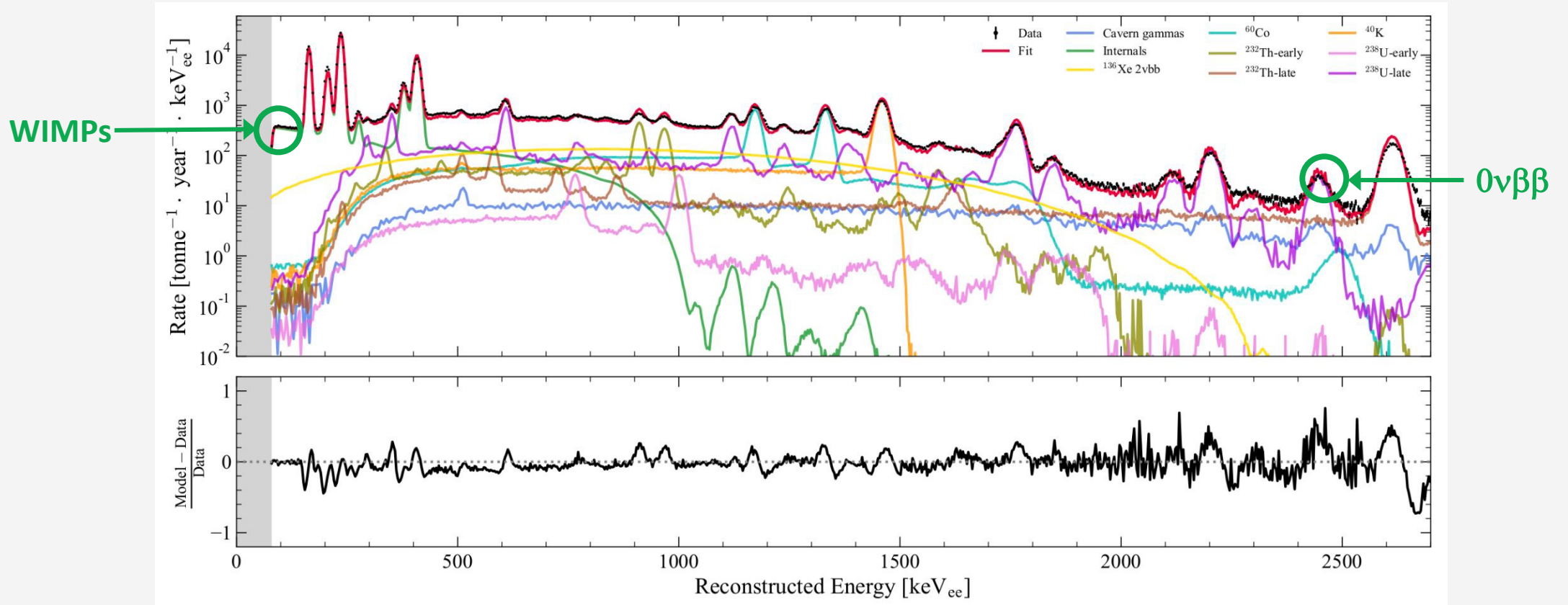
disclaimer: $m_{\beta\beta}$ are valid only, if $0\nu\beta\beta$ works dominantly via ν exchange

Discovery of $0\nu\beta\beta$ would proof lepton number violation !



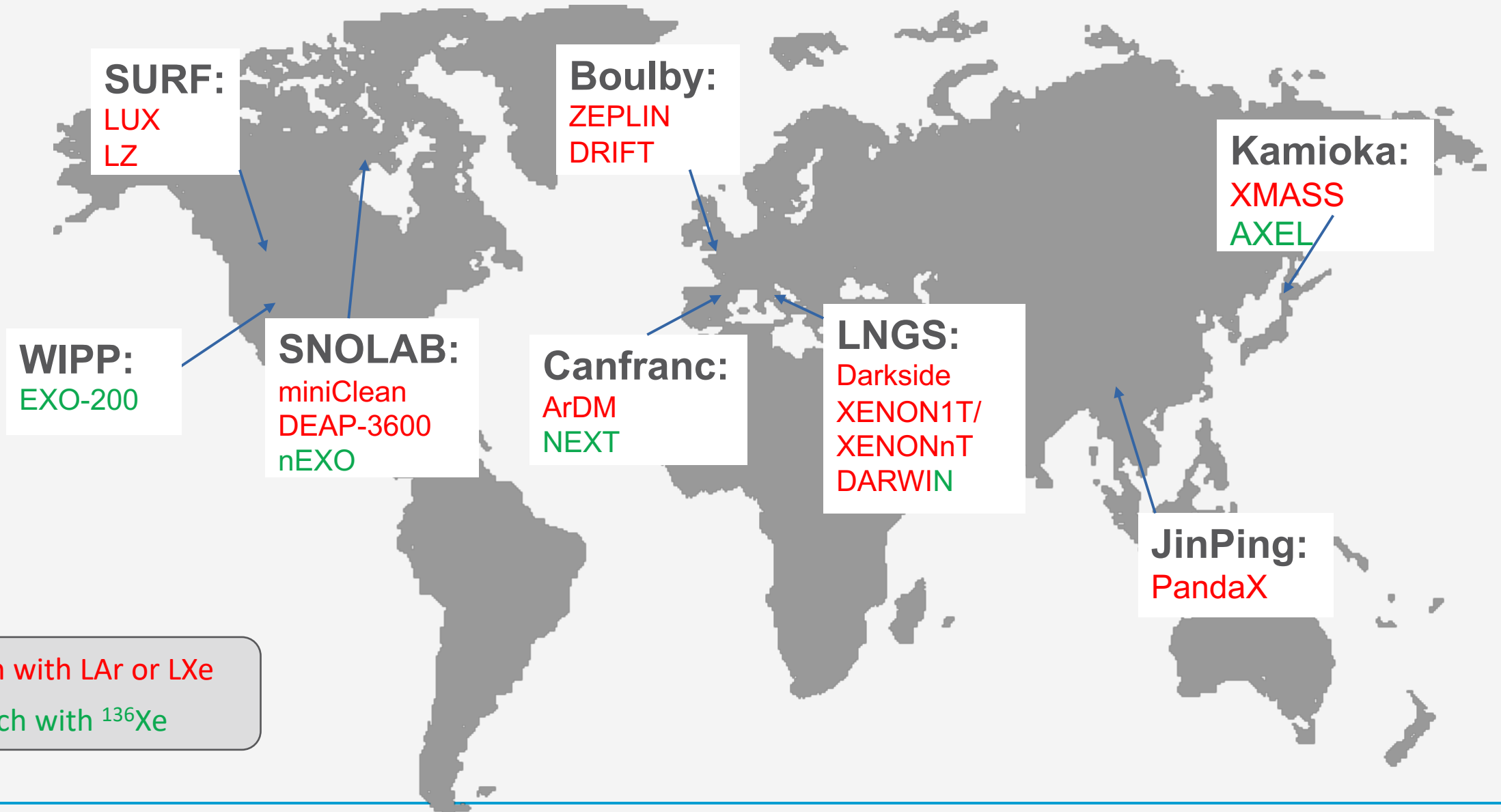
$T_{1/2} \approx 10^{26}$ yr

WIMPs and $0\nu\beta\beta$: rather different energy scales - different requirements as well?



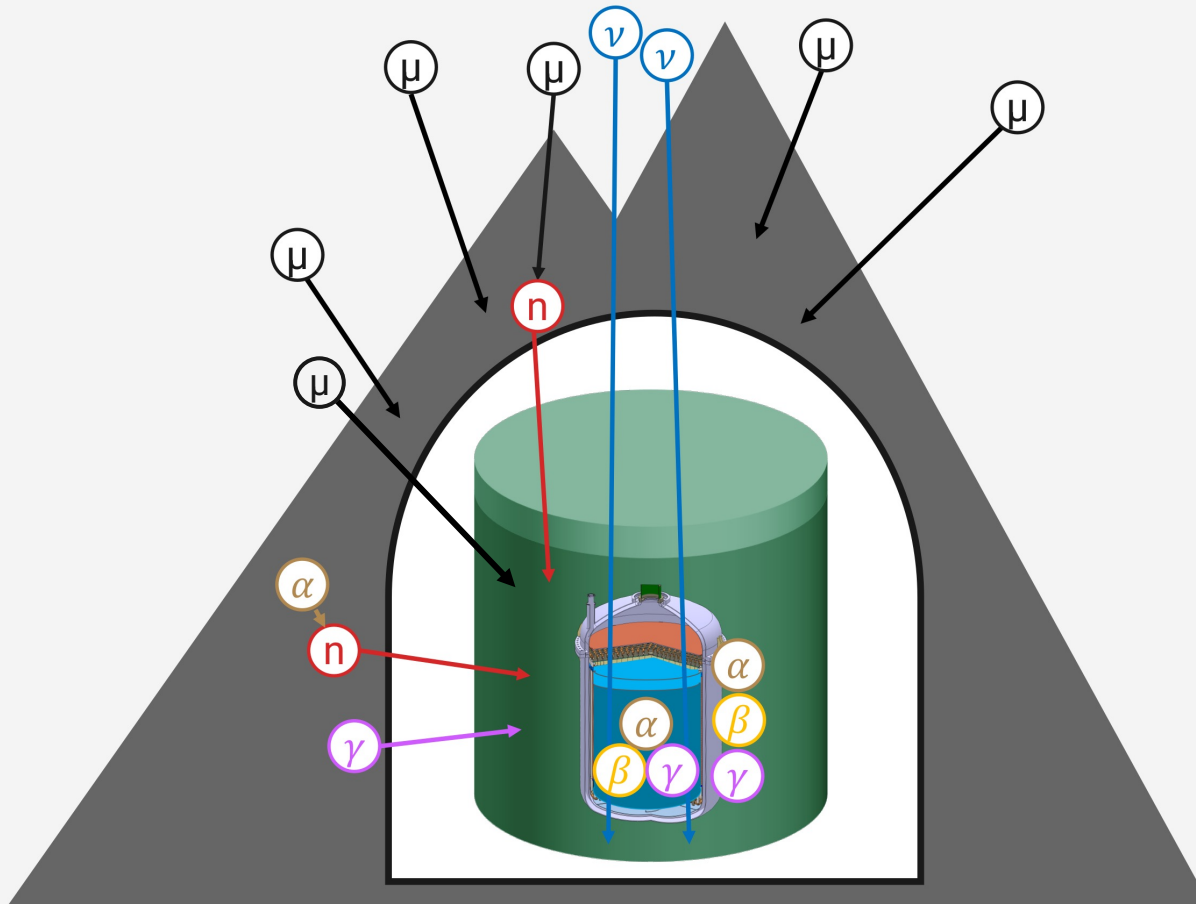
from LZ, see talk by P. Brás at XeSAT2023

Direct Cold Dark Matter and $0\nu\beta\beta$ searches with noble gases



Limiting backgrounds in noble gas DM detectors

cosmic radiation



Expected dark matter scattering rate:

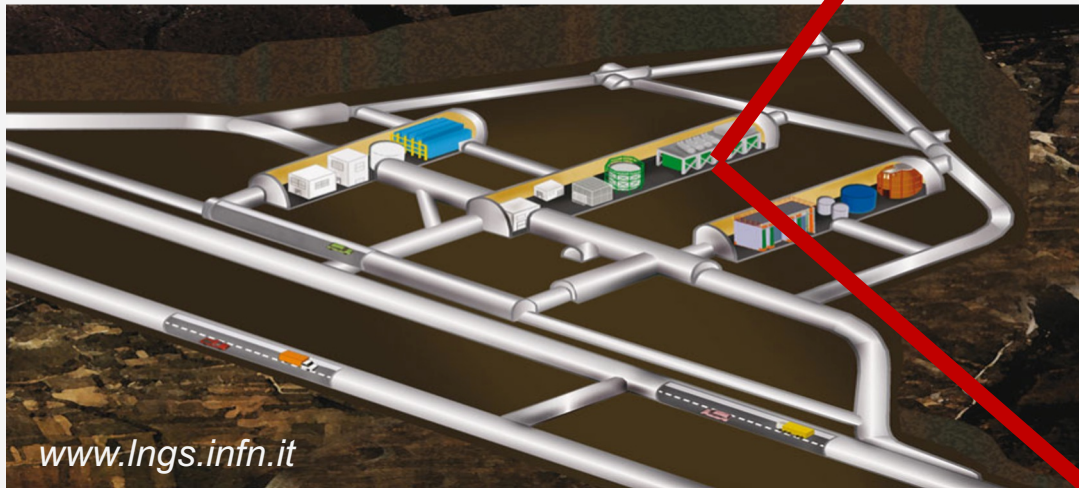
1 event per 10t and year

**⇒ Profit only from larger experiments
if the experiment remains background-free**

Most background problems solved by

- going underground
- extra shieldings & vetos

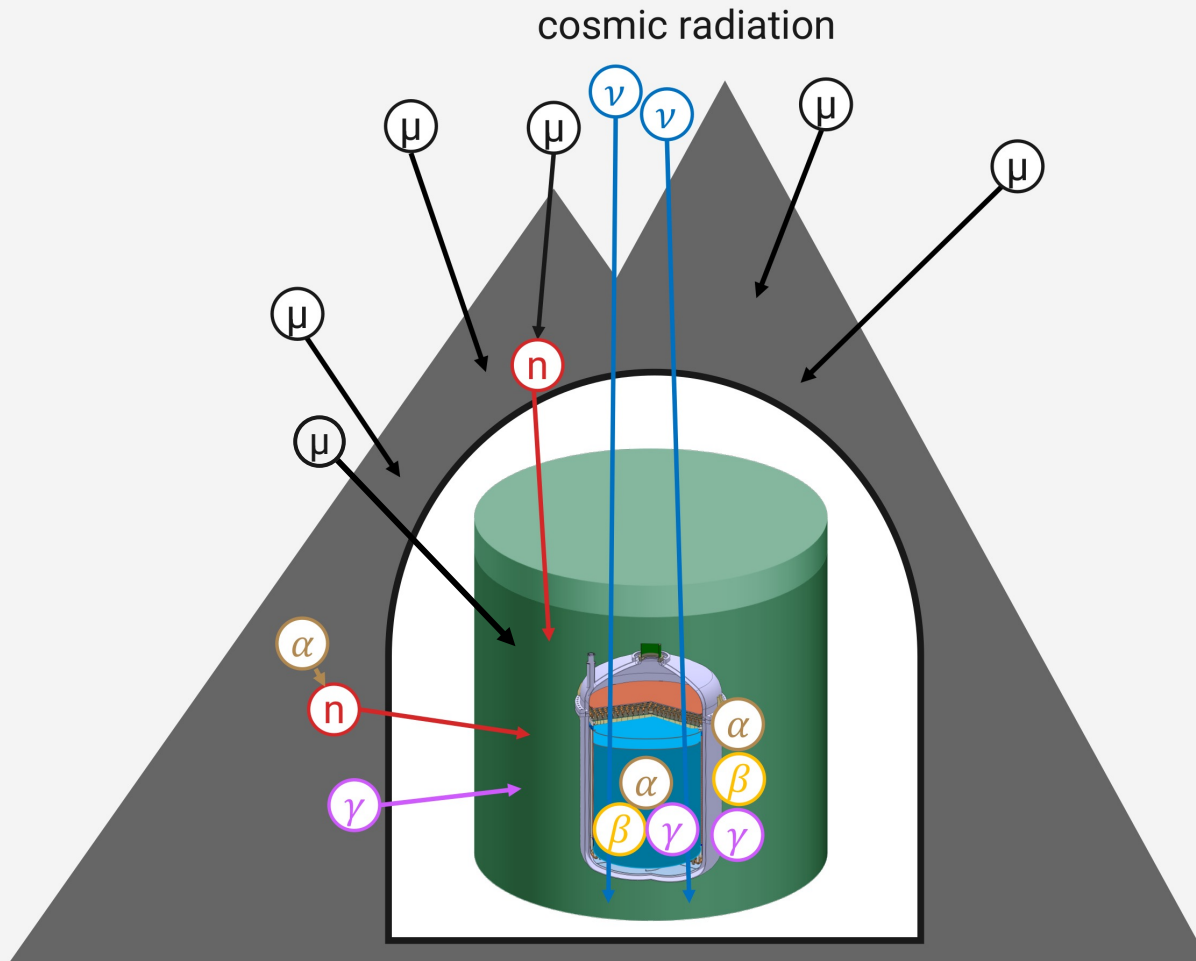
Direct search for dark matter XENON1T/nT @Laboratori Nazionali Gran Sasso (LNGS)



Laboratori Nazionali del Gran Sasso (LNGS), Italy



Limiting backgrounds in noble gas DM detectors



Expected dark matter scattering rate:
1 event per 10t and year

⇒ Profit only from larger experiments
if the experiment remains background-free

Most background problems solved by

- going underground
- extra shieldings & vetos

Two remaining backgrounds:

- solar neutrinos, non-shieldable
- **intrinsic radioactive noble gases:**
 ^{85}Kr , ^{222}Rn and progenies, (^{37}Ar , ^{39}Ar , ^{136}Xe)

Dual Phase Xenon Time Projection Chamber

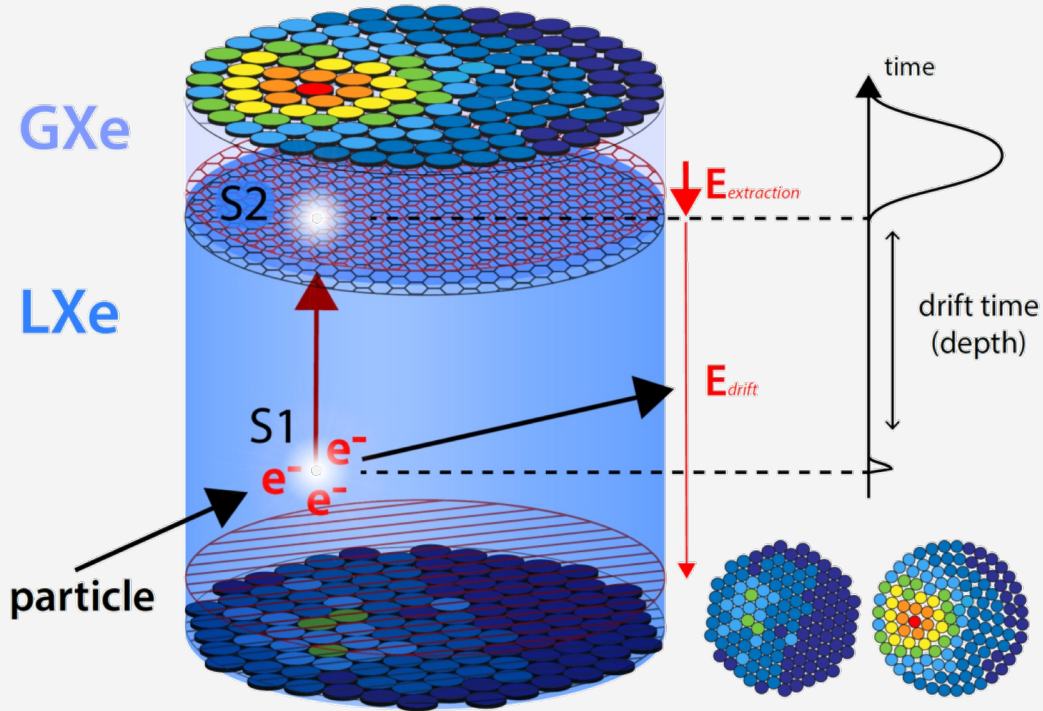


Image by L. Althüser

S1 light signal:

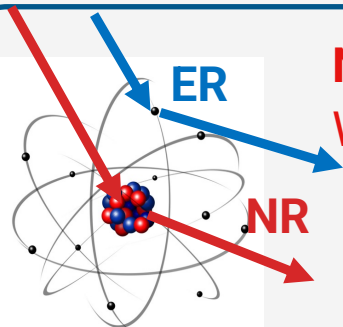
- prompt scintillation photons

S2 charge signal:

- secondary scintillation photons from electroluminescence in GXe due to drifted electrons

3D vertex reconstruction (-> fiducialisation):

- X,Y: S2 hit pattern
- Z: drift time S2-S1



NR (Nuclear Recoils)

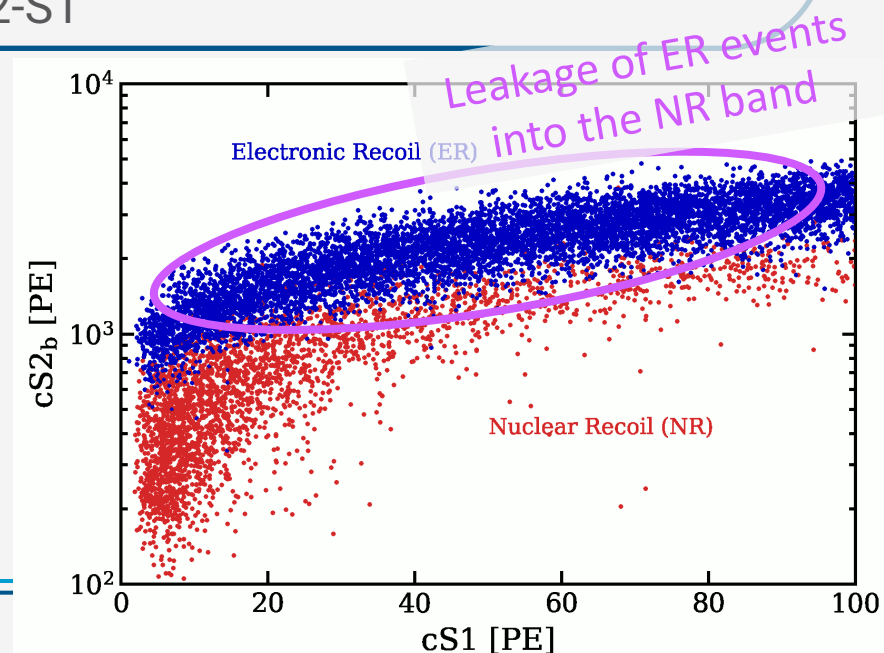
WIMP signal, neutrons, CEvNs

ER (Electronic Recoils)

γ , β backgrounds

Discrimination from S2/S1

Larger for ER than NR

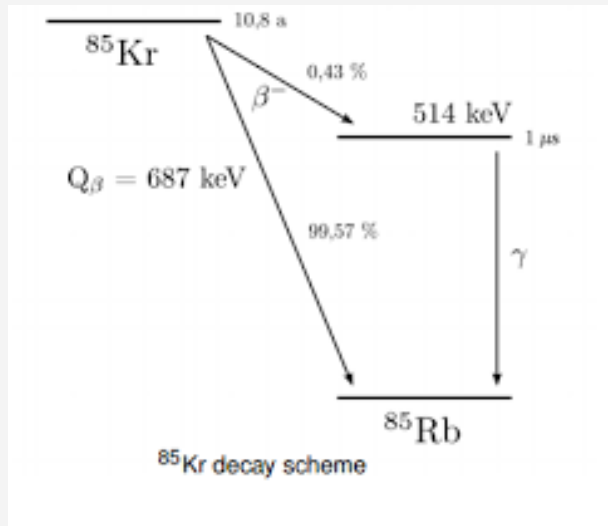


A closer look to ^{85}Kr and ^{222}Rn with its progenities

Why: intrinsic noble gas contaminants ^{85}Kr and ^{222}Rn (\rightarrow ^{214}Pb) (as well as calibrating isotopes, e.g. ^{37}Ar)

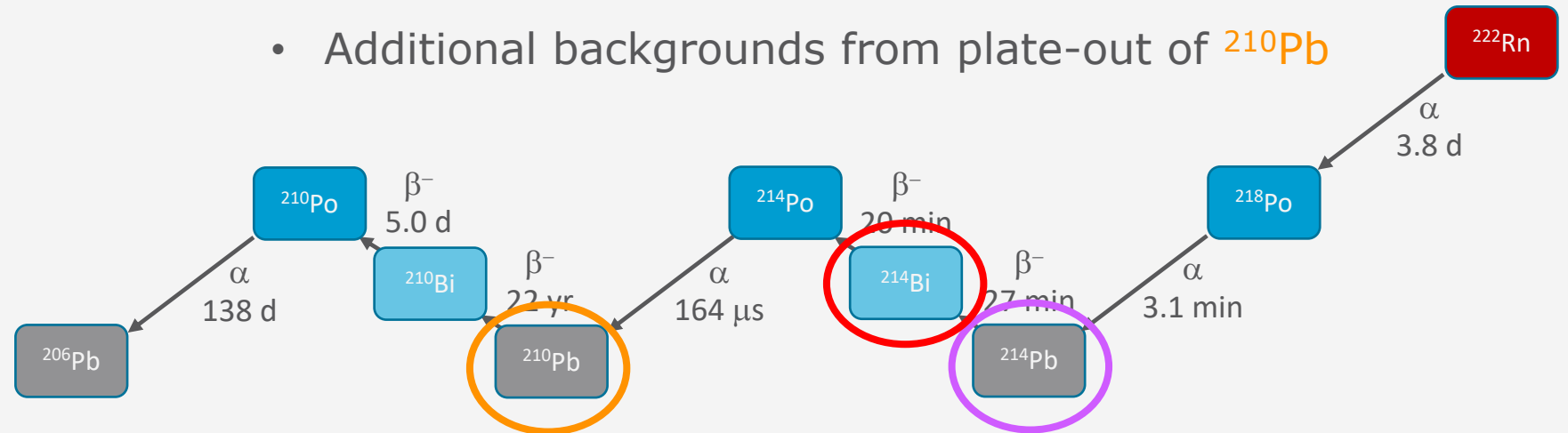
- leakage events from the low energy β -spectrum contaminate ROI for dark matter WIMP search
- searches for new physics inside the electronic recoil spectrum only possible with low levels of impurities

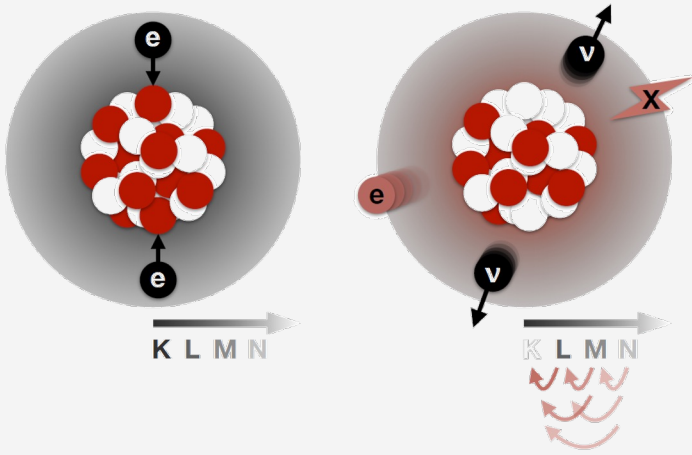
^{85}Kr : $1 - 2 \cdot 10^{-11}$ in $^{\text{nat}}\text{Kr}$, man-made



^{222}Rn : $t_{1/2} = 3.8$ d, continuously emanating from detector materials,

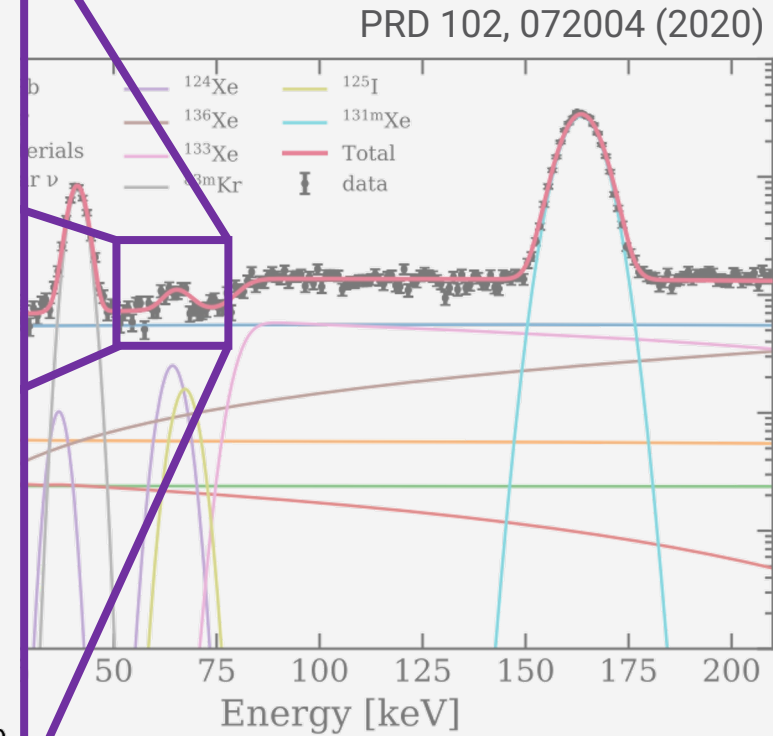
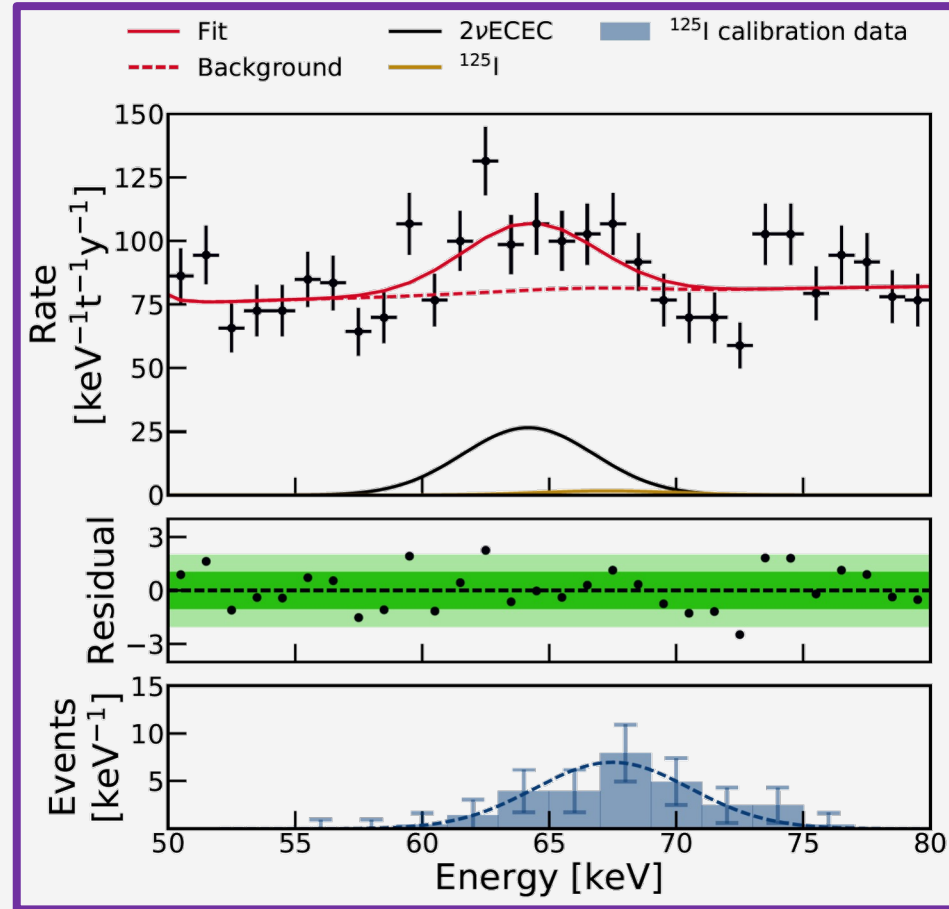
- Background from β -decay of ^{214}Pb which cannot be identified by accompanying α -decay
- Background from γ -decay of ^{214}Bi for $0\nu\beta\beta$ decay searches if not fully BiPo-tagged
- Additional backgrounds from plate-out of ^{210}Pb





Double K-shell capture

- X-ray + Auger electrons create line at 64.3 keV



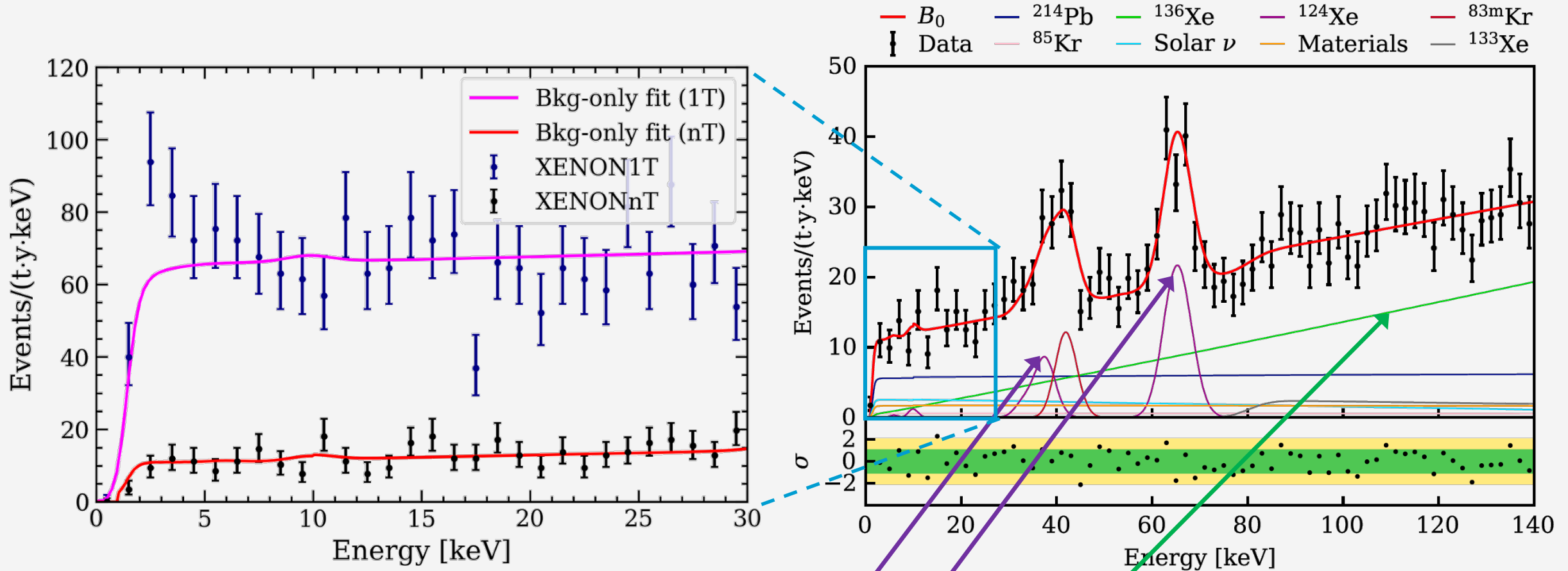
$$T_{1/2}^{KK} = (1.8 \pm 0.5_{\text{stat}} \pm 0.1_{\text{sys}}) \times 10^{22} \text{ yr}$$

Nature 568, 532-535 (2019)

Main authors: A. Fieguth, C. Wittweg, U Münster



Significantly lower background of XENONnT



Much lower electron recoil background:

- Spectrum dominated by double weak decays (ECEC of ^{124}Xe , $2\nu\beta\beta$ of ^{136}Xe)
- XENON1T excess is gone, was likely tritium contamination, which now got purified

Phys. Rev. Lett. 129, 161805 (2022)

Removal of Intrinsic radioactive contaminants: ^{85}Kr

Intrinsic noble gas contaminants Kr-85 and Rn-222 (Pb-214) inside the xenon

- electronic recoil events from the low energy β -spectrum of these radioactivities contaminate ROI for dark matter WIMP search
- searches for new physics using electronic recoil signals becomes possible when these radioactivities are drastically reduced
- Kr-85 originates from xenon extraction from air and needs to be **removed once** before the dark matter search to $\text{Kr-nat}/\text{Xe} < 0.2$ ppt
- commercial xenon comes with $\text{natKr}/\text{Xe} > 1$ ppb with $^{85}\text{Kr-85} / \text{natKr} \sim 2 \times 10^{-11}$

XENON1T/nT



Cryogenic Distillation
“online” at LNGS

- Initial Kr removal during filling
- Online removal by distilling the GXe inside the cryostat
- Online mode also removes radioactive Ar-37
- $\text{Kr}/\text{Xe} < 0.05$ ppt

LZ



Gas charcoal chromatography “offline” at SLAC

- Xe then shipped to SURF
- Handle Xe of varying initial Kr contamination - multiple passes if necessary
- $\text{Kr}/\text{Xe} = 0.14$ ppt

PandaX-4T



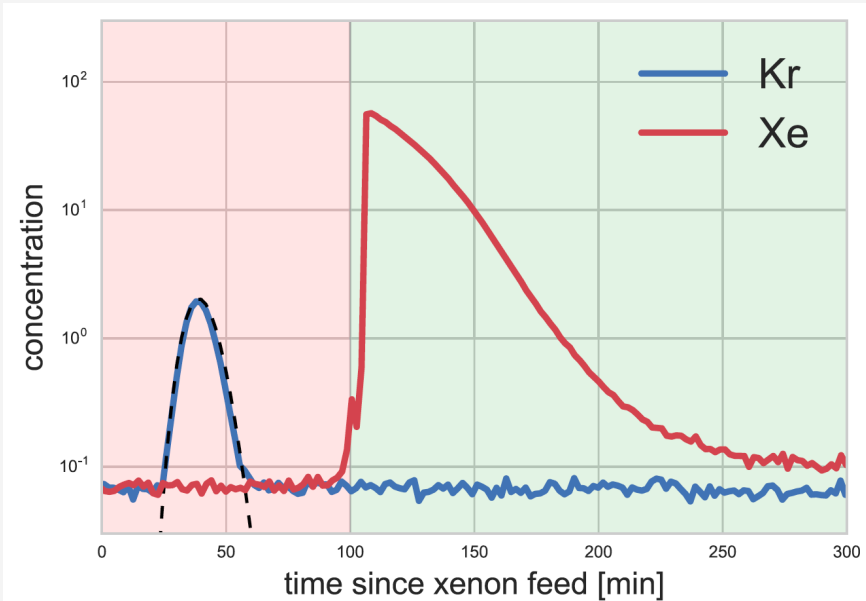
X. Cui et al 2021 JINST 16 P07046

Cryogenic Distillation “online” at CJPL

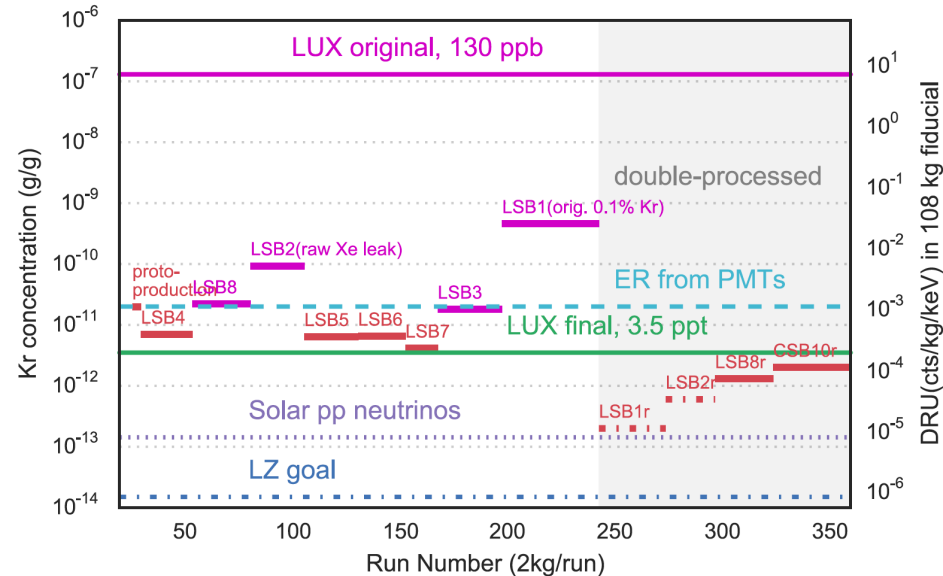
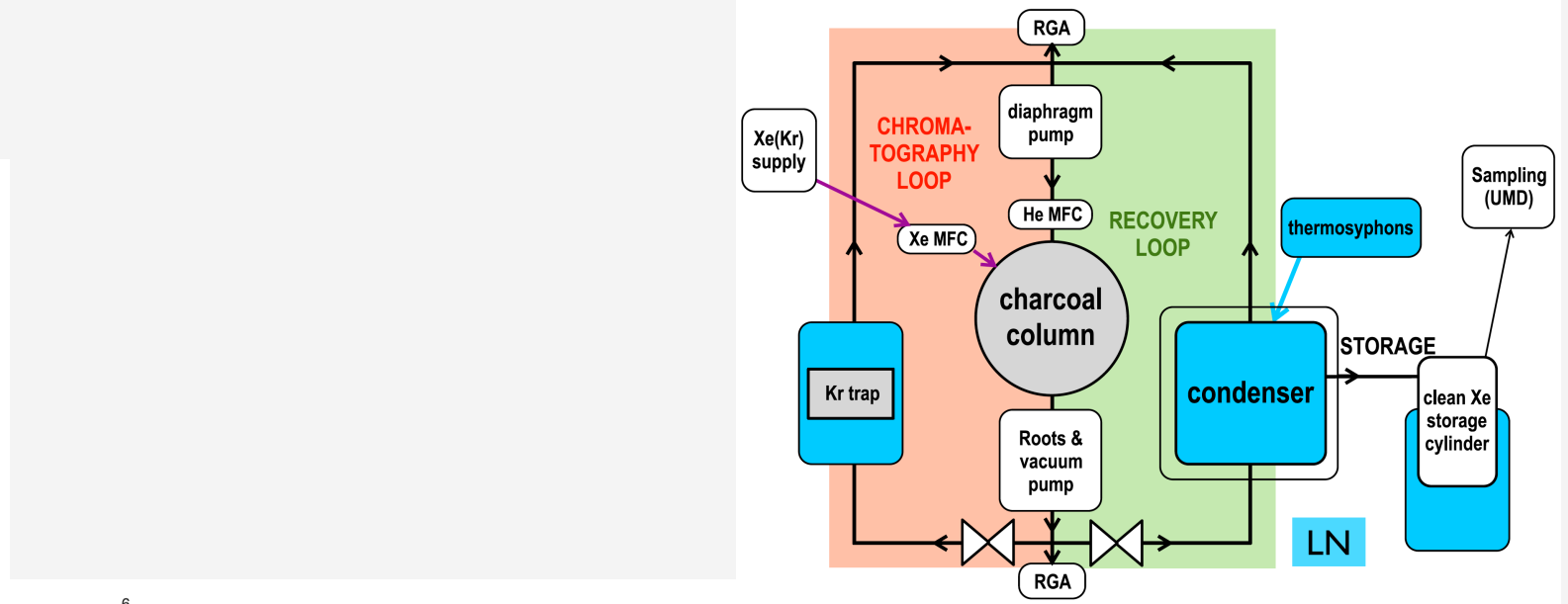
- Initial Kr removal during filling
- Online removal by distilling the GXe inside the cryostat
- Online mode also removes radioactive Ar-37
- $\text{Kr}/\text{Xe} = 0.33$ ppt

courtesy:
Elena Aprile

^{85}Kr removal by charcoal chromatography for LZ before the start of the experiment



J. Albers et al., arXiv:2211.17120



Cryogenic distillation for removing noble gas contaminants like ^{85}Kr , ^{222}Rn (and ^{37}Ar , ^{39}Ar)

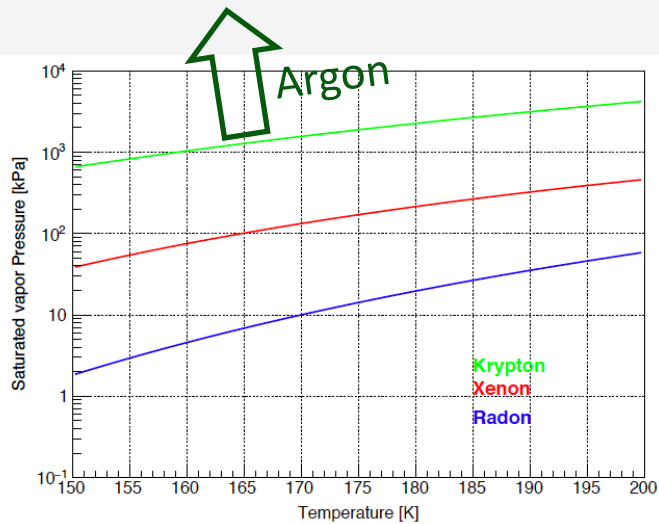
How: Making use of the different vapor pressure of the different noble gas elements

- brought into our field by **XMASS** for Kr removal:

Astropart. Phys. 31, 290-296 (2009).

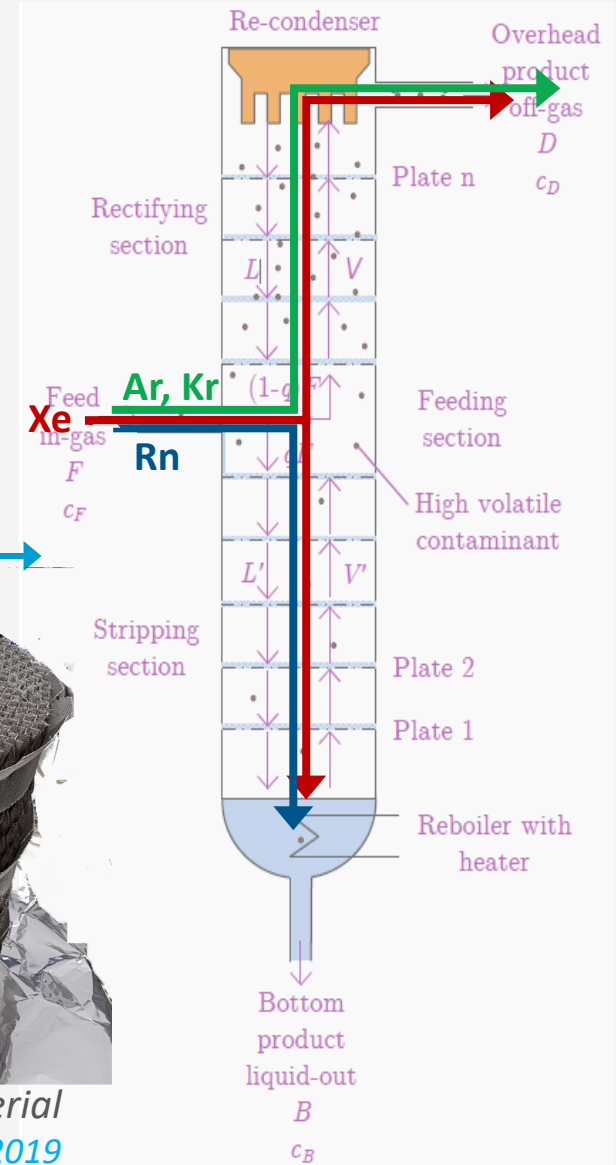
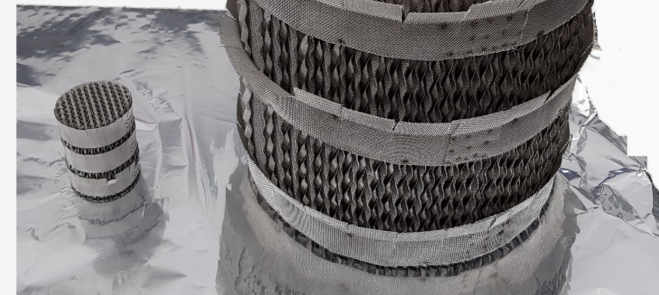
- continued by **XENON** and enhanced to “online” Kr & Rn removal:

EPJ C 77, 277 (2017), EPJ C77 358 (2017), PTEP 2022 (2022) 053H01, EPJ C 82 (2022) 1104



Transition probabilities of single noble gas atoms from gas to liquid and vice versa: saturation vapor pressure

Ø 20cm



X. Cui et al. (PandaX Collaboration, JINST 16 (2021) P0704)

Multi-stage rectification column: in reality continuous package material M. Murra, PhD thesis, University of Münster 2019

Removing Kr by cryogenic distillation at XENON1T/nT



Make use of the different vapour pressures (volatilities) of Kr, Xe and Rn

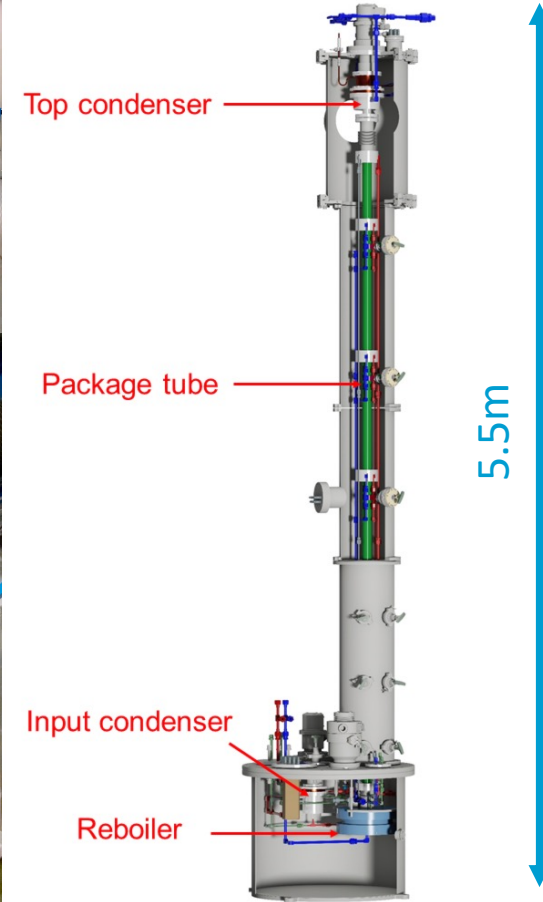
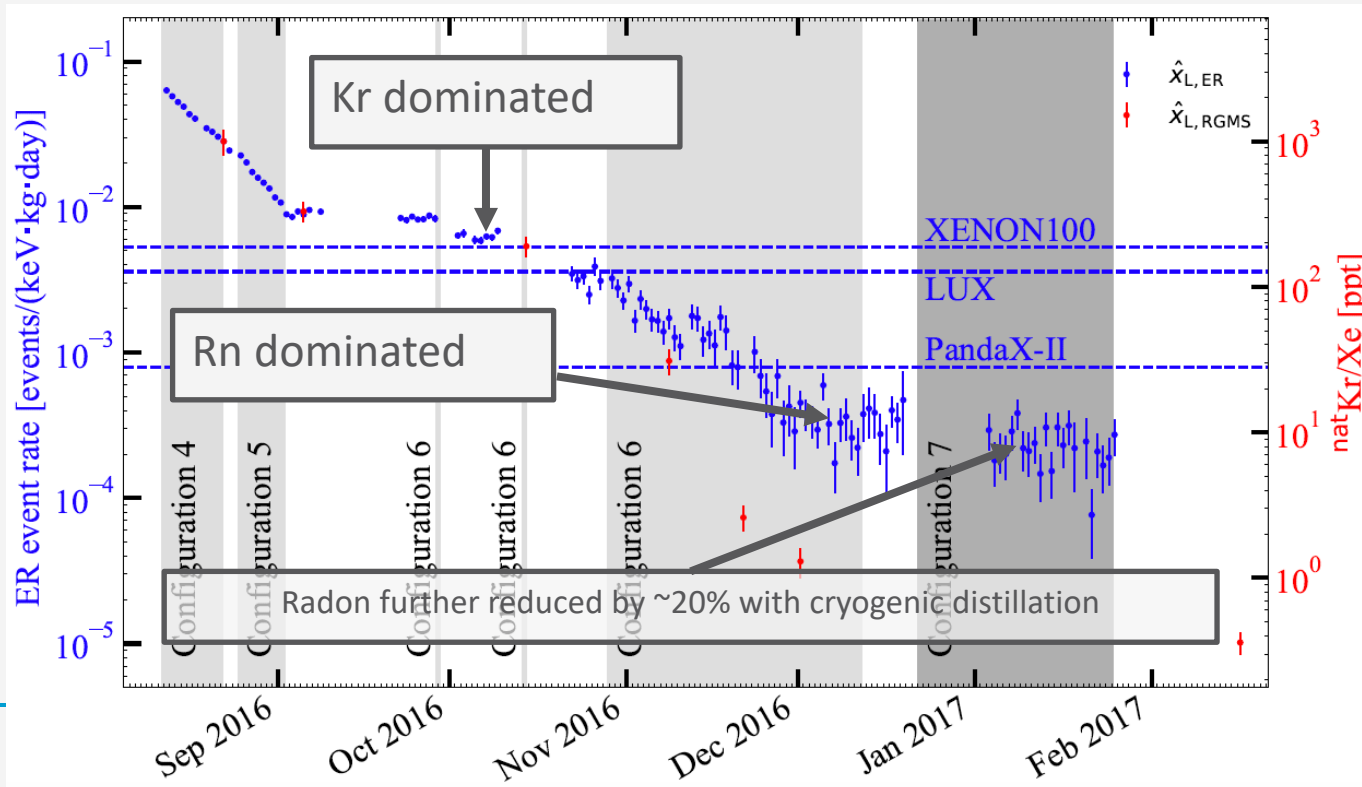
Processing flow rate: up to 6.5 kg/h (18 SLPM)

Separation factor: $6.4^{+1.9}_{-1.3} \cdot 10^5$



Kr removal (XENONnT): $\frac{\text{natKr}}{\text{Xe}} < 2 \cdot 10^{-13}$, achieved: $\frac{\text{natKr}}{\text{Xe}} < 4.8 \cdot 10^{-14} = 48 \text{ ppq}$

Kr removal achieved by “online distillation”: E. Aprile et al., PTEP 5, 053H01 (2022)



Eur. Phys. J. C77, 275 (2017)

Low ^{39}Ar – UAr & ARIA project

^{39}Ar : typically 1 Bq/kg

β emitter, $T_{1/2} = 269$ yr, $E_0 = 565$ keV

-> too much background rate for dark matter searches

A first approach:

Use underground Ar (UAr)

from Urania plant in Cortez/USA

with a reduction in ^{39}Ar by a factor 1400

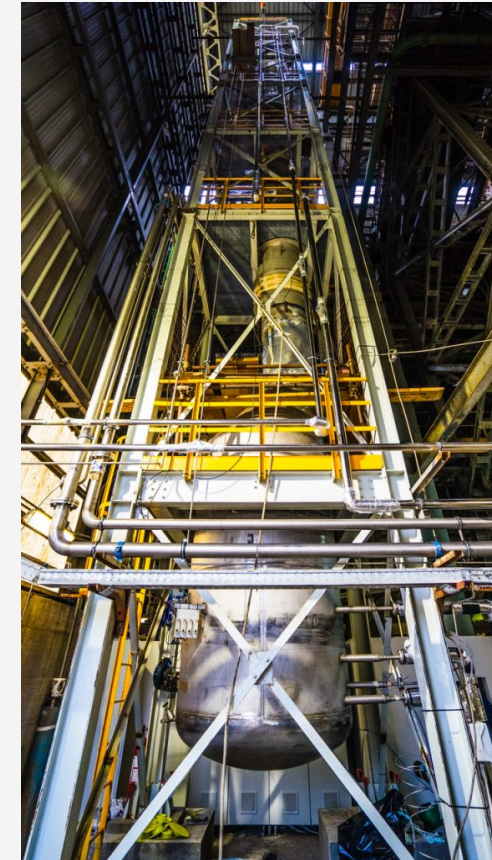
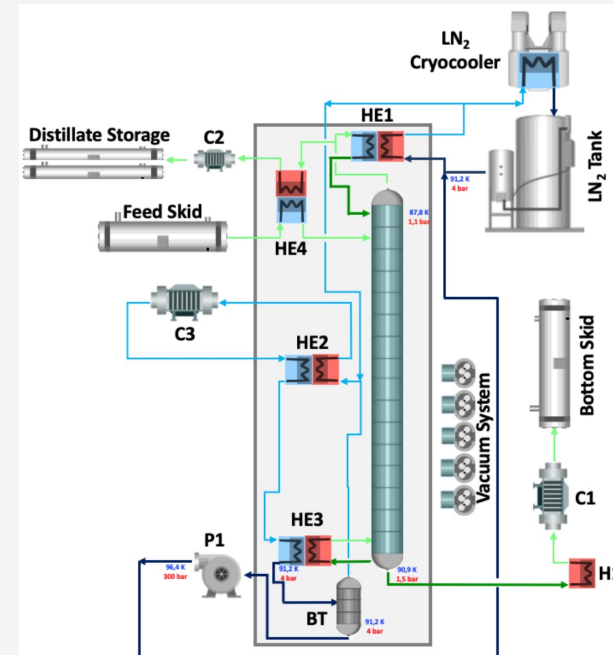
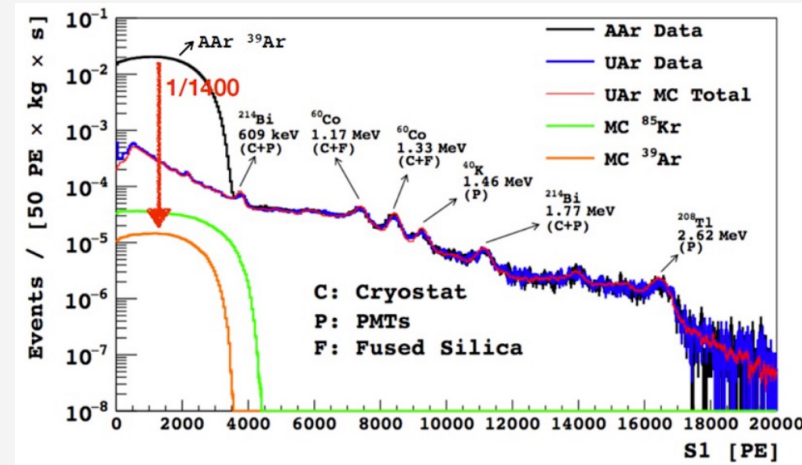
clean the Argon by using the ARIA distillation column at Sardinia

EPJ C81 (2021) 359

To go even lower in ^{39}Ar :

Cryogenic distillation for isotope separation:

ARIA-project: Factor 10 per pass, about 10 kg/d



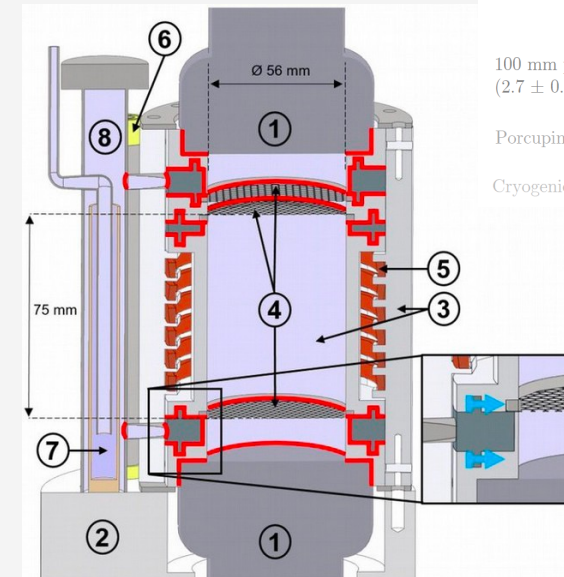
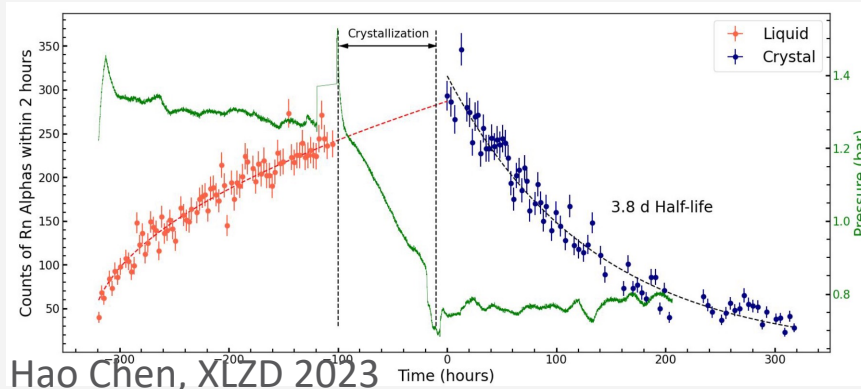
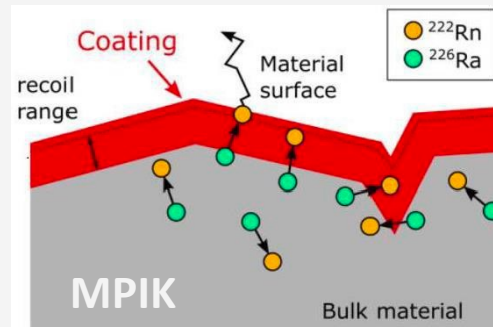
Rn mitigation

1. Screen materials for gamma emission and radon emanation and selection

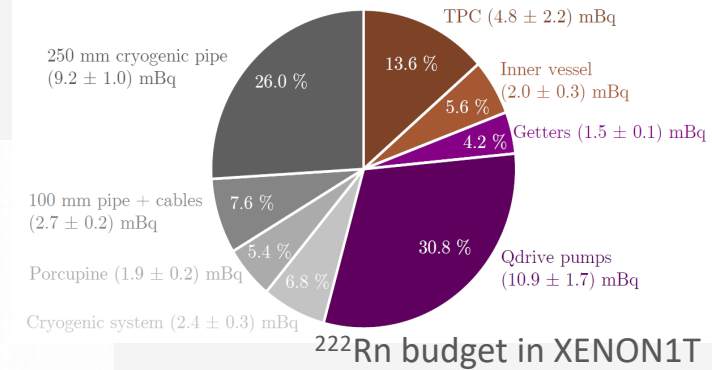


2. Avoiding Rn to move in:

- coating (MPIK)
- hermetic TPC (U Freiburg)
- xenon ice (LBNL,, U Texas)



EPJ C83 (2023) 9



3. Online removal by cryogenic distillation

also needed for $0\nu\beta\beta$ because of Bi-214 (BiPo tagging and self-shielding is not 100%)

Removal of Intrinsic radioactive contaminants: ^{222}Rn

Intrinsic noble gas contaminants ^{85}Kr and ^{222}Rn (^{214}Pb) inside the xenon

- Electronic recoil events from the low energy β -spectrum of these radioactivities contaminate ROI for dark matter WIMP search
- Searches for new physics using electronic recoil signals becomes possible when these radioactivities are drastically reduced
- ^{222}Rn emanates from detector materials and needs to be **avoided or removed continuously**
- Material screening and selection to avoid ^{222}Rn in the first place
- Main background in XENON1T, LUX and PandaX at $^{222}\text{Rn}/\text{Xe} \sim 10\mu\text{Bq}/\text{kg}$ - challenge for current experiments to reach $1\mu\text{Bq}/\text{kg}$
- Challenge met with continuous Rn removal by cryogenic distillation (XENONnT and PandaX) or by gas chromatography (LZ)

XENONnT



Cryogenic Distillation done “online” at XENONnT

- High-flow distillation column with LXe and GXe in- and outlets
- 200 slpm (1.8 t/d) LXe-phase mode and 25 slpm (0.2 t/d) GXe-phase mode
- $^{222}\text{Rn}/\text{Xe} = 1.7 \mu\text{Bq}/\text{kg}$ (GXe-only mode)
- $^{222}\text{Rn}/\text{Xe} = 0.8 \mu\text{Bq}/\text{kg}$ (GXe+LXe mode)

LZ



Gas chromatography done “online” at LZ

- 0.5 slpm from gas conduits run to 10 kg cold synthetic charcoal
- keep Rn in charcoal for >3 half-lives
- Ongoing R&D to further suppress Rn
- $^{222}\text{Rn}/\text{Xe} = 4.6 \mu\text{Bq}/\text{kg}$

J. Aalbers et al, arXiv:2211.17120, 2022

PandaX-4T



Cryogenic Distillation done “online” at PandaX-4T

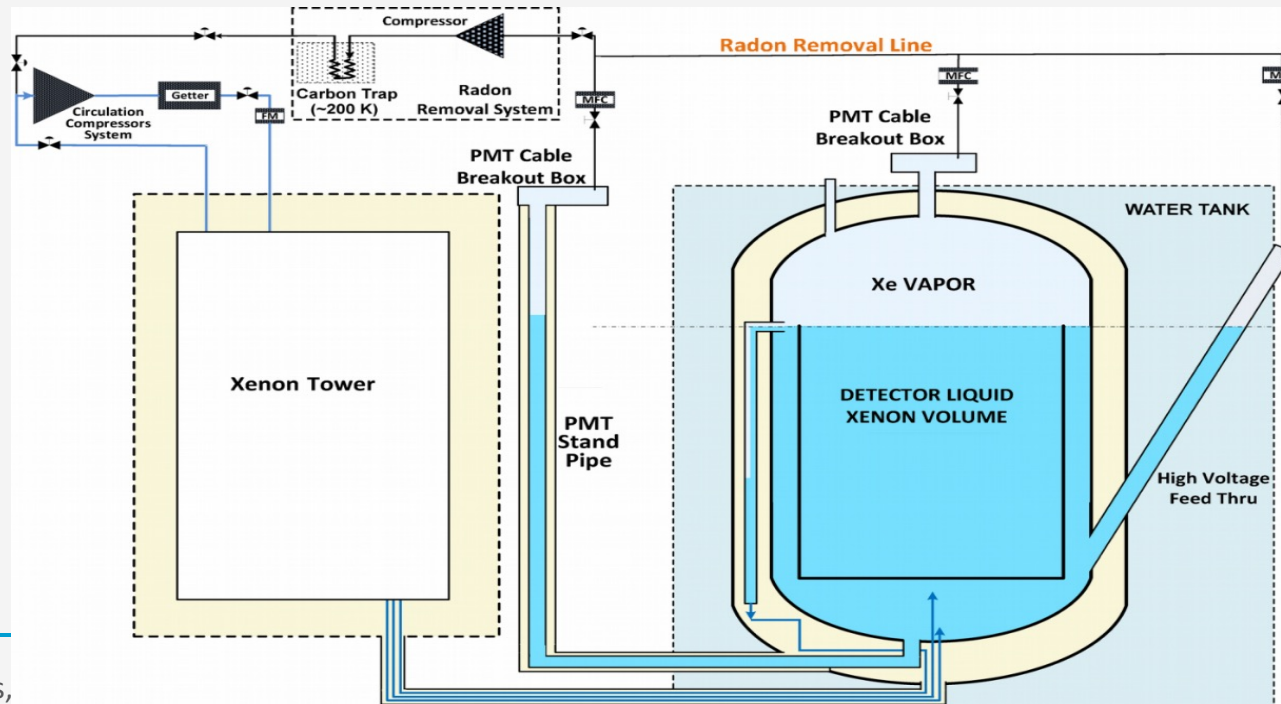
- Same as Kr removal system, but in reverse mode
- GXe in- and outlets
- 160 slpm (1.4 t/d)
- $^{222}\text{Rn}/\text{Xe} = 4.2 \mu\text{Bq}/\text{kg}$

X. Cui et al 2021 JINST 16 P07046

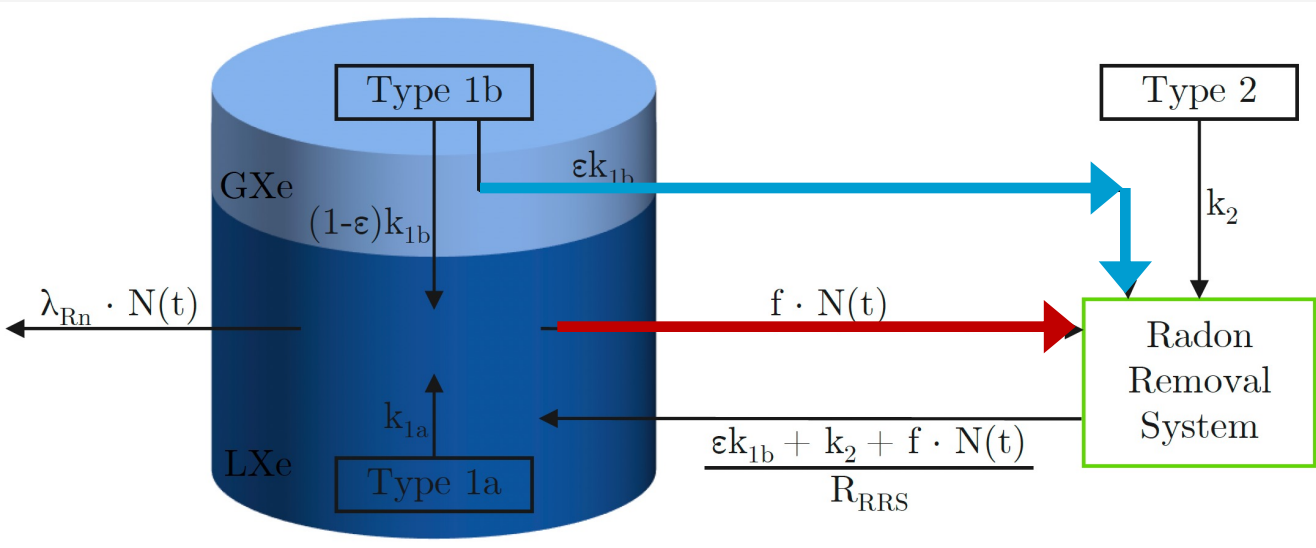
courtesy:
Elena Aprile

LZ: In-line Radon reduction system

- reduce ^{222}Rn background from in warm parts only (feedthroughs, cables, etc.)
 - tiny fraction of entire volume: 1 slpm (GXe) : 500 slpm (LXe)
 - expected to contribute $\sim 50\%$ of Rn burden in TPC
- not set up to purify all 10 t of LXe
- sequestration of atoms in activated carbon trap until most ^{222}Rn nuclei decay
 - chromatographic separation: $v(\text{Xe})/v(\text{Rn})$ (-85 C) ≈ 1000
- to obtain reduction of 90% (10x), sequestration time $\geq \ln(10) \cdot \tau_{\text{Rn}} = 12.7$ days



courtesy:
Wolfgang Lorenzon
University of Michigan



Different Rn source types:

- **Type 1b:** enters the GXe phase with a **rate** k_{1b} from cables or lines to the outside, can be extracted directly with a fraction ϵ to the RRS
- **Type 1a:** directly enters the LXe phase in the detector with a **rate** k_{1a} , can be extracted with a **relative low** f to the RRS
- **Type 2:** enters the Rn removal system with a **rate** k_2 before the LXe phase

$$r(R_{RRS} \rightarrow \infty, f, \epsilon) = \underbrace{\frac{\lambda_{Rn} + f}{\lambda_{Rn}}}_{\text{LXe extraction reduction factor}} \cdot \underbrace{\frac{k_{tot}}{k_{1a} + (1 - \epsilon)k_{1b}}}_{\text{GXe extraction reduction factor}}$$

$\approx 2 \cdot 2 = 4$ for a XENONnT at a flow of ≈ 75 kg/h

LXe extraction reduction factor with total LXe exchange time T :
 $r_{LXe} \approx 1 + \tau_{Rn}/T \approx 2$ to 4

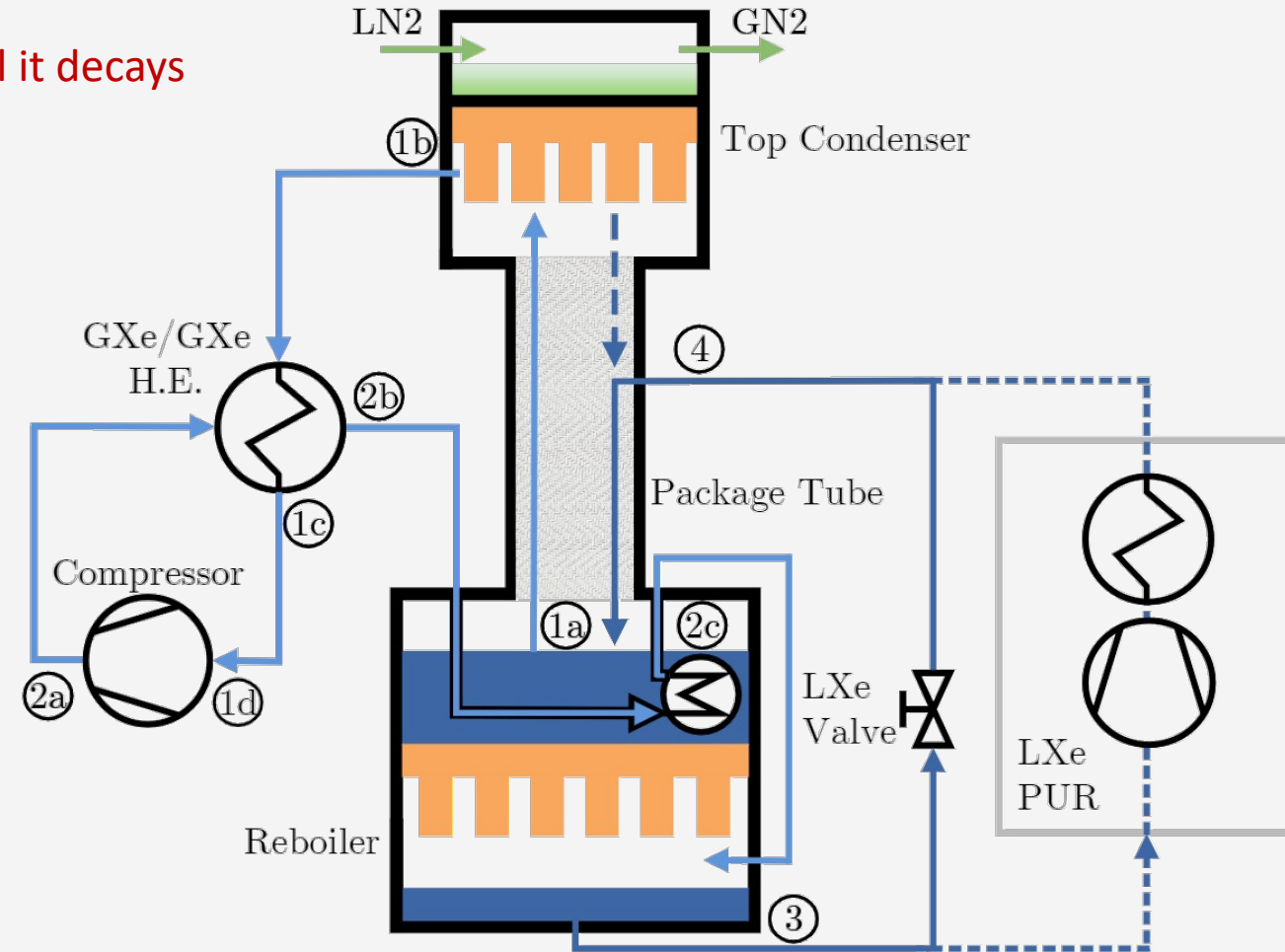
GXe extraction reduction factor, typically
 $r_{GXe} \approx 2$ (XENONnT)

Online distillation:
 E. Aprile et al. (XENON Collab.)
 Prog. Theo. Exp. Phys. 5 (2022) 053H01



High-flow radon removal distillation column

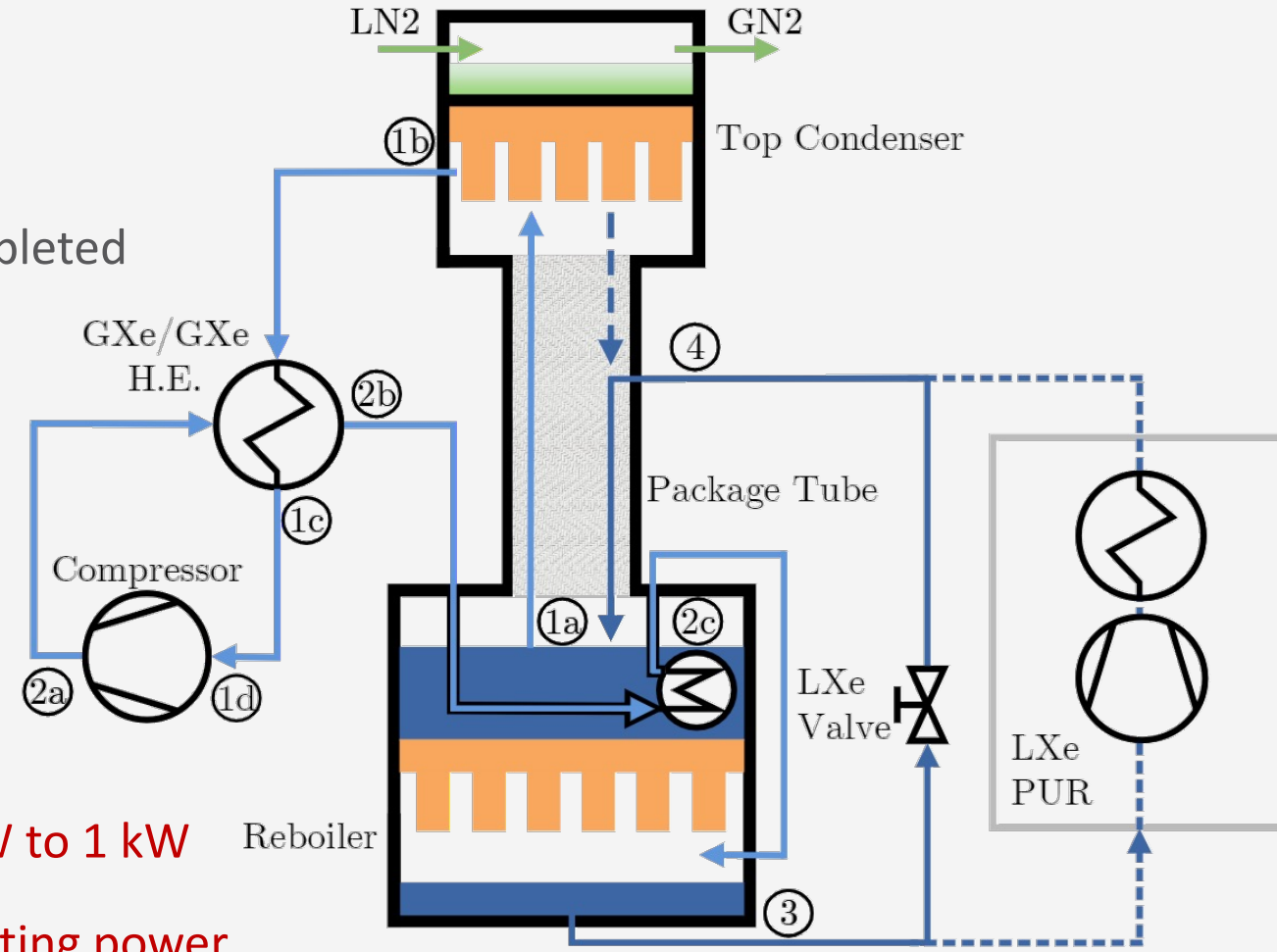
- Radon as less volatile noble gas is trapped in reboiler until it decays
- Radon-depleted GXe extracted from the top condenser
- Target flow: 72 kg/h (200 slpm) ($T < \tau_{Rn}$)
- Reduction factor: 100 between inlet and top
- Enrichment factor: 1000 between inlet and bottom
- Reflux ratio: 0.5
- 1 kW cooling power required at top
- LXe inlet and outlet
- Requires additional >2 kW cooling power for LXe outlet





Thermodynamic concept:

- Kind of heat pump: Clausius-Rankine cycle with phase changing medium xenon
- Reboiler acts as heat exchanger to liquefy radon-depleted GXe with stored radon-enriched LXe
- Compressor acts as heat-pump
- Requires two special hardware developments
Radon-free compressor
Radon-free heat exchangers
- Reduce required external cooling power from >3 kW to 1 kW
- Drastically reduce LN₂ consumption & electrical heating power



Radon removal system



XeS

Radon removal system for XENONnT



Top Condenser

Custom bath-type LN₂/GXe heat exchanger
(arXiv:2203.01026)

PackageTube

Large-surface package material

Auxiliary

Commercial GXe/GXe heat exchangers

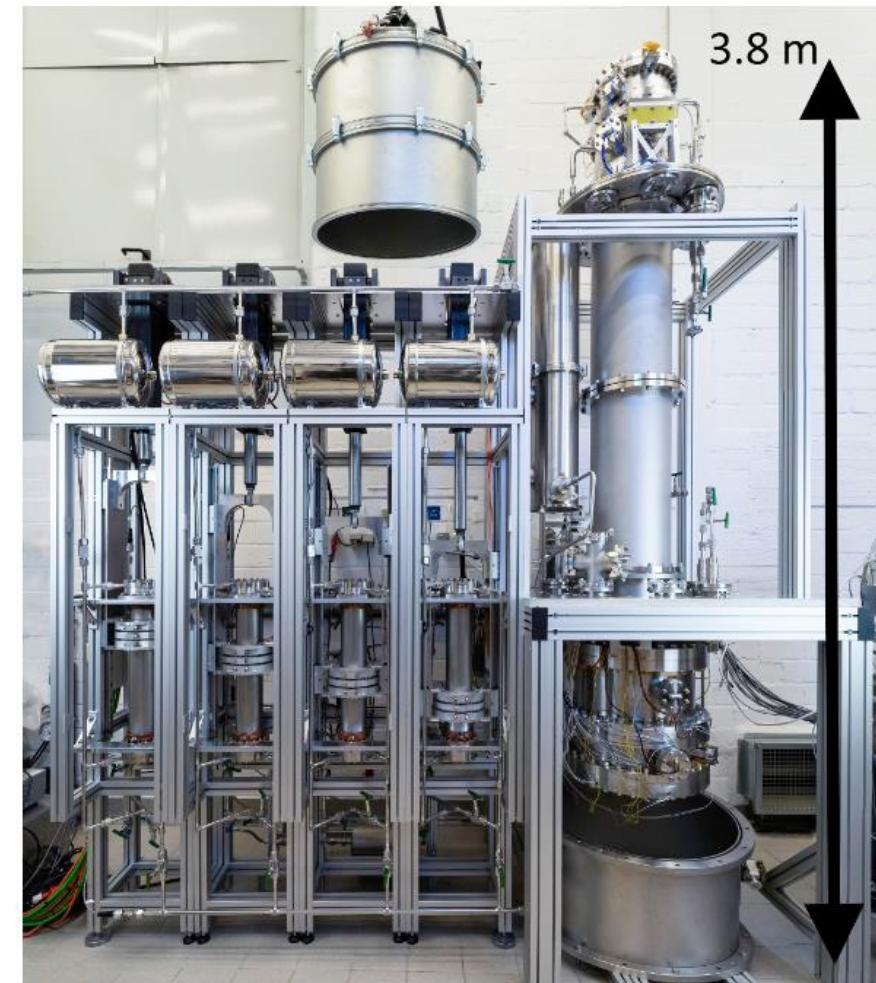
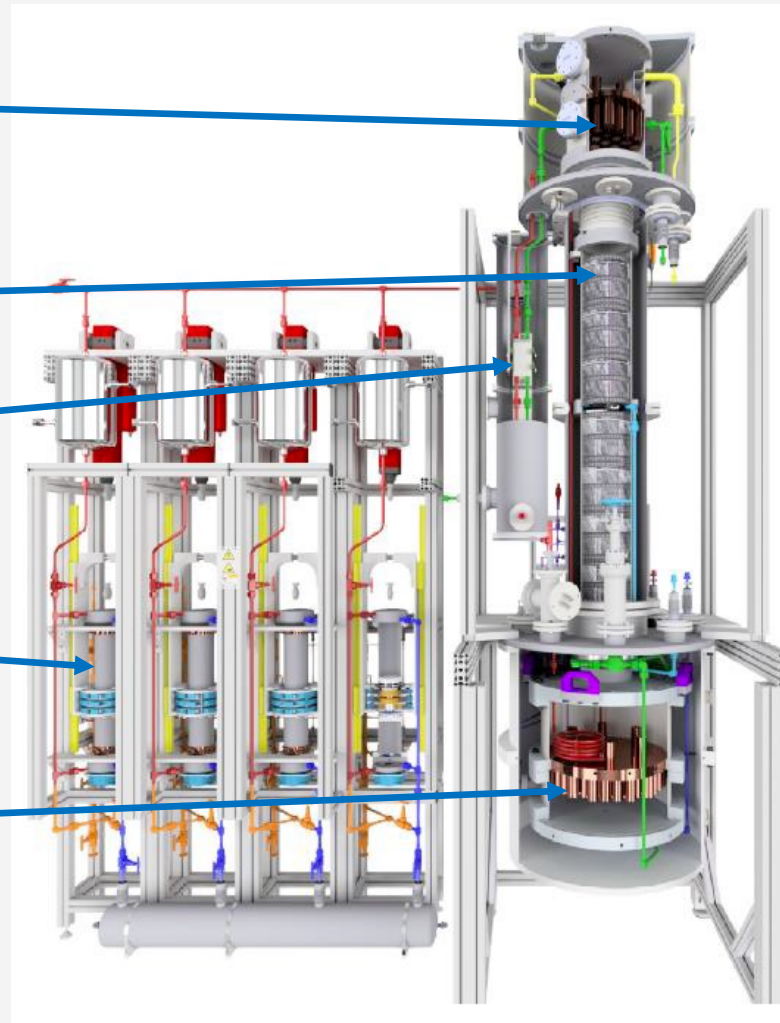
Compressor

Custom four cylinder magnetically-coupled
piston pump: JINST 16 (2021) P09011

Reboiler

Custom bath-type Xe/Xe heat exchanger
JINST 17 (2022) P05037

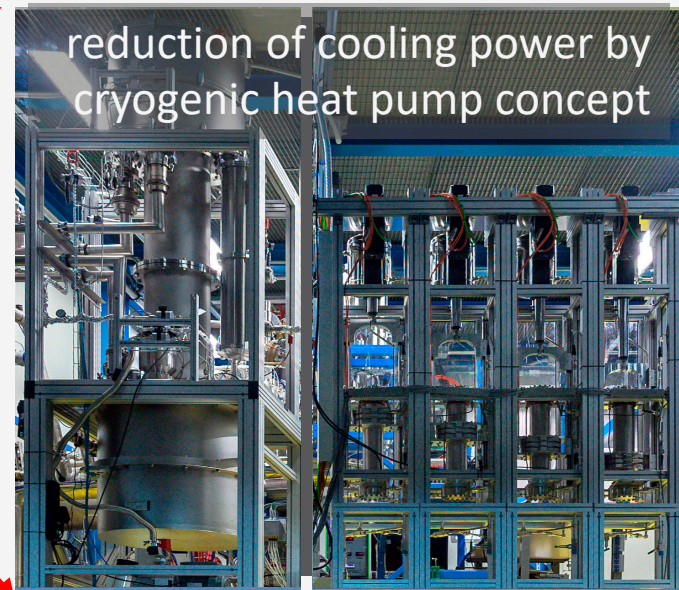
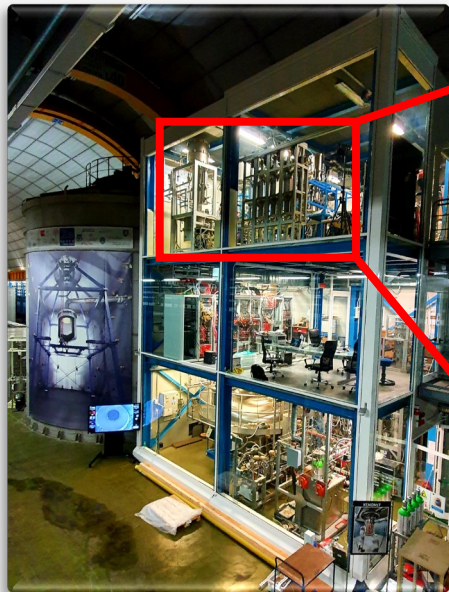
Overall system: EPJ C82 (2022) 1114



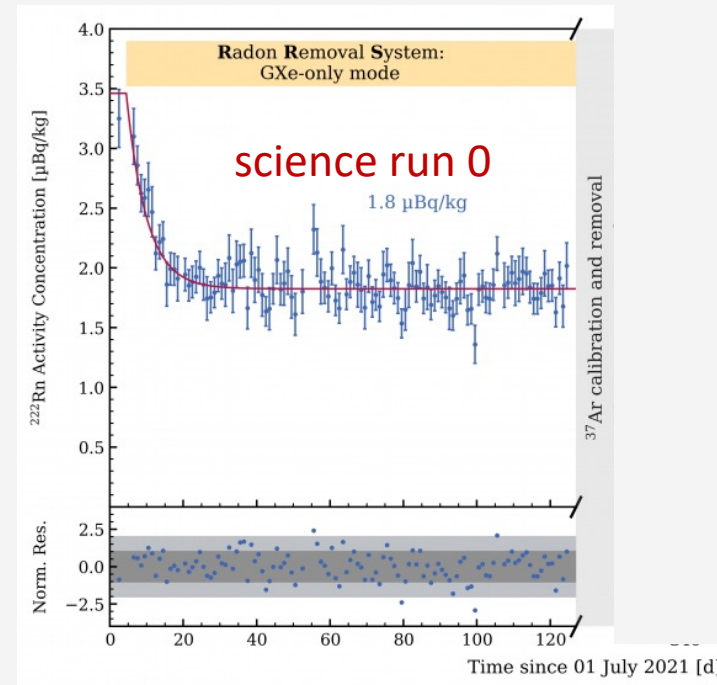


Again cryogenic distillation - key parameter:

- liquid xenon inlet and outlet
- flow of 0.4 l/min LXe = 200 SLPM \approx 70 kg/h
- **reduction by factor of 2** for sources within detector by LXe extraction
- **another reduction factor 2** for cryogenics' sources by GXe extraction



Radon concentration at XENONnT



Proof of basic removal concept:

Eur. Phys. J C77, 358 (2017)

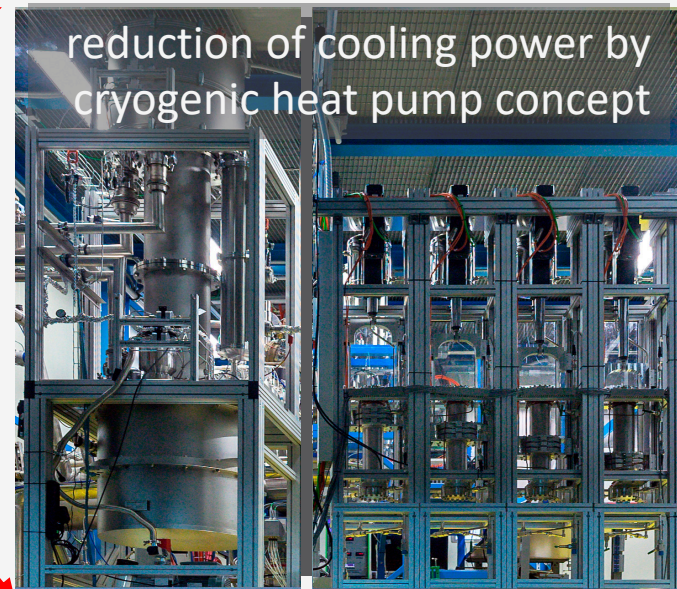
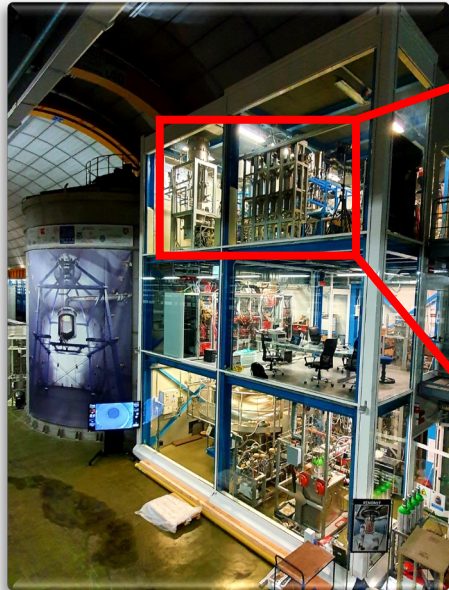
Design of XENONnT system:

Eur. Phys. J. C82, 1104 (2022)

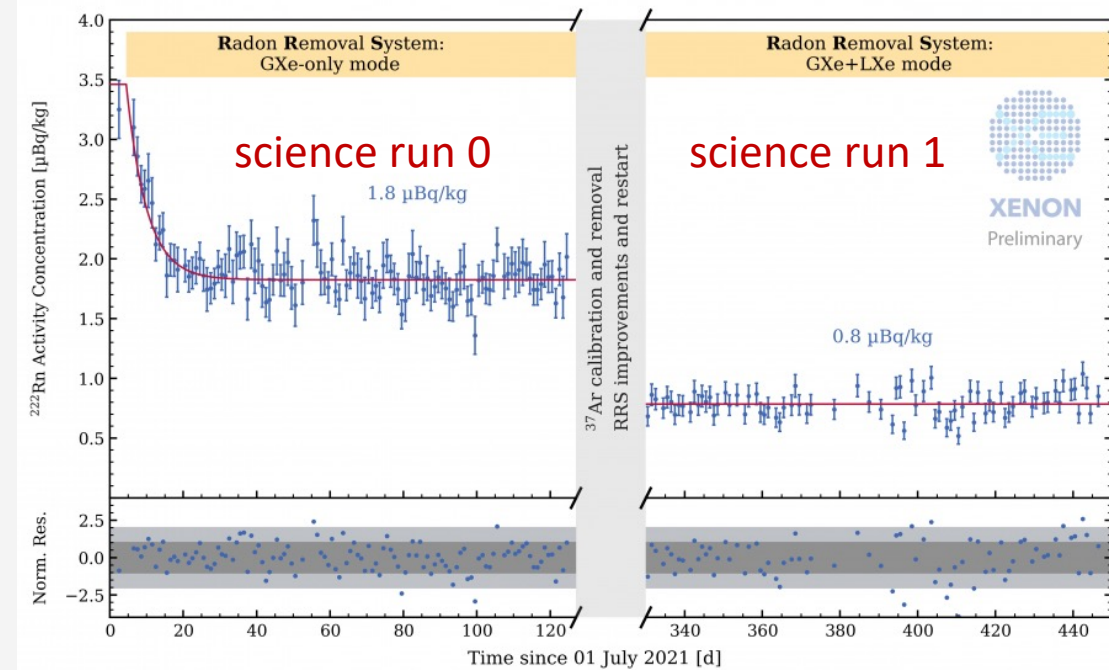


Again cryogenic distillation - key parameter:

- liquid xenon inlet and outlet
- flow of 0.4 l/min LXe = 200 SLPM \approx 70 kg/h
- **reduction by factor of 2** for sources within detector by LXe extraction
- **another reduction factor 2** for cryogenics' sources by GXe extraction



Radon concentration at XENONnT



Proof of basic removal concept:

Eur. Phys. J C77, 358 (2017)

Design of XENONnT system:

Eur. Phys. J. C82, 1104 (2022)



Again cryogenic distillation - key parameter:

- liquid x
- flow o
- **reduc**
- by LX
- **anoth**
- GXe e

Key feature 1: Ultra-high vacuum technology and ultra-pure materials

- absolute leak tightness
- very low emanation of impurities
- material control (Rn emanation measurements (MPIK), most components custom-built)

Key feature 2: Online diagnostics

- process control: level meter, flow, (differential) pressure, temperature, ...
- Rn concentration by decay measurements in XENONnT

Key feature 3: Cooling power

- heat-exchangers, heat pump concept, LN₂ evaporation

Key feature 4: Liquid xenon flow

- very high flows require LXe from the detector to the radon removal system and back



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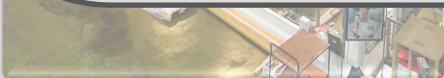
Argon cryogenic distillation, key parameters

Kryogenic distillation was brought into our field by **XMASS** for Kr removal: *Astropart. Phys.* 31, 290-296 (2009)

This technology was taken over by the Columbia University group for XENON100 and taken over for XENON1T/nT by Münster University.

Publications on cryogenic distillation related topics by Münster group/XENON:

1. M. Murra, D. Schulte, C. Huhmann, C. Weinheimer, *Design, construction and commissioning of a high-flow radon removal system for XENONnT*, *Eur. Phys. J. C* 82 (2022) 1104
2. M. Murra, D. Schulte, I. Cristescu, J.-M. Disdier, C. Huhmann, D. Tatananni, C. Weinheimer, *Cryogenic bath-type heat exchangers for ultra-pure noble gas applications*, *JINST* 17 (2022) P05037
3. E. Aprile et al. [XENON Collaboration], *Application and modeling of an online distillation method to reduce krypton and argon in XENON1T*, *Prog. Theor. Exp. Phys.* 2022 (2022) 053H01
4. D. Schulte, M. Murra, P. Schulte, C. Huhmann, C. Weinheimer, *Ultra-clean radon-free four cylinder magnetically-coupled piston pump*, *JINST* 16 (2021) P09011
5. E. Aprile et al. [XENON Collaboration], *^{222}Rn emanation measurements for the XENON1T experiment*, *EPJ C* 81 (2021) 337
6. E. Brown, A. Buss, A. Fieguth, C. Huhmann, M. Murra, H.-W. Ortjohann, S. Rosendahl, A. Schubert, D. Schulte, D. Tosi, G. Gratta, C. Weinheimer, *Magnetically-coupled piston pump for high-purity gas applications*, *Eur. Phys. J. C* 78 (2018) 604
7. E. Aprile et al. [XENON Collaboration], *Online ^{222}Rn removal by cryogenic distillation in the XENON100 experiment*, *Eur. Phys. J. C* 77 (2017) 358
8. E. Aprile et al. [XENON Collaboration], *Removing krypton from xenon by cryogenic distillation to the ppq level*, *Eur. Phys. J. C* 77 (2017) 275
9. R. Rosendahl et al., *Determination of the separation efficiencies of a single-stage cryogenic distillation setup to remove krypton out of xenon by using a $^{83\text{m}}\text{Kr}$ tracer method*, *Rev. Scient. Instr.* 86 (2015) 11, 115104
10. S. Rosendahl, K. Bokeloh, E. Brown, I. Cristescu, A. Fieguth, C. Huhmann, O. Lebeda, C. Levy, M. Murra, S. Schneider, D. Venos and C. Weinheimer, *A novel $^{83\text{m}}\text{Kr}$ tracer method for characterizing xenon gas and cryogenic distillation systems*, *JINST* 9 (2014) 10, P10010
11. E. Brown, S. Rosendahl, C. Huhmann, C. Weinheimer and H. Kettling, *In situ measurements of Krypton in Xenon gas with a quadrupole mass spectrometer following a cold-trap at a temporarily reduced pumping speed*, *JINST* 8 (2013) P02011



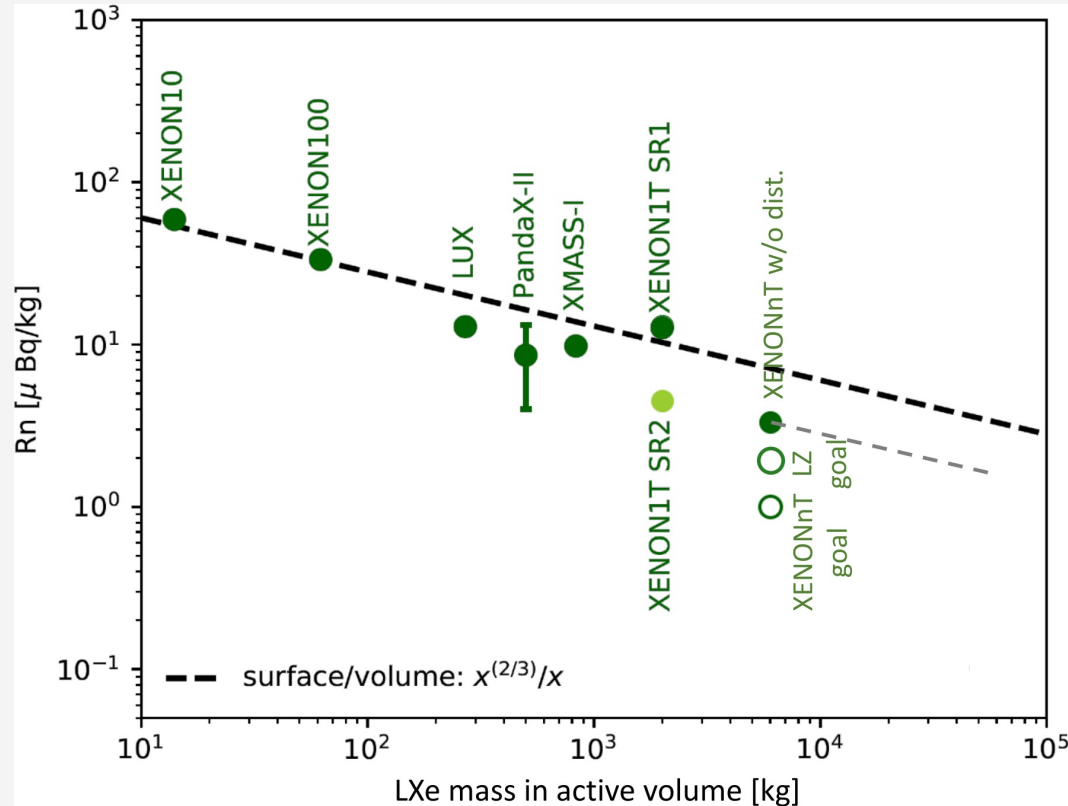
Design of XENONnT system:
Eur. Phys. J. C 82, 1104 (2022)

Rn requirements for xenon-based dark matter exp.

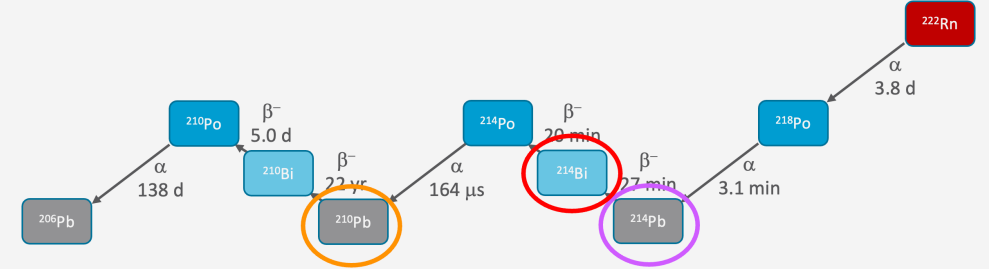
^{222}Rn : $t_{1/2} = 3.8 \text{ d}$,

continuously emanating from detector materials

^{222}Rn concentrations achieved so far



Yamashita-san plot



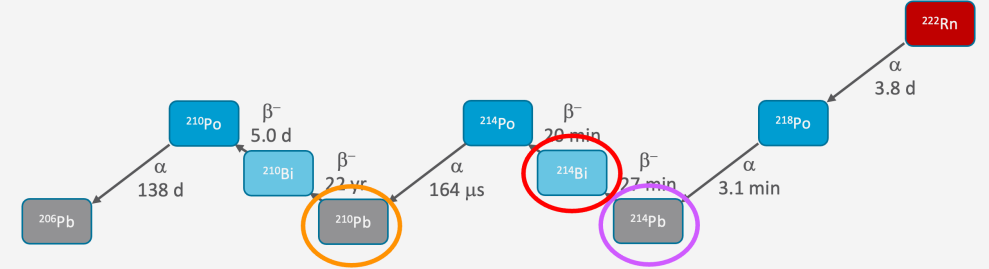
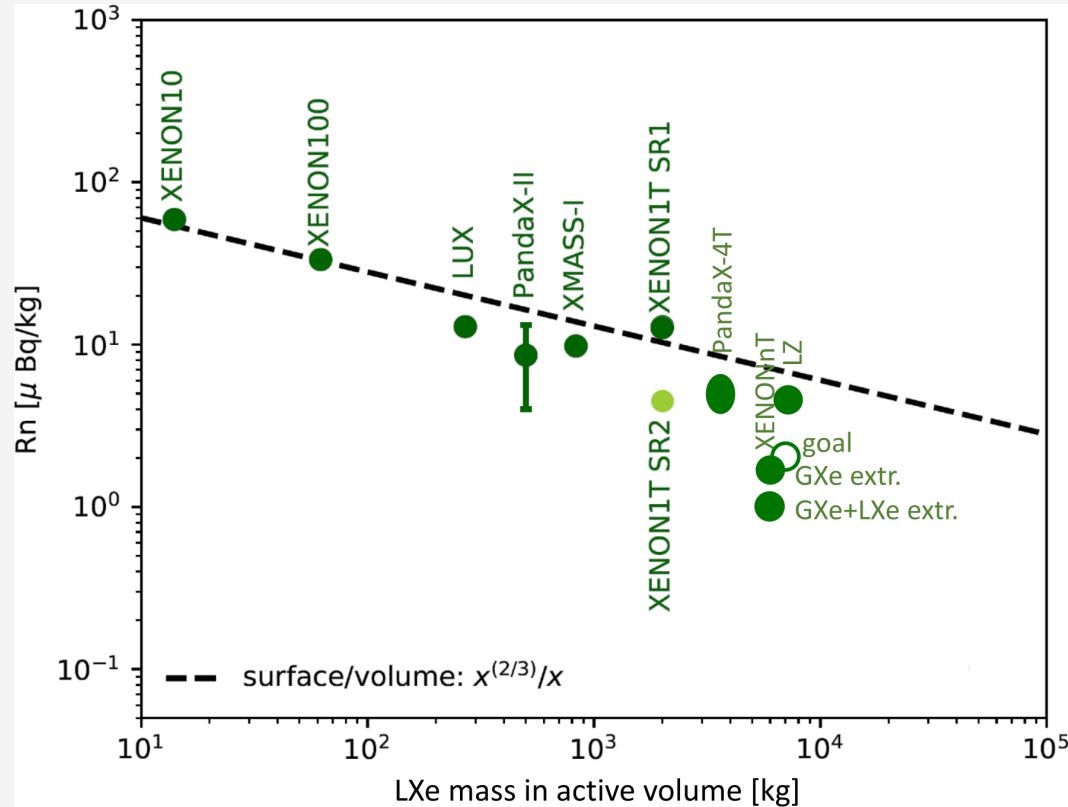
- Background from β -decay of ^{214}Pb which cannot be identified by accompanying α -decay
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- Additional backgrounds from plate-out of ^{210}Pb

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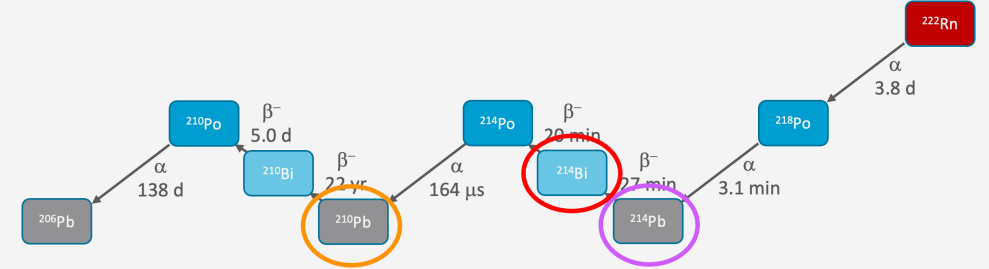
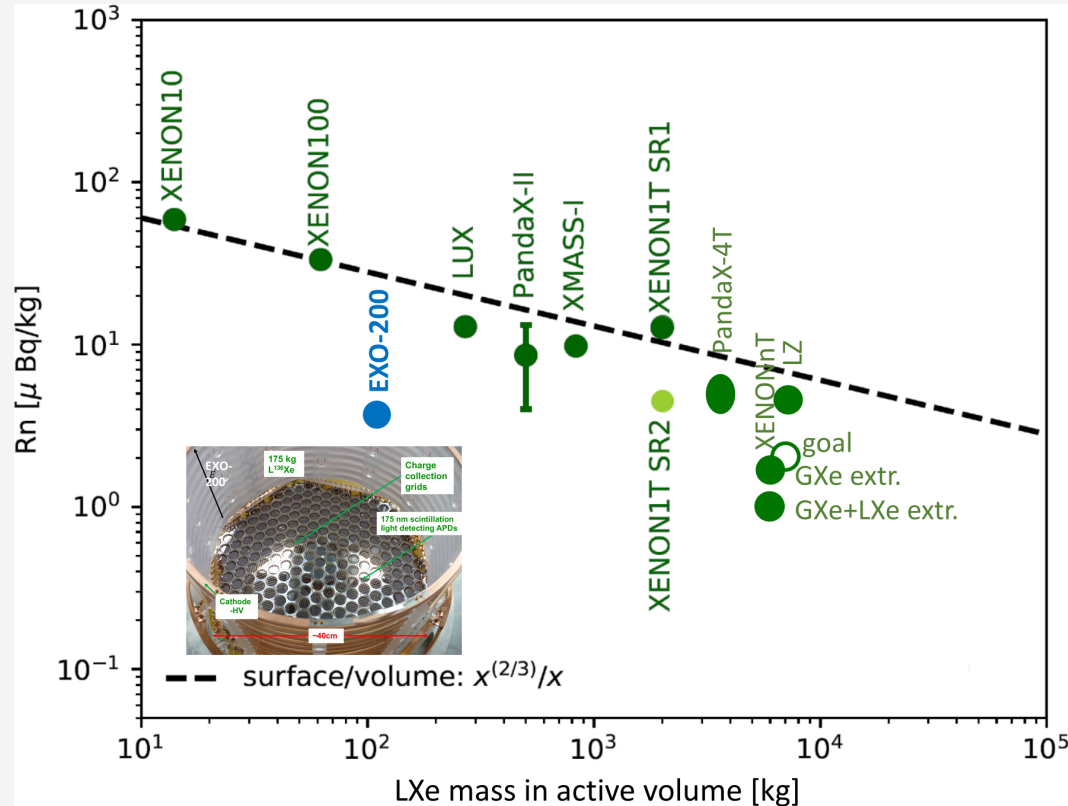
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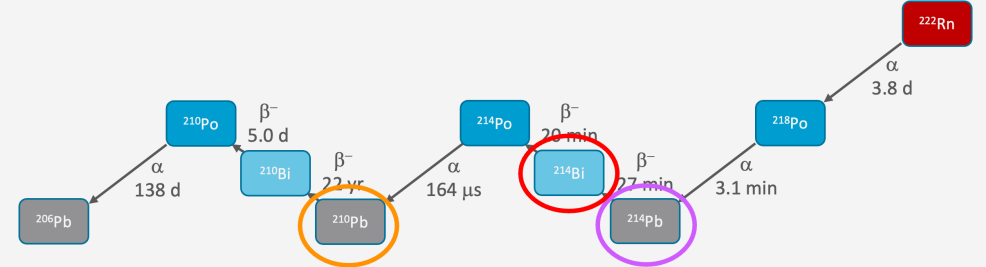
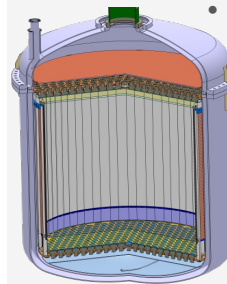
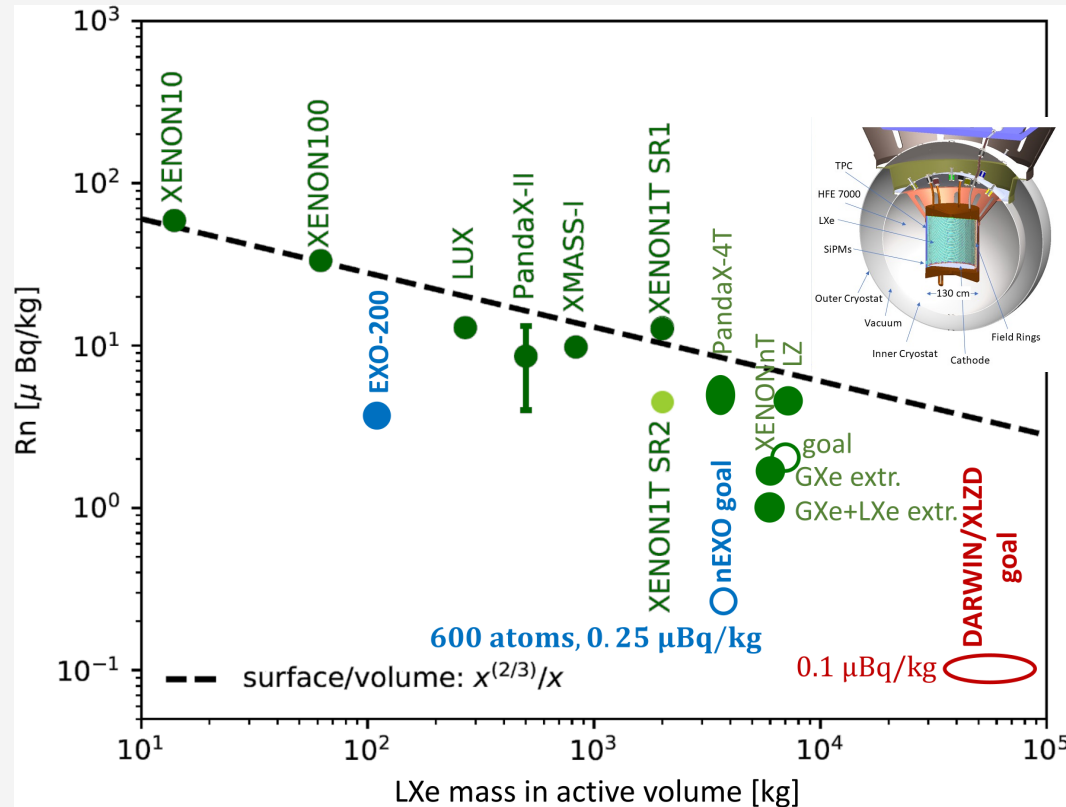
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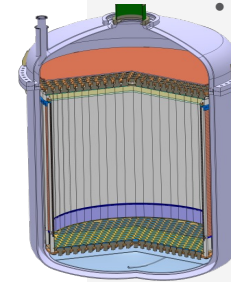
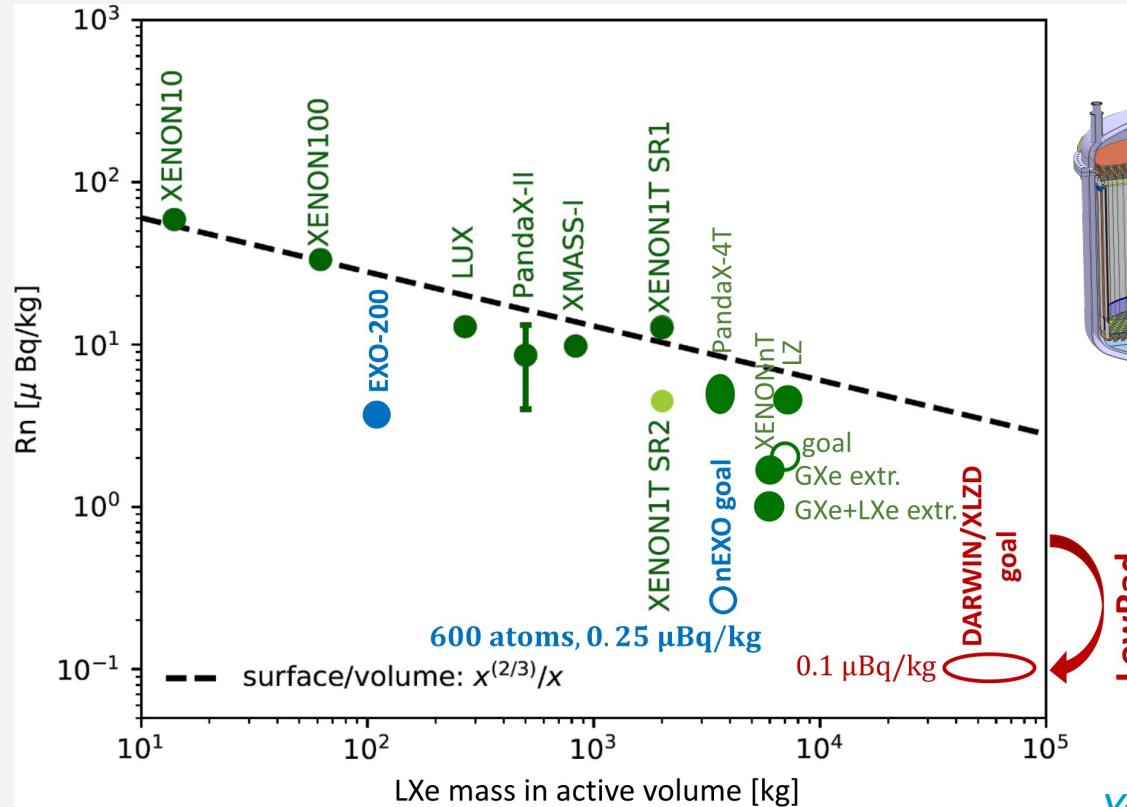
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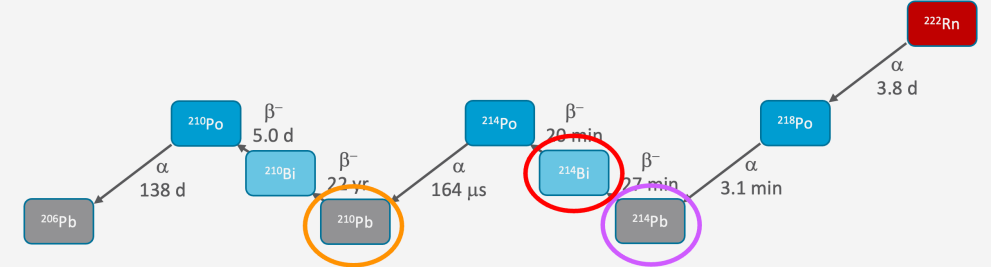
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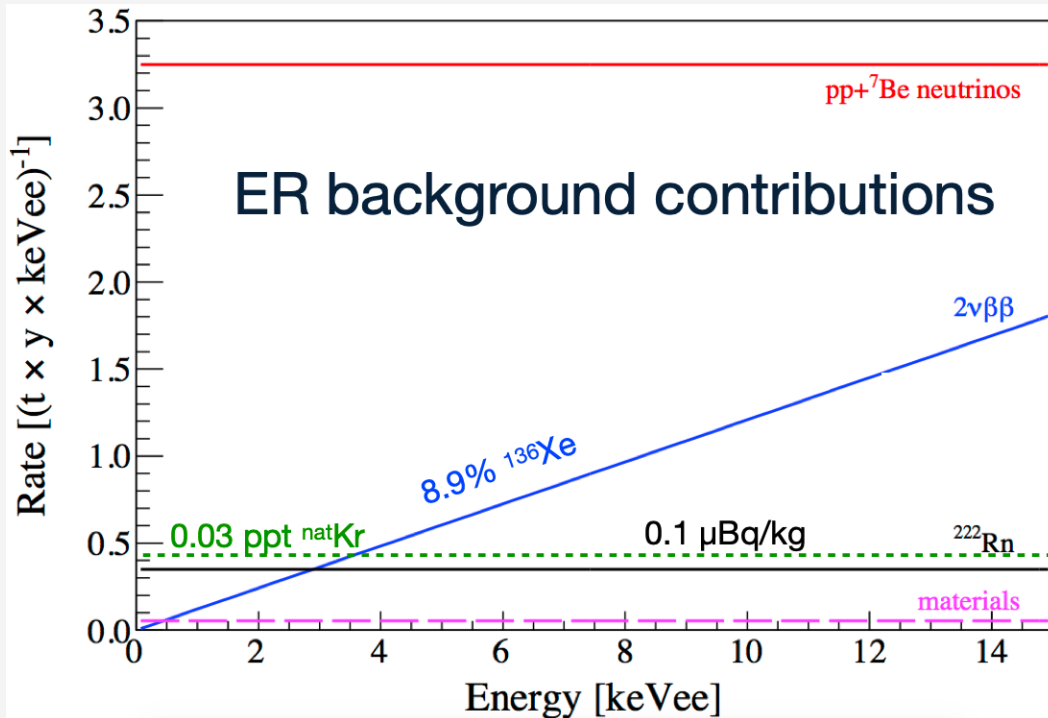


LowRad erc Yamashita-san plot



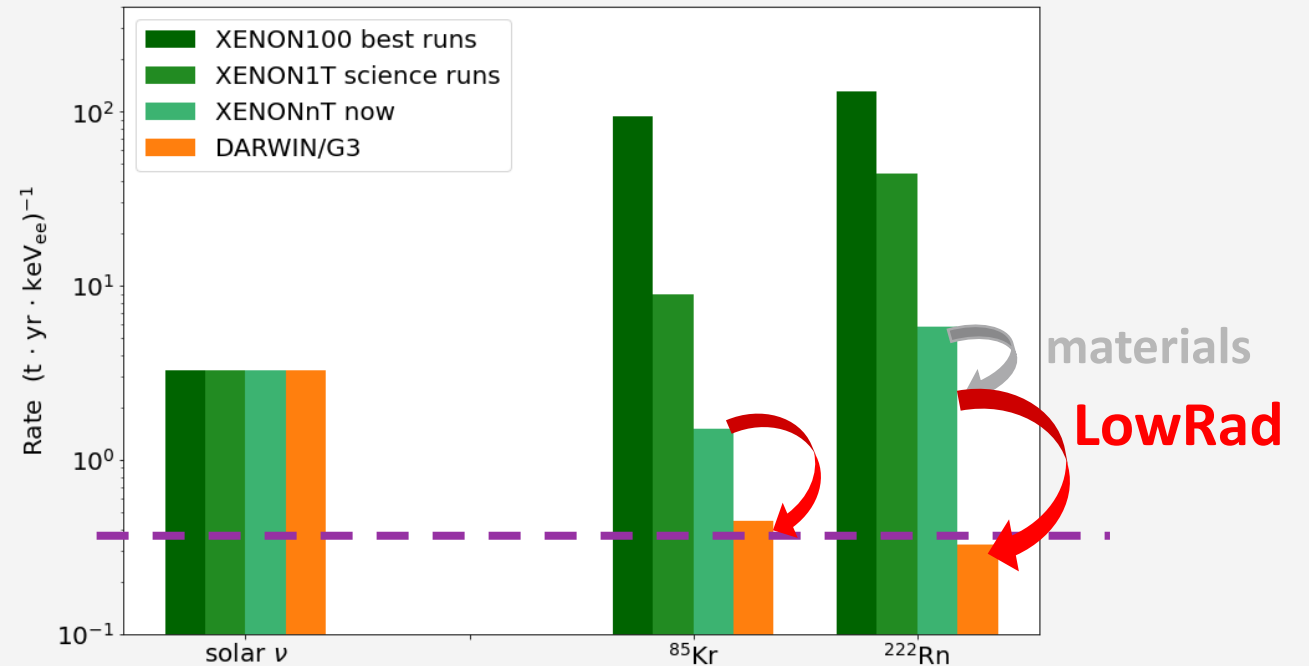
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LowRad: Low radon and low internal radioactivity for dark matter and rare event xenon detectors



Goals:

- Develop technologies to reach another factor 10 lower in ⁸⁵Kr (30 ppq ^{nat}Kr) and ²²²Rn (0.1 μBq/kg) by online removal: “less than 1 Rn atom in 100 mol of xenon”



LowRad: Low radon and low internal radioactivity for dark matter and rare event xenon detectors

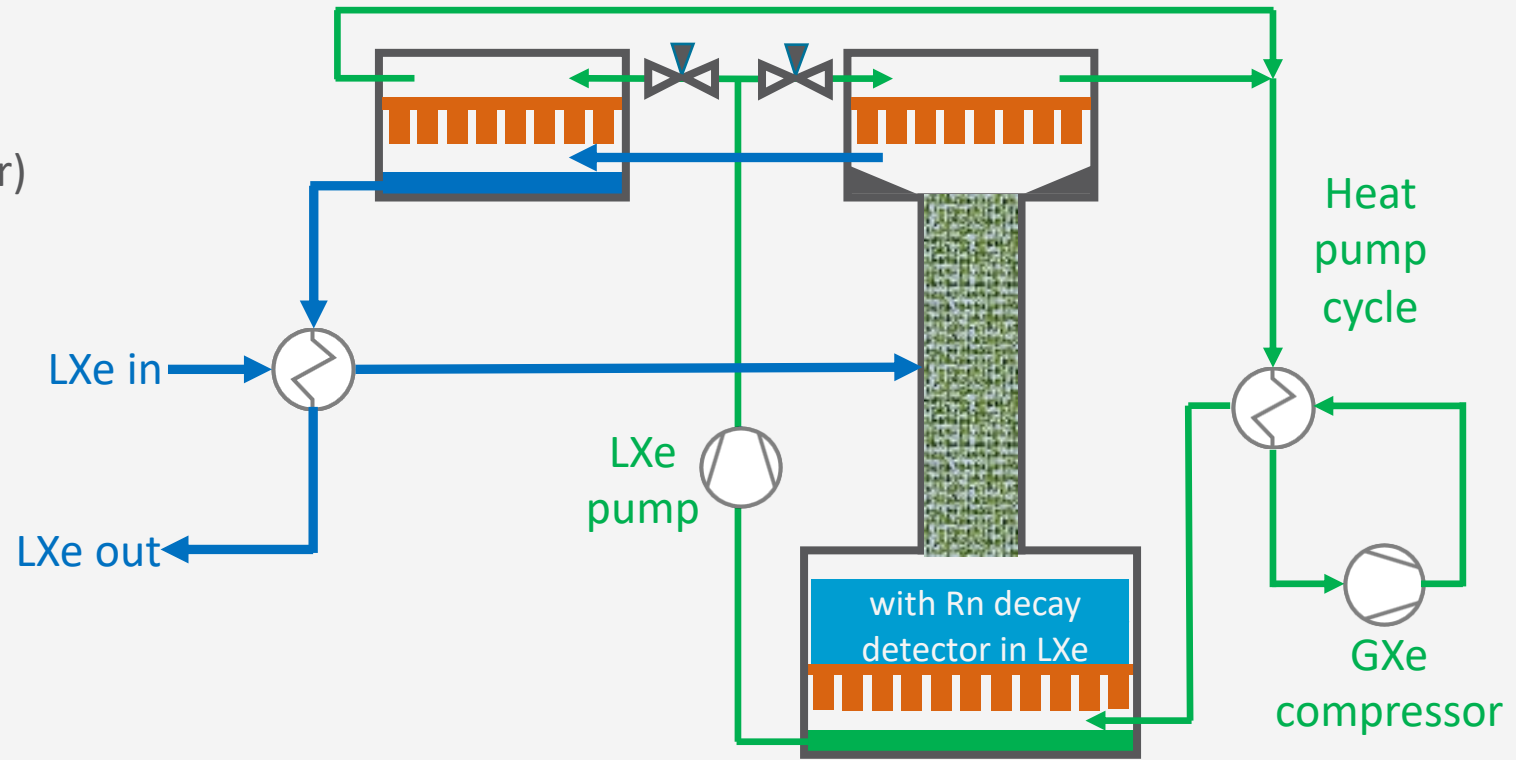
Challenges/tasks:

- Prepare for continuous/often online Kr removal:
→ develop quasi lossless method (much less off gas)
- Develop Rn removal technology with very high throughput $O(1 \text{ t/h})$
→ need efficient use of cooling power: cryogenic heat pump
- Make use of the fact, that cryogenic distillation depletes on one output,
but concentrates on the other output
→ integrate very sensitive diagnostics
- High flow cryogenic distillation and purification from electronegative impurities
→ integrate system in one together with radioactive noble gas calibration and diagnostics

Cryogenic distillation system for DARWIN/XLZD

How to purify 50 t of Xe from Rn in $\leq 2d$?

- Full heat pump to achieve enormous cooling
throughput: 75 kg/h (LowRad demonstrator)
750 kg/h (final system)
- Demonstrator
radon-free heat exchangers
2nd Xe heat pump cycle
should include online Rn decay monitor
- Final system:
should be integrated with purification system
from electronegative impurities
and with online Kr removal system
should be installed in a water shield to avoid Xe activation
- R&D and demonstrator within ERC AdG LowRad



- Clear need for removal of radioactive noble gases from xenon for search for dark matter and $0\nu\beta\beta$:
 ^{85}Kr , ^{222}Rn and progenies, (^{37}Ar , ^{39}Ar , ^{136}Xe)
(some overlap with LAr dark matter experiments)
- Charcoal chromatography can reach the low concentrations but quite some effort
→ will stay very important for diagnostics and very dirty samples,
but maybe even more by LXe Rn removal by a “swing” system
- Cryogenic distillation is a robust and efficient method yielding ultralow concentrations at XENONnT:
 ^{85}Kr (≈ 100 ppq $^{\text{nat}}\text{Kr}$) and ^{222}Rn (≈ 1 $\mu\text{Bq/kg}$)
Should be the default method for DARWIN/XLZD (and nEXO)
after all primary mitigation strategies have been explored:
 - ^{85}Kr : removal at beginning, absolute leak tightness
 - ^{222}Rn : material screening and emanation tests as well as selection, coating, apparatus design
- LowRad kickoff meeting at Münster “Low radon in dark matter and neutrino experiments”, June 28-30, 2023

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DFG

