Universität Münster

Radon distillation for future LXe experiments

low radon and low internal radioactivity LowRad

Technology and Instrumentation in Future Liquid Noble Gas Detectors

Nagoya – **Lutz Althüser** – 16.02.2024

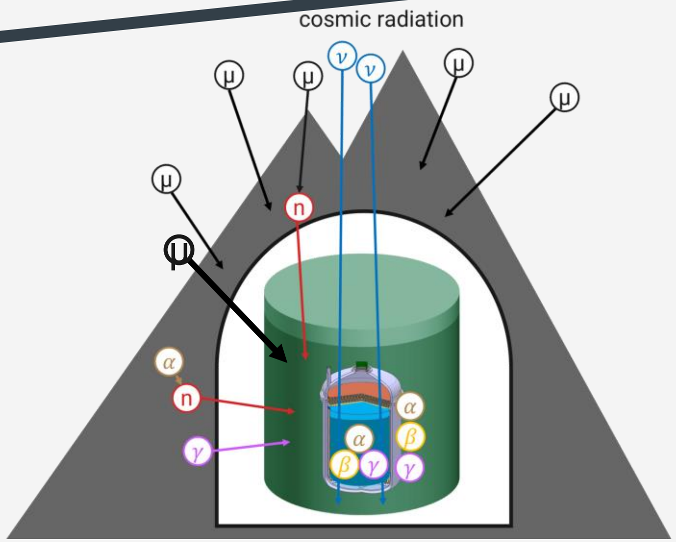
On behalf of the ERC AdG LowRad at University of Münster
With information from the XENON collaboration

- Intrinsic radioactive backgrounds
- Cryogenic distillation
- XENONnT Kr/Rn removal systems
- LowRad project

XENON

erc LowRad

living.knowledge



Intrinsic radioactive backgrounds

Intrinsic noble gas contaminants ^{85}Kr, ^{37}Ar and ^{222}Rn

- Leakage events from the low energy β-spectrum (ER) contaminate the ROI for NR DM searches
- Physics searches in the ER band only sensitive with low background levels

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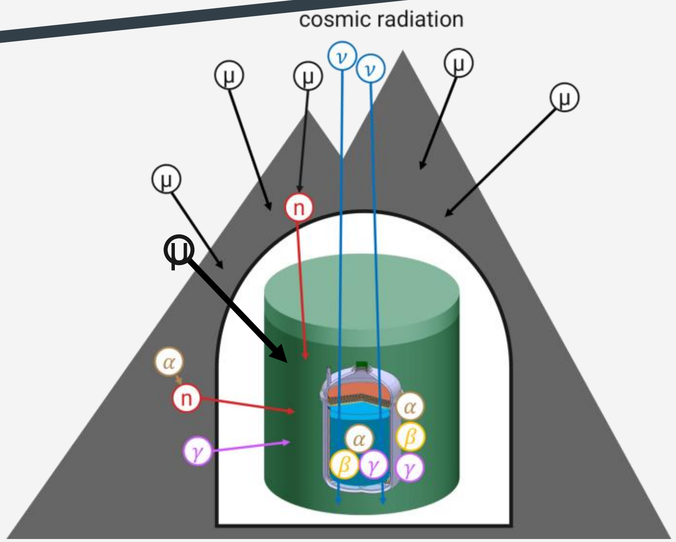

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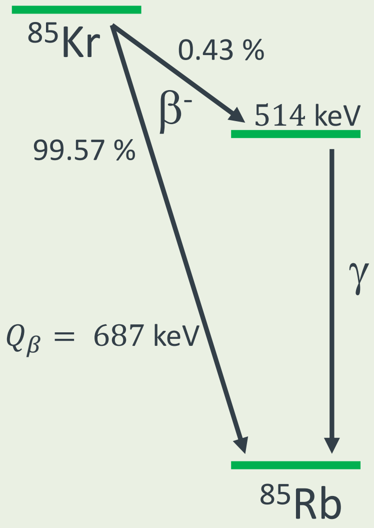


^{85}Kr

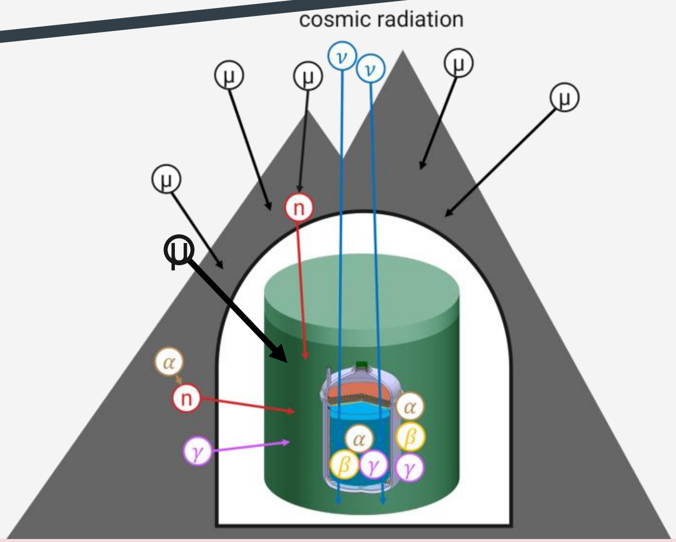
- Anthropogenic production (nuclear fission)
- Originates from Xe extraction from air
  2×10^{-11} ^{85}Kr in ^{nat}Kr
- Commercial Xe: ^{nat}Kr/Xe > 10^{-9} (ppb)

- Needs to be **removed once**
- Kr can re-enter via (tiny) air leaks

- Monitoring: Coincidence analysis, RGMS, …

Requirements for ^{nat}Kr/Xe

XENON1T: $< 0.5 \times 10^{-12}$ (0.5 ppt)
XENONnT: $< 0.2 \times 10^{-12}$ (0.2 ppt)
DARWIN: $< 0.05 \times 10^{-12}$ (0.05 ppt)



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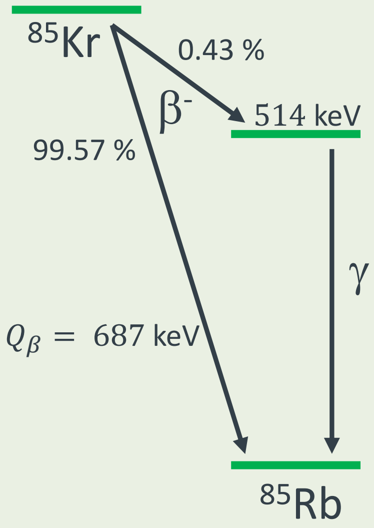

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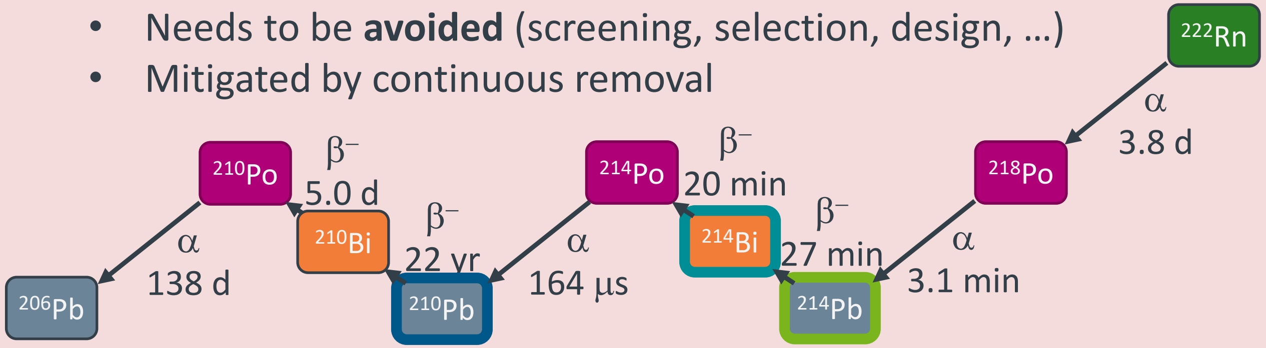
^{222}Rn

- Continuous emanation from detector materials
- Background from **β-decay**, **γ-decay (0νββ)**, **plate-out**
- Main background in current and next-gen Xe experiments

- Needs to be **avoided** (screening, selection, design, …)
- Mitigated by continuous removal

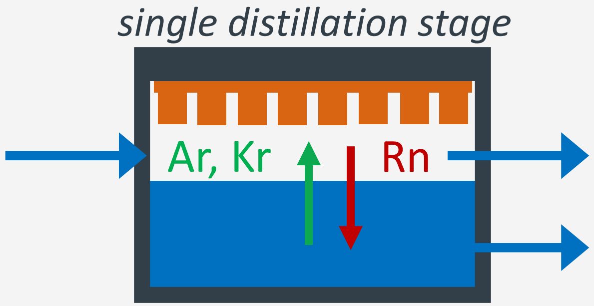


Monitoring: α-analysis, Bi-Po, …

Requirements for ^{222}Rn/Xe

XENON1T: ~10 µBq/kg (10^{-24})
XENONnT: ~1 µBq/kg (10^{-25})
DARWIN: ~0.1 µBq/kg (10^{-26})

Cryogenic distillation



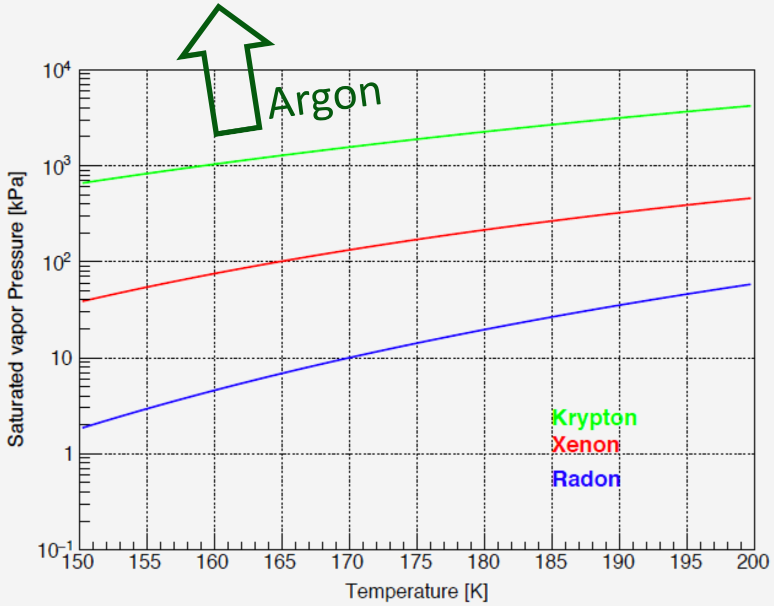
Difference in vapor pressure of the noble gas (ng) elements

- 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 053H01 (2022), EPJ C 82 1104 (2022)

Relative volatility: $\alpha = P_{\mathrm{ng}}/P_{\mathrm{Xe}}$ (@ 178 K)

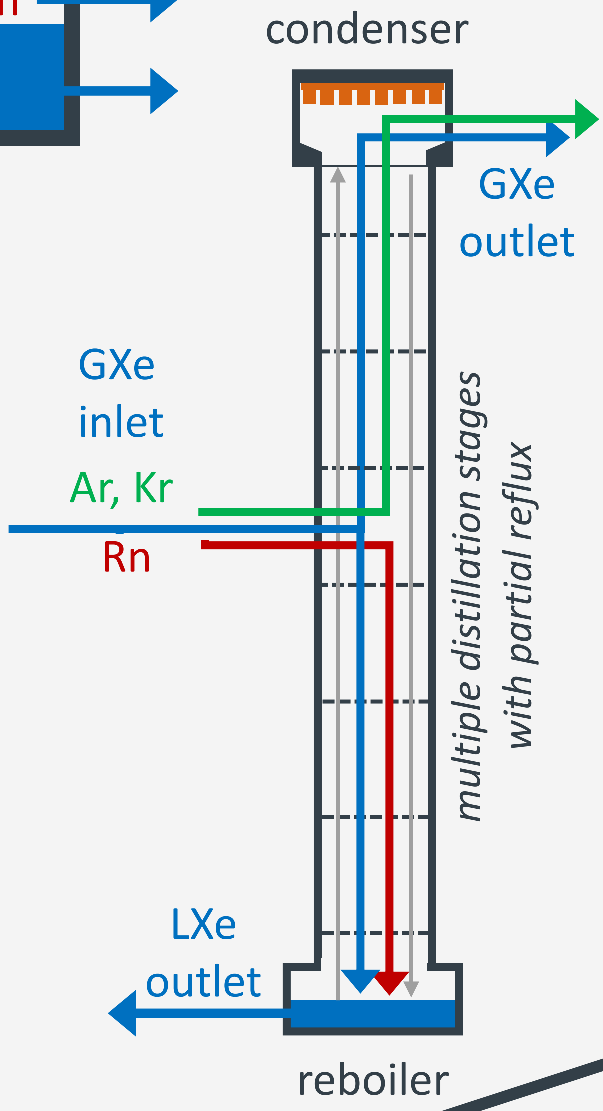
^{85}**Kr**
- $\alpha = 10.5$ (@ -100 °C)
- **More volatile** Kr collected at the **top**
- Extract Kr-enriched Xe from the top (off-gas)
- Extract Kr-depleted Xe from the bottom
- Xe off-gas considered as loss

^{222}**Rn**
- $\alpha = 0.1$ (@ -100 °C)
- **Less volatile** Rn collected at the **bottom**
- Trap Rn in the reboiler until decay ($t_{1/2} = 3.8$ d)
- Extract Rn-depleted Xe from the top



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

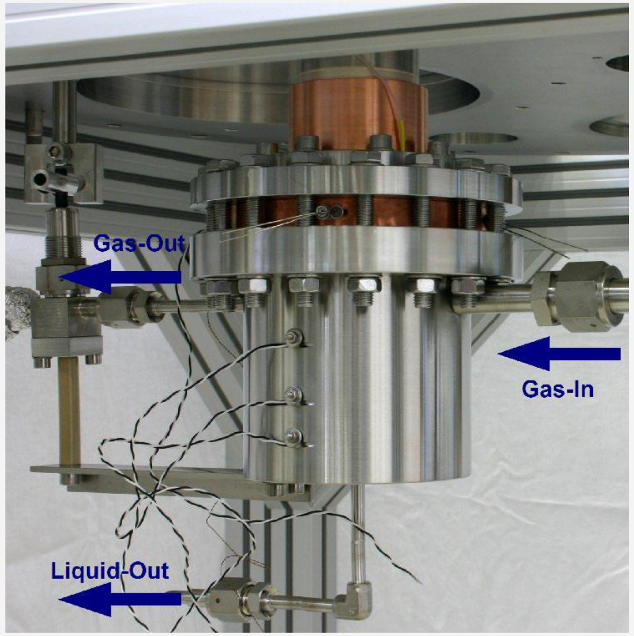
Transition probabilities of single noble gas atoms from gas to liquid and vice versa: **saturation vapor pressure**



Krypton distillation column development

Single stage DST
- Separation factor ~10
- Operation in <ppt conc.
  ^{83m}Kr tracer method

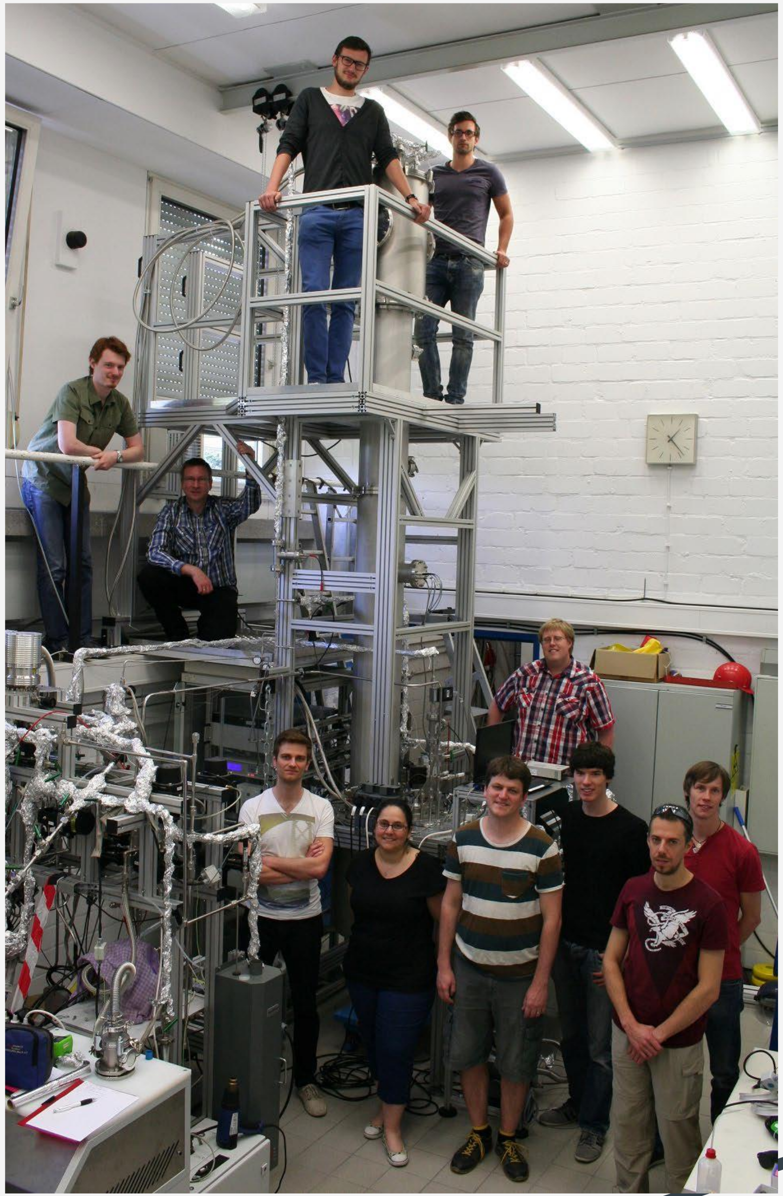
Rev Sci Instrum 86, 115104 (2015)
PhD S. Rosendahl (2015)



Phase-1 DST column
- Separation factor >5000
- ^{nat}Kr/Xe $< 0.026 \times 10^{-12}$ (26 ppq) by multiple distillation cycles

PhD S. Rosendahl (2015)

Phase-2 DST column

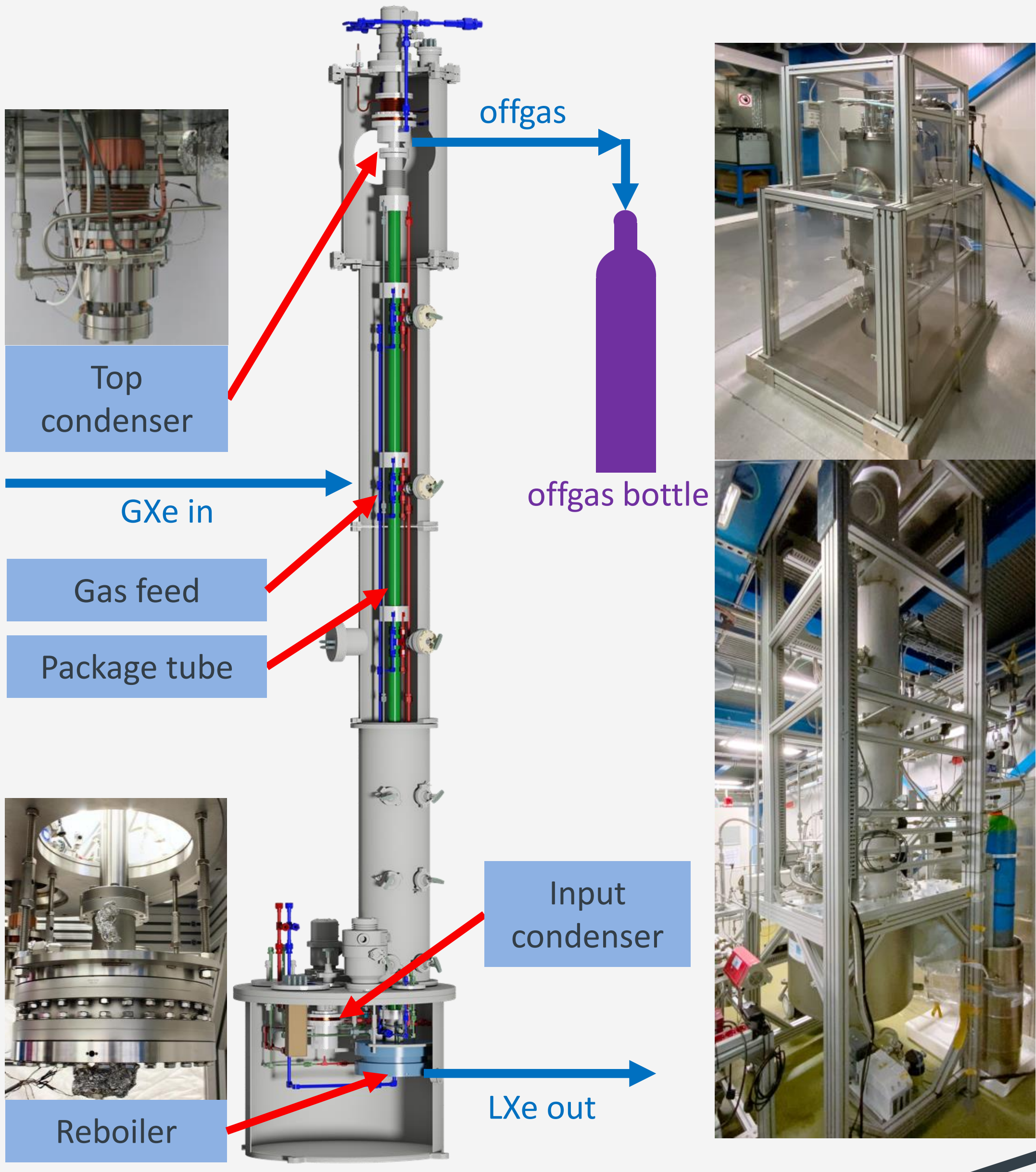


Krypton distillation column for XENON1T/nT

Phase-2 DST column (design parameter | @XENON1T)

- Feeding flow rate: 8.3 slpm (3 kg/h)
  Thermodynamically stable up to 18 slpm (6.5 kg/h)
- Separation factor: 10^4 - 10^5
  Measured separation of $6.4^{+1.9}_{-1.4} \times 10^5$ at 8.3 slpm
- Kr removal: $^{nat}Kr/Xe < 0.2 \times 10^{-12}$ (0.2 ppt)
  $^{nat}Kr/Xe < 0.026 \times 10^{-12}$ (26 ppq) (Phase-1) (multiple cycles)
  $^{nat}Kr/Xe < 0.048 \times 10^{-12}$ (48 ppq) (Phase-2) (single cycle)
- Xe recovery: 99% (1% offgas)
- Performance sufficient for XENONnT and DARWIN/XLZD

PhD S. Rosendahl (2015)
Eur. Phys. J. C 77 (2017) 275
PhD M. Murra (2019)
PTEP 5, 053H01 (2022)



Eur. Phys. J. C 77 (2017) 275

Online krypton removal for XENON1T

Requirements for ^{85}Kr removal system:

- Needs to be **removed once**
- Kr can re-enter via (tiny) air leaks

Reality check @XENON1T:

- Estimated 3-4 weeks distillation for 3.2 t Xe
- **Filled TPC without distillation** to test functionality

[a few weeks of commissioning]

- **Need** to apply **^{85}Kr removal**! But **without re-filling**!
- Development of the **online Kr removal method**

  Idea: S. Lindemann, M. Murra, G. Plante

  PTEP 5, 053H01 (2022)

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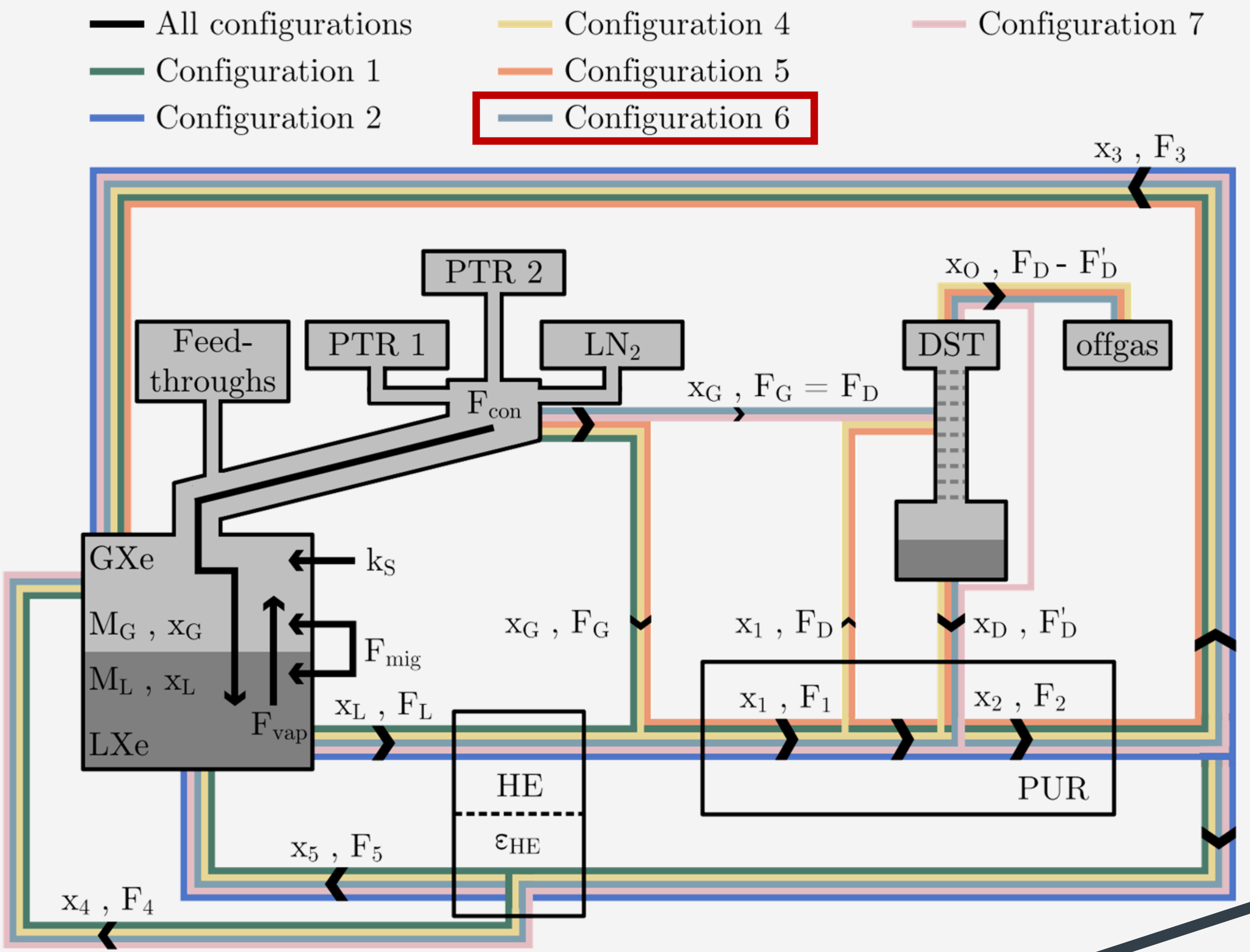
  PTEP 5, 053H01 (2022)

Online Kr removal method

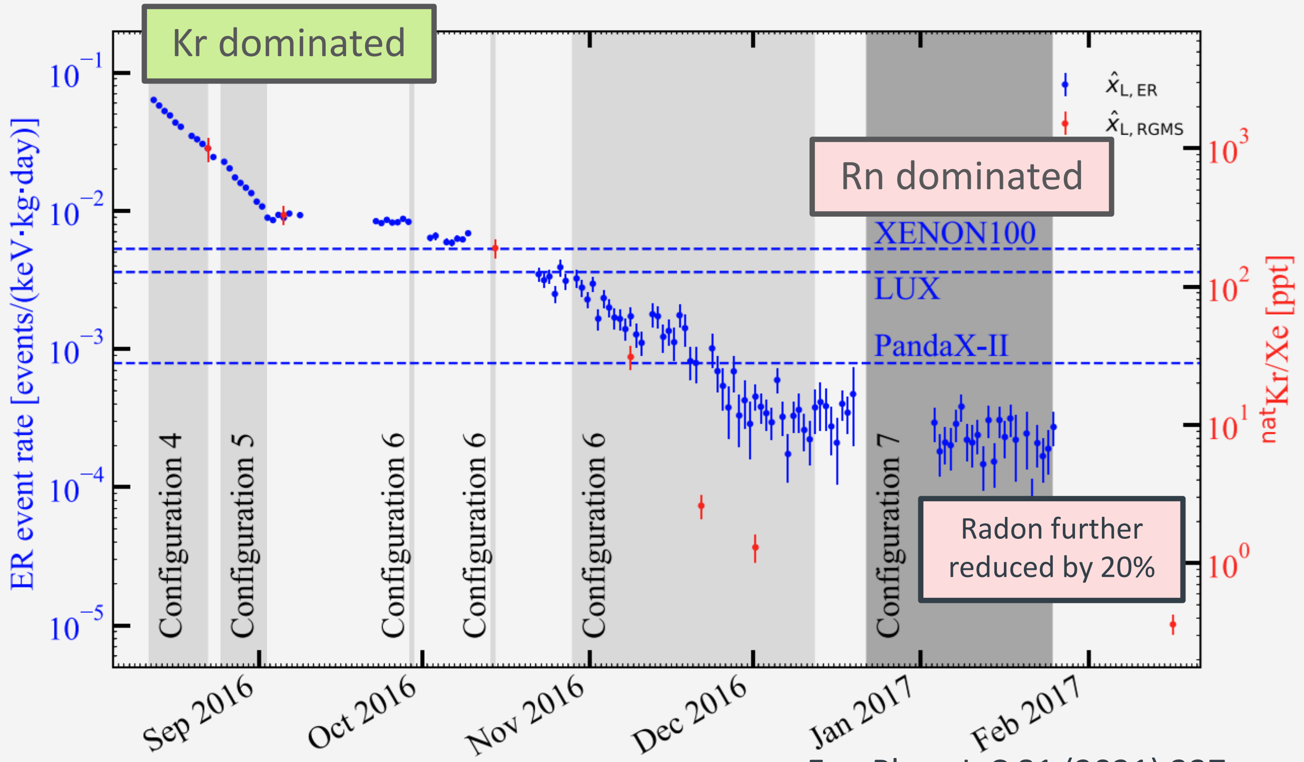
- Make use of the TPC as single stage DST
- Remove Kr from GXe, disturb equilibrium
- Kr migrates from LXe to GXe
- 1% offgas (0.85 kg/d | 6 kg/week), $\tau_{\text{eff, Kr}} = 6$ d



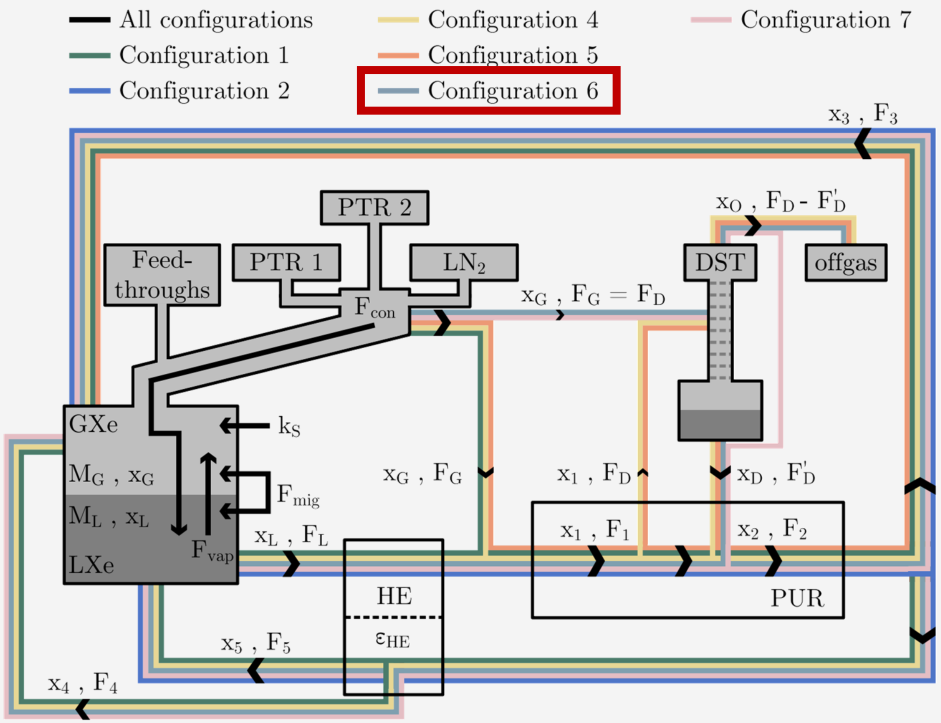
Online krypton removal for XENON1T

- Online Kr removal in configuration 4/5/6
- Reached a Kr conc. of ^{nat}Kr/Xe $< (360 \pm 60) \times 10^{-15}$ (ppq)

- Online Rn removal in configuration 7
- 20% reduction, no Kr conc. increase observed



Eur. Phys. J. C 81 (2021) 337



Event Rate in TPC (**ER**): Data give direct „online“ insight to innermost 700 kg of LXe (FV)

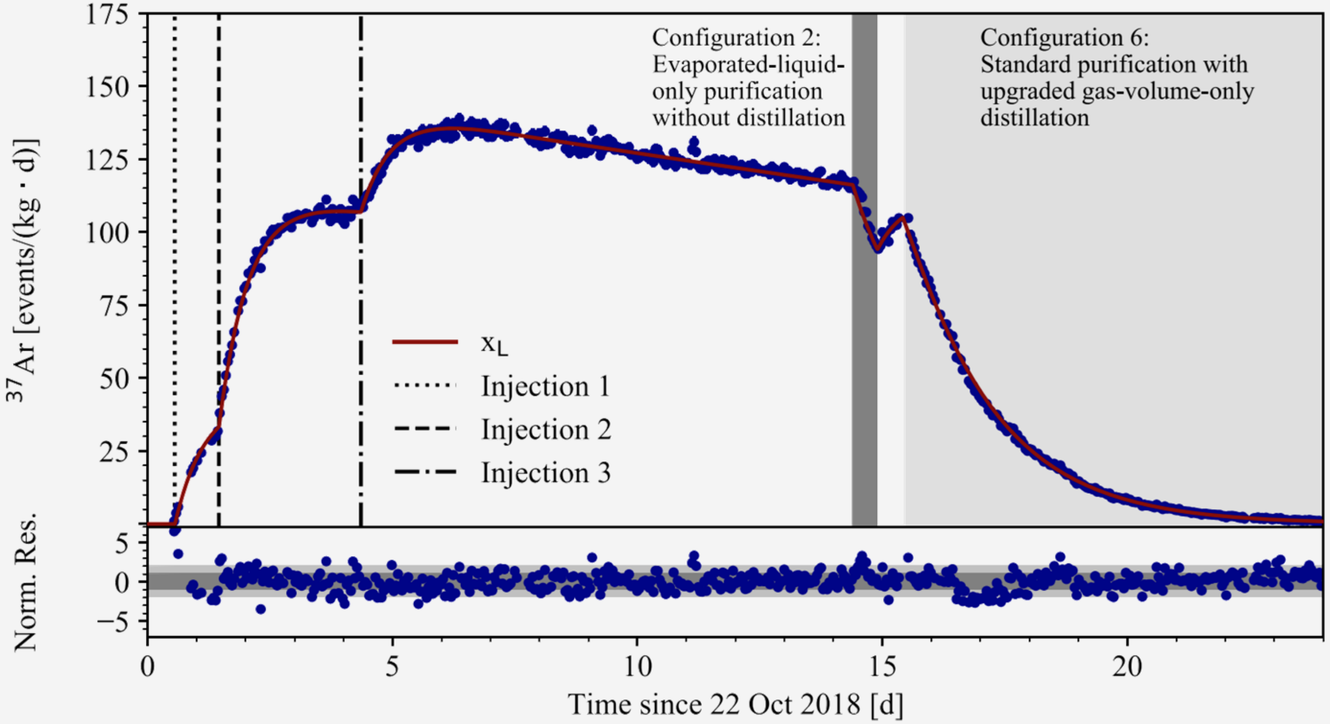
Rare Gas Mass Spectrometer (**RGMS**): Samples extracted from LXe for off-site analysis at MPIK Heidelberg

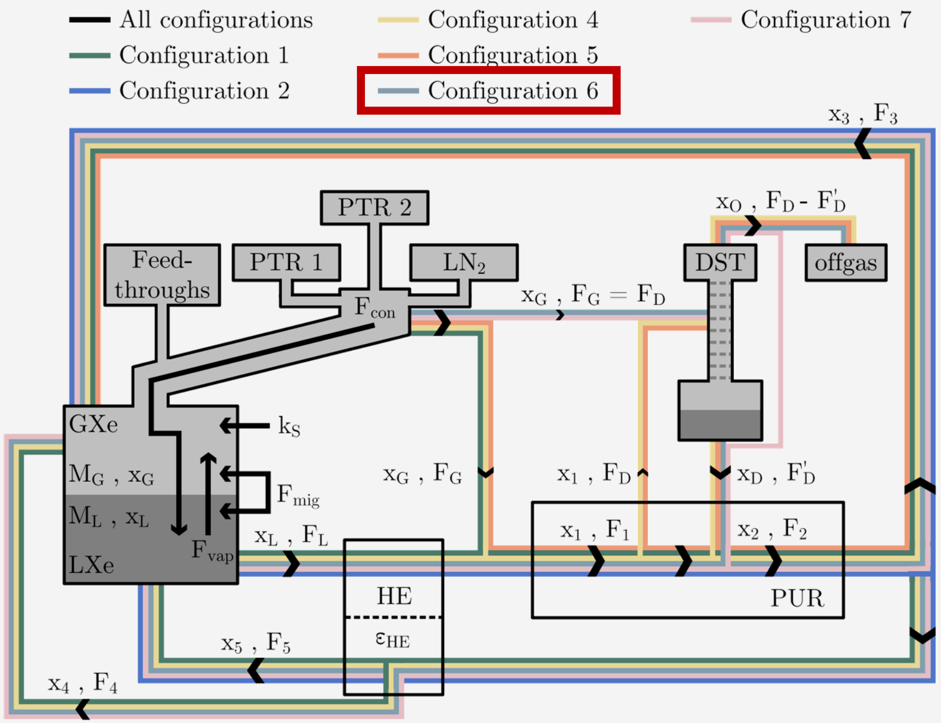
Eur. Phys. J. C (2014) 74:2746

Online Argon removal method for XENON1T

^{37}Ar calibration source:

- A gaseous ^{37}Ar source was deployed in XENON1T in October 2018
  Eur. Phys. J. C 83, 542 (2023)
- ER calibration at 2.8 keV and 0.27 keV (from EC)
- Half-life of 35 days – too long for regular use

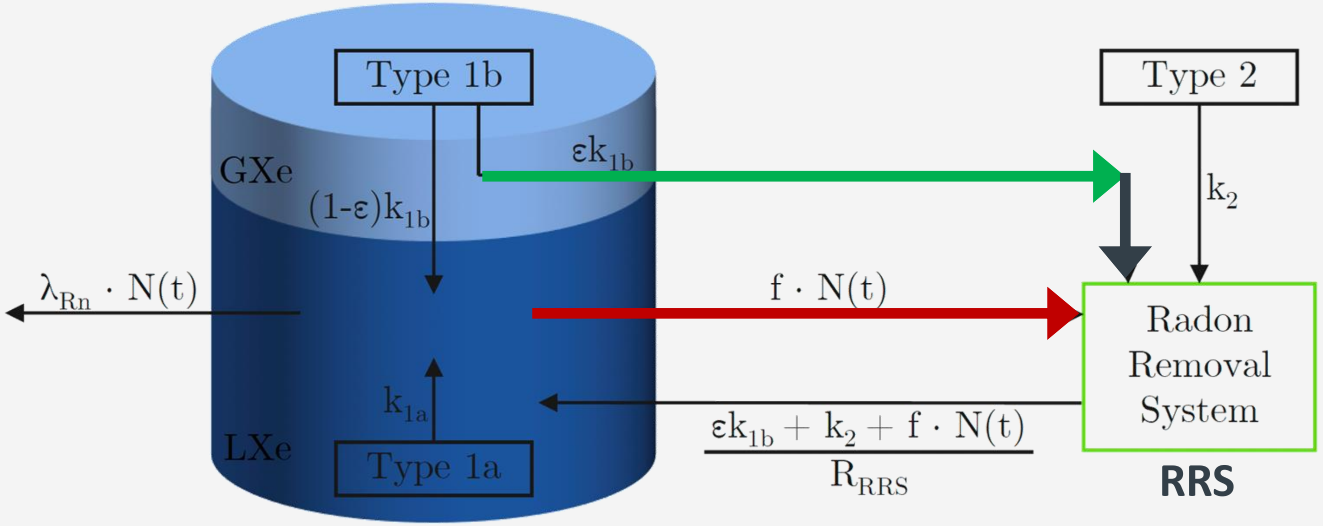




- Online Ar distillation in standard Kr removal configuration mode
- Reduction of the ^{37}Ar event rate with effective time constant of $\tau_{\text{eff, Ar}} = 1.7$ d
- Enabled ^{37}Ar as calibration source in XENONnT

Design, construction and commissioning of a high-flow radon removal system for XENONnT
Eur. Phys. J. C 82, 1104 (2022)

Radon removal strategy



Different Rn source types:

- **Type 1:** enters the detector **before RRS**
  - **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:** enters the **LXe phase** in the detector with a rate k_{1a}, can be extracted with a **low effective flow f to the RRS**
- **Type 2: enters the RRS** with a rate k_2 before the LXe phase

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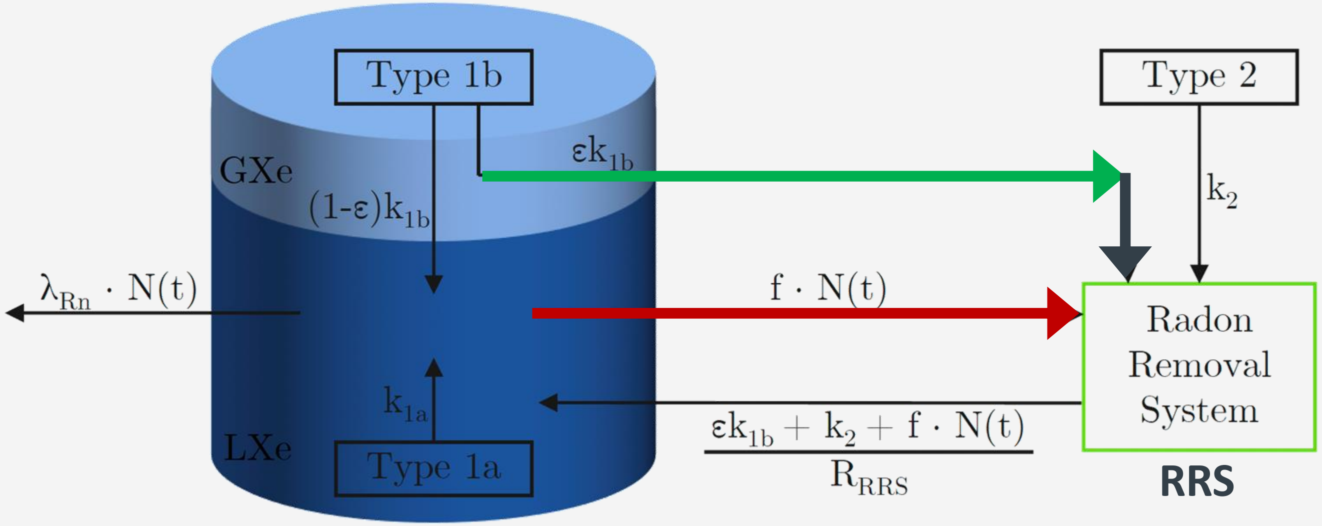

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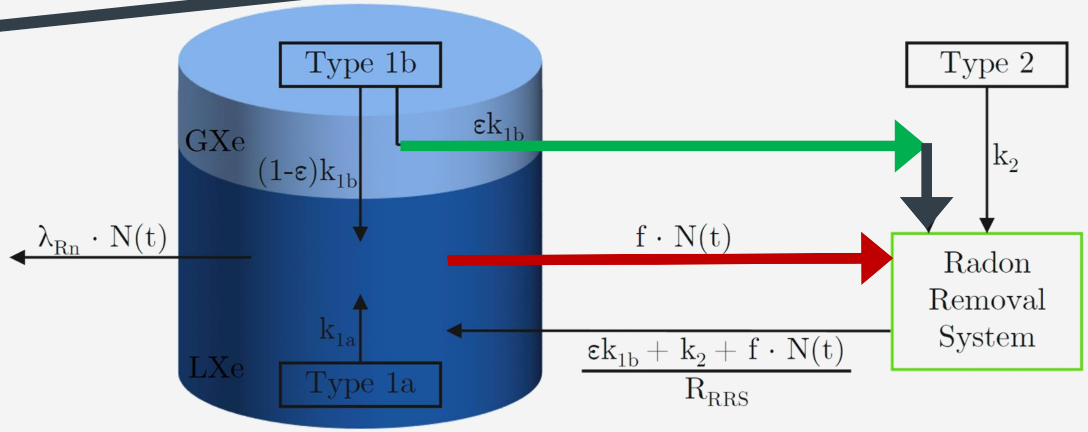
  - **Type 1a:** enters the **LXe phase** in the detector with a rate k_{1a}, can be extracted with a **low effective flow f to the RRS**
- **Type 2: enters the RRS** with a rate k_2 before the LXe phase

$$r(R_{\mathrm{RRS}} \to \infty, f, \epsilon) = \frac{\lambda_{\mathrm{Rn}} + f}{\lambda_{\mathrm{Rn}}} \cdot \frac{k_{\mathrm{tot}}}{k_{\mathrm{1a}} + (1-\epsilon) k_{\mathrm{1b}}} \approx 2 \cdot 2 = 4$$ for **XENONnT at a flow of ≈ 80 kg/h**

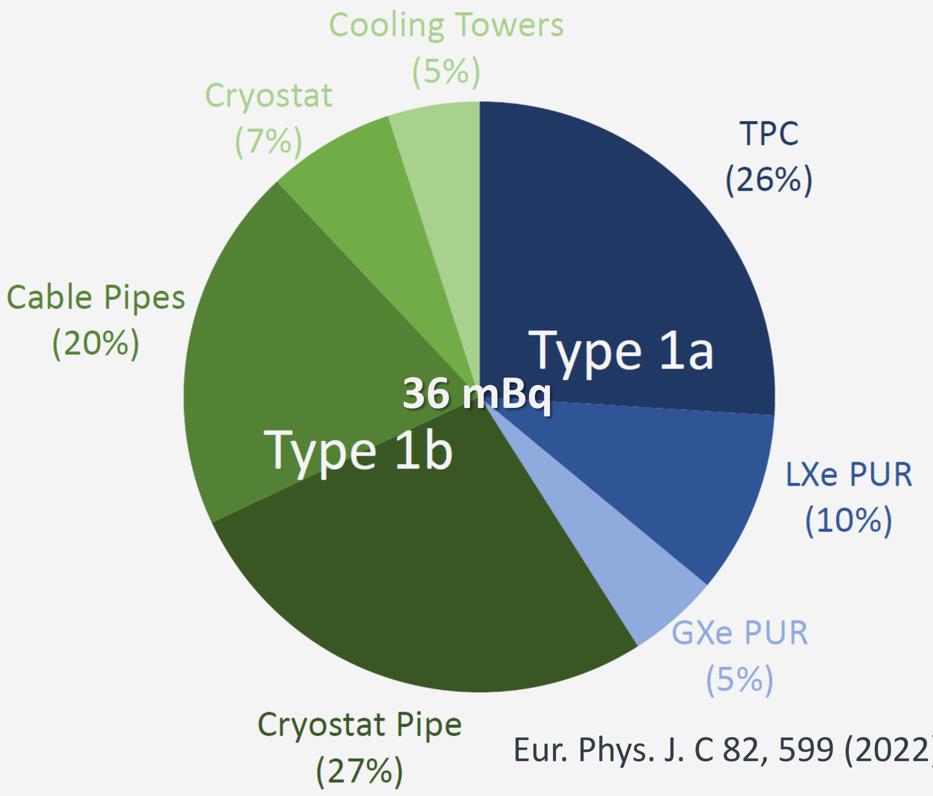
No GXe extraction ($\epsilon = 0$):
- Reduction of type 1 -> factor 2 for $f = \lambda_{\mathrm{Rn}}$
- Reduction factor with total LXe exchange time T: $r_{\mathrm{LXe}} \approx 1 + {}^{\tau_{\mathrm{Rn}}}/_{T} \approx 2$ to 4

GXe extraction ($\epsilon > 0$):
- Additional reduction converting type 1b to type 2
- Depending on extraction efficiency and source distribution of the experiment
- For XENONnT typically $r_{\mathrm{GXe}} \approx 2$

Radon removal strategy @XENONnT

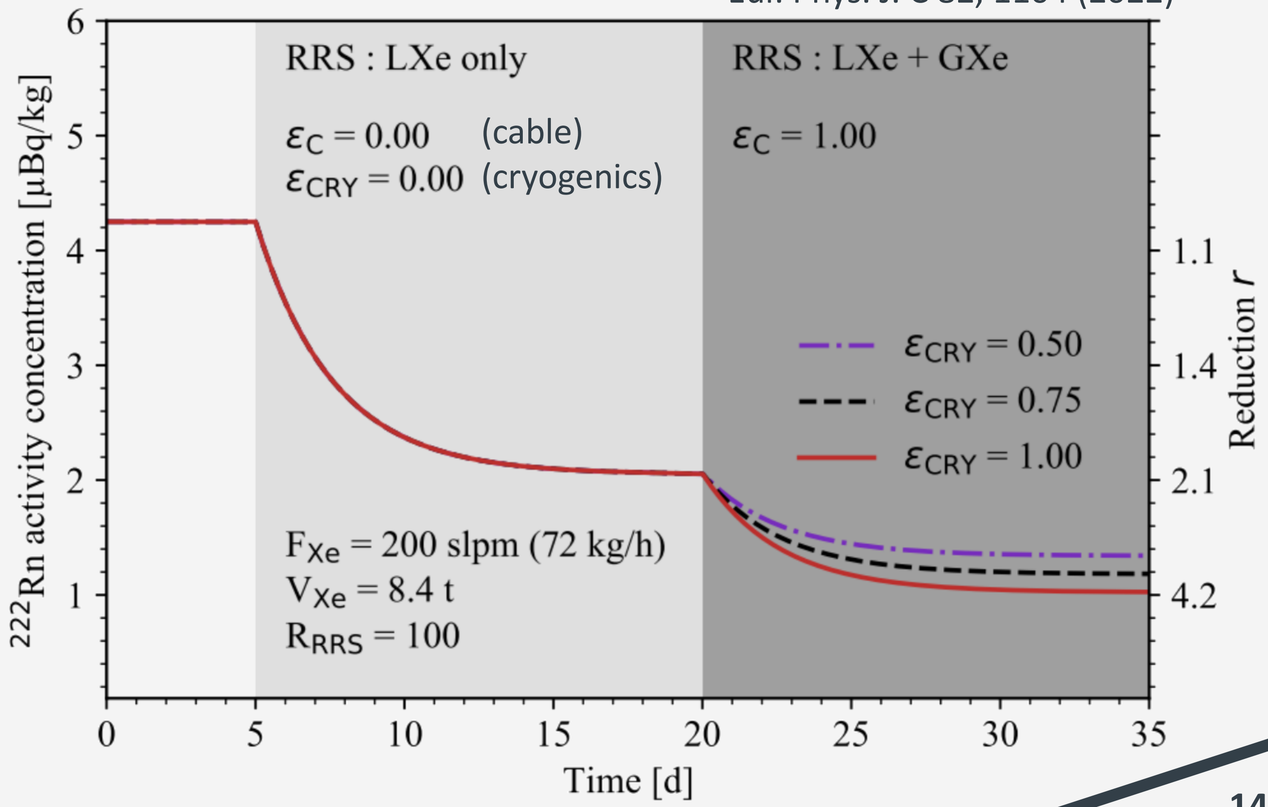


- Input from **material screening** and selection studies
- **XENONnT expectation** using the online radon removal model



Eur. Phys. J. C 82, 599 (2022)

Eur. Phys. J. C 82, 1104 (2022)



Eur. Phys. J. C 82, 1104 (2022)

Radon removal system for XENONnT

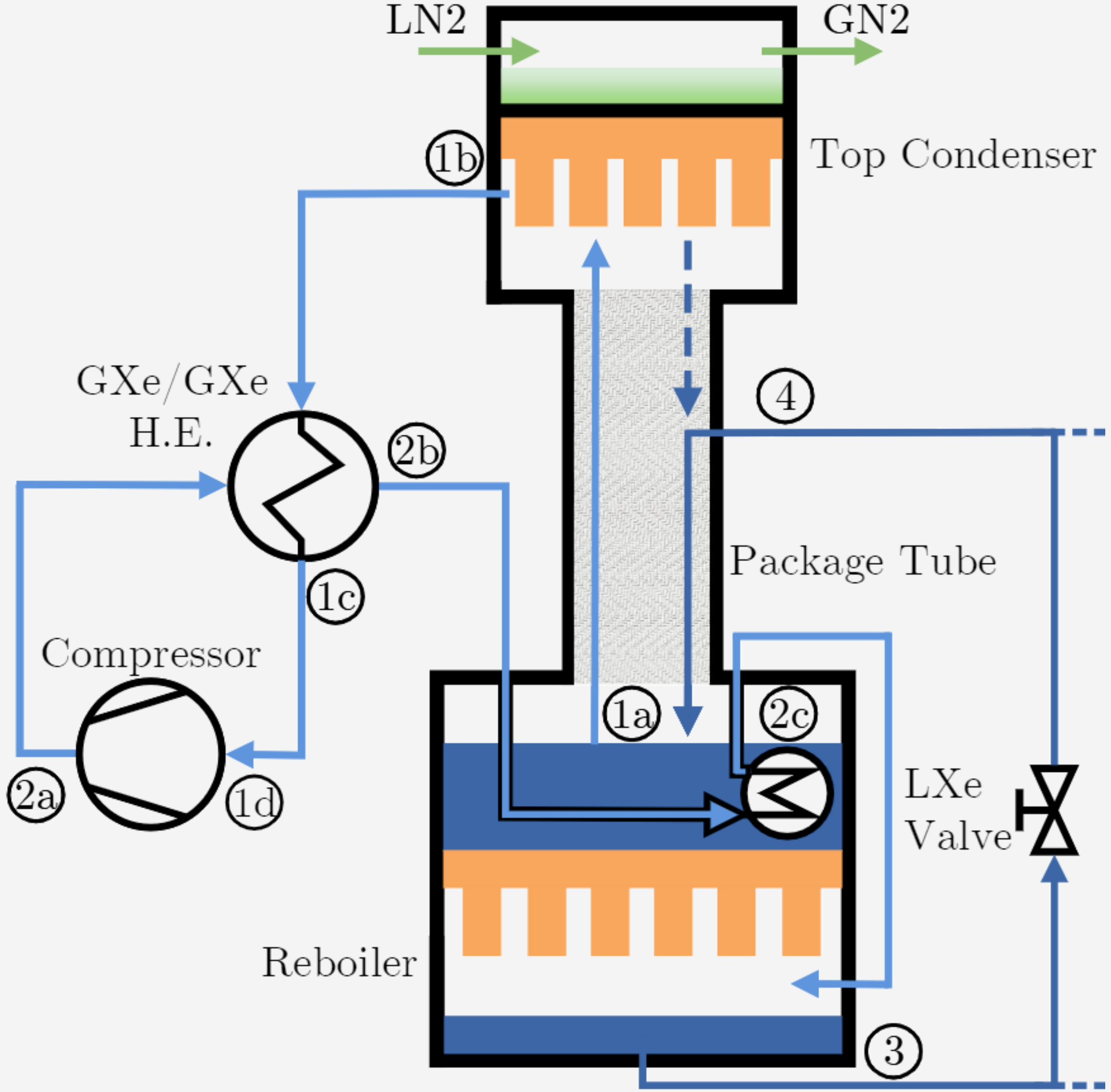
High-flow radon distillation column:

- Less volatile Rn trapped in reboiler until it decays
- Rn-depleted GXe extracted from top condenser

Design parameters:

- **Target flow:** 72 kg/h (200 slpm)
- **Reduction factor:** 100 between inlet and top
- **Enrichment factor:** 1000 between inlet and bottom
- **Reflux ratio:** 0.5
- LXe inlet and outlet

- Requires 1 kW cooling power at top
- Requires additional 2 kW cooling for LXe outlet



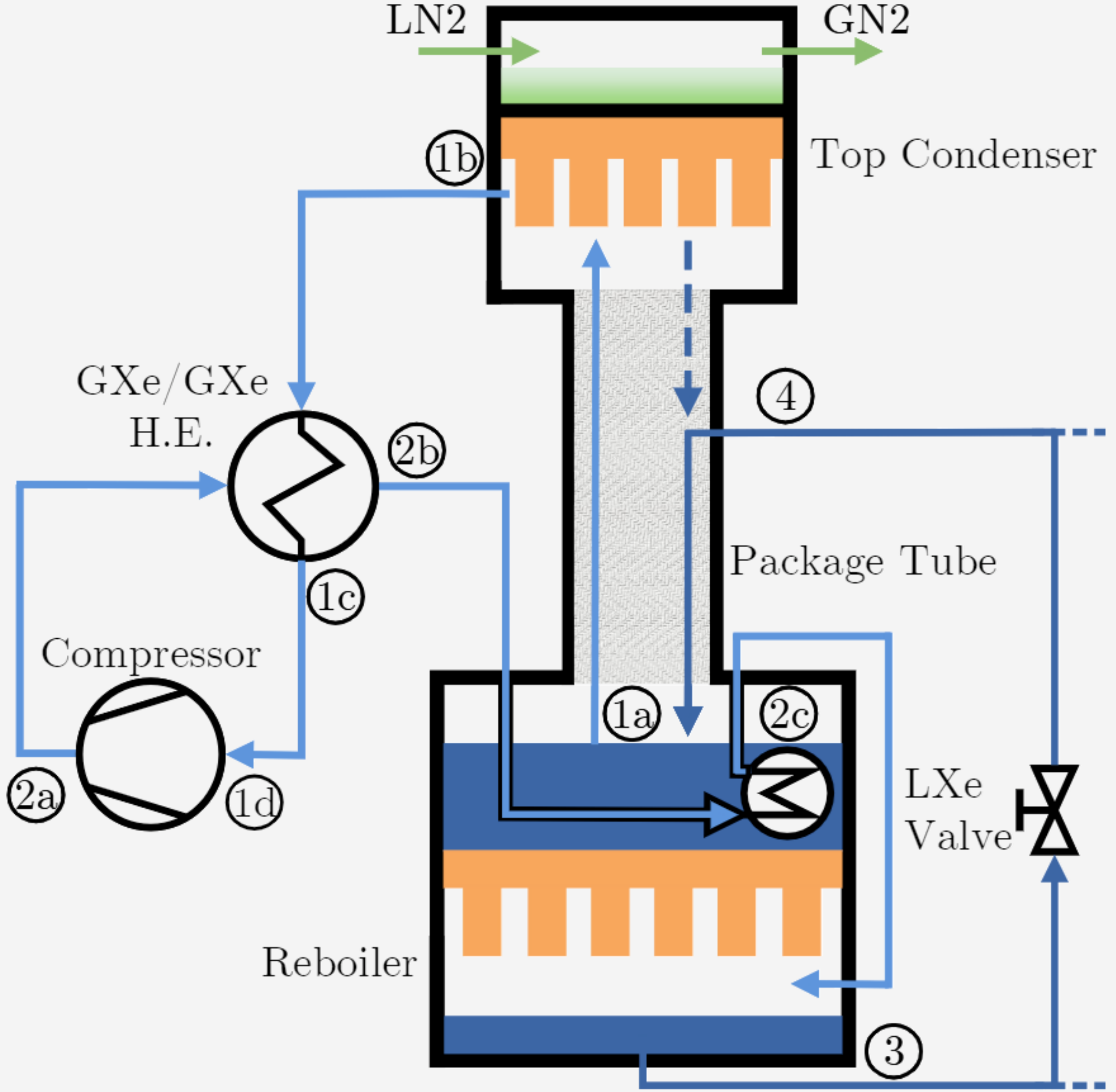
Eur. Phys. J. C 82, 1104 (2022)

Radon removal system for XENONnT

Thermodynamic concept:

- **Clausius-Rankine cycle** with phase changing medium xenon
- Reboiler acts as **heat exchanger** to liquefy Rn-depleted GXe with the stored Rn-enriched LXe
- Compressor acts as **heat-pump**
- Requires two special hardware developments
  - **Radon-free compressor**
    JINST 16 P09011 (2022), based on EPJ C78 604 (2018)
  - **Radon-free heat exchangers**
    JINST 17 P05037 (2022)

- Reduce required external cooling power from **3 kW to 1 kW**
- Drastically **reduce nitrogen consumption** and **electrical heating power**



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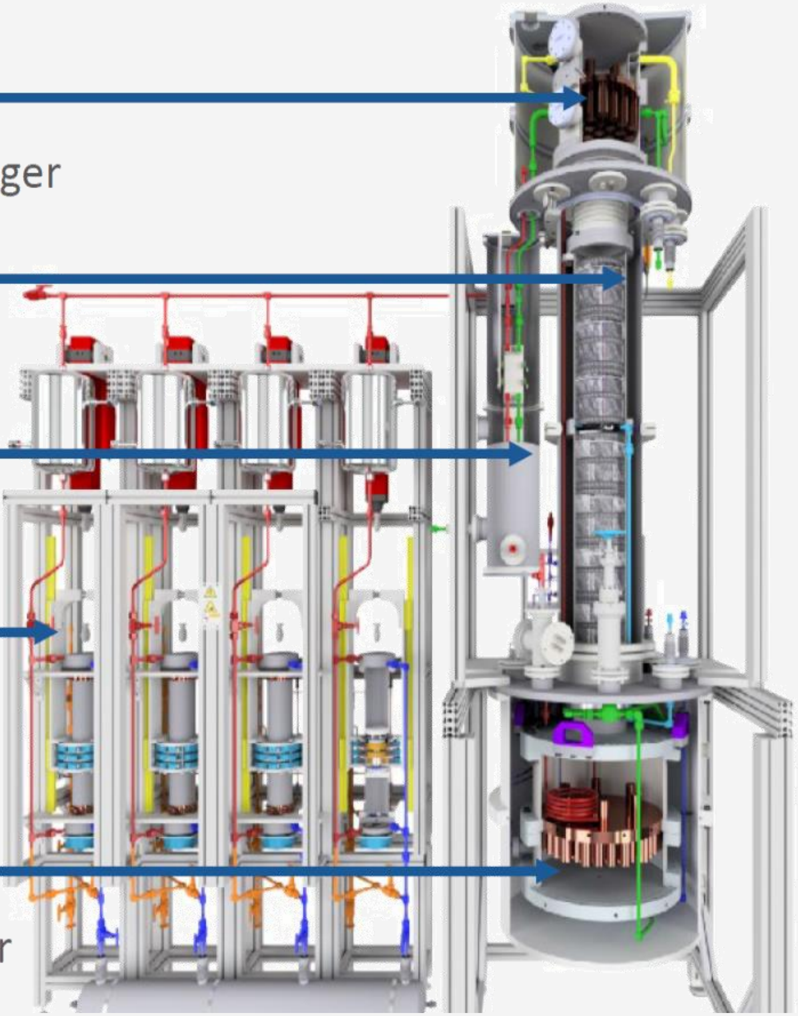


Top Condenser

Custom bath-type LN_2/GXe heat exhanger

Package Tube

Large surface package material

Auxiliary

Commercial GXe/GXe heat exchangers

Compressor

Custom four cylinder magnetically-coupled piston pump

Reboiler

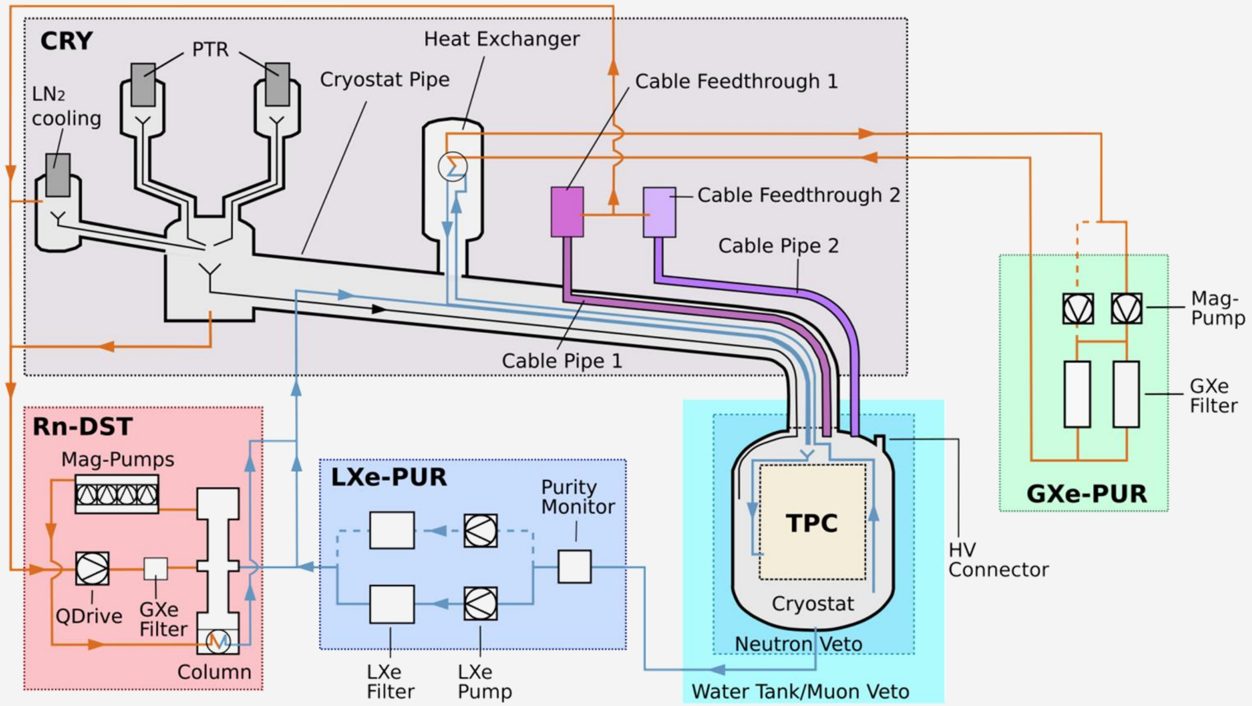
Custom bath-type Xe/Xe heat exhanger

Eur. Phys. J. C 82, 599 (2022)

Radon removal system for XENONnT

Operation @XENONnT:

- **Reduction factor 2** for cryogenics‘ sources by **GXe extraction at 25 slpm**
- **Another reduction by factor of 2** for sources within detector by **high-flow LXe extraction at 200 slpm**



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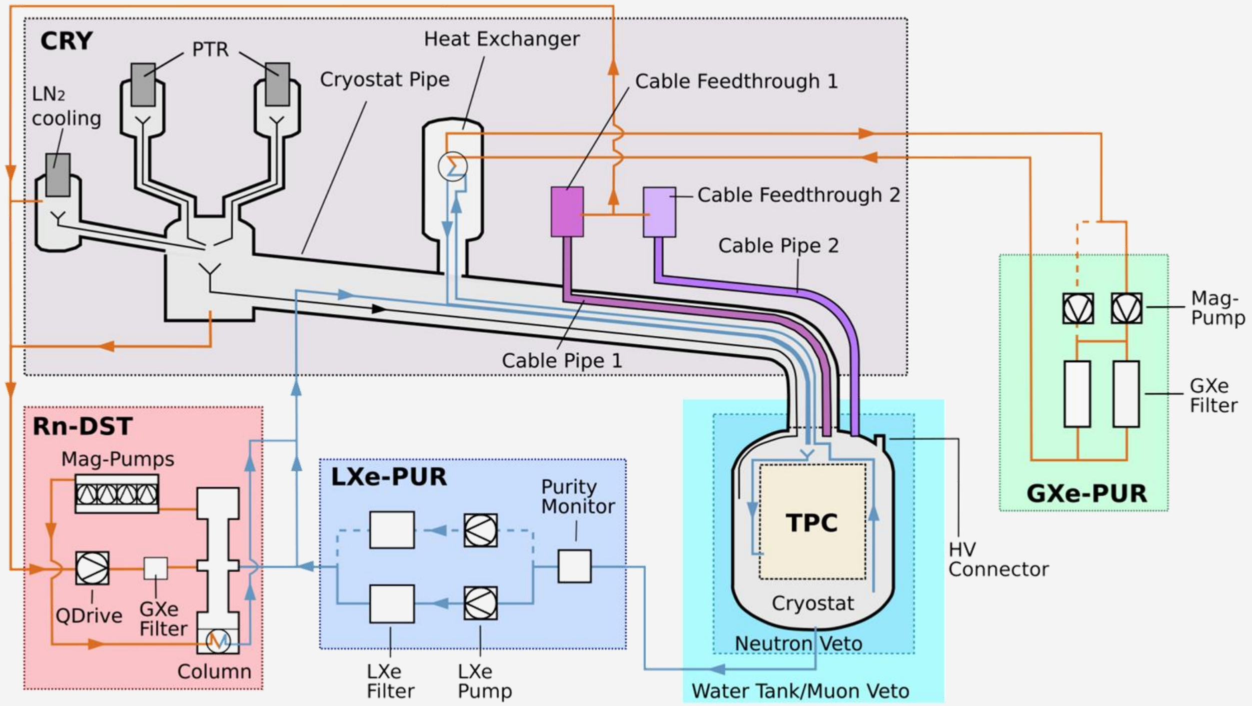
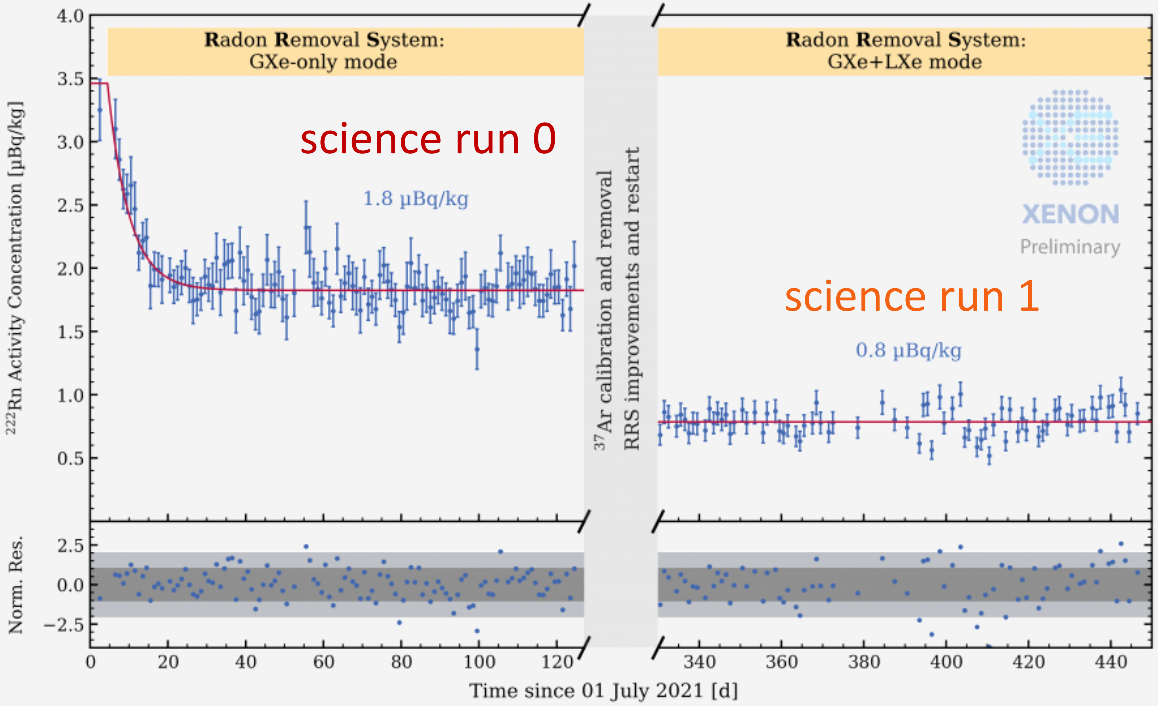


Radon concentration at XENONnT

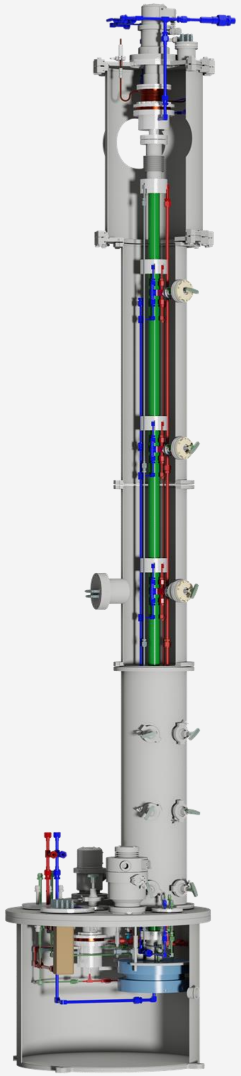


XENONnT as Rn source and monitor:

- **No RRS:** 3.4 µBq/kg
- SR0 – **GXe-only:** 1.8 µBq/kg
- SR1 – **GXe + LXe:** 0.8 µBq/kg

Rn-induced background rate in [1, 10] keV same as expected by solar pp neutrinos induced rate

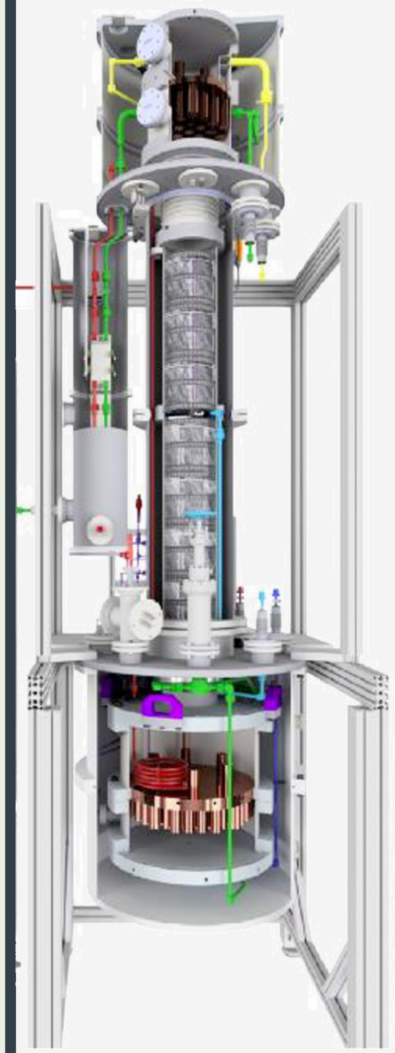
Krypton and radon removal @XENONnT



Krypton distillation

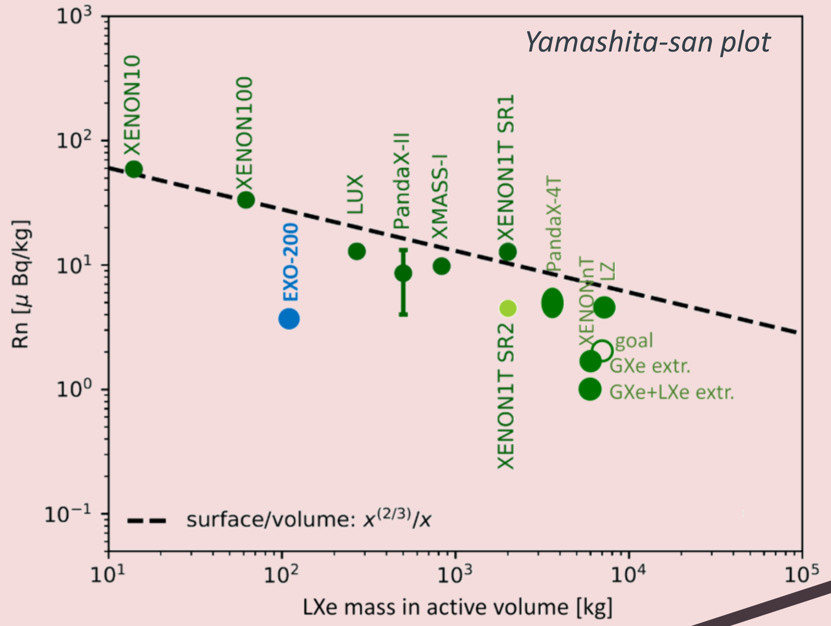
- **Offline distillation demonstrated**
  ^{nat}Kr/Xe $< 0.026 \times 10^{-12}$ (26 ppq)
- **Online distillation @XENON1T**
  ^{nat}Kr/Xe $< (360 \pm 60) \times 10^{-15}$ (ppq)
- **Offline + online distillation @XENONnT**
  ^{nat}Kr/Xe $< (56 \pm 36) \times 10^{-15}$ (ppq)
  - Processed 6 t commercial xenon offline
  - Additional 3 weeks of online distillation

- **Xe recovery:** 99% (1% offgas)
- Kr can re-enter via (tiny) air leaks
- **1 day of Kr distillation generates ~ 0.7 kg offgas**



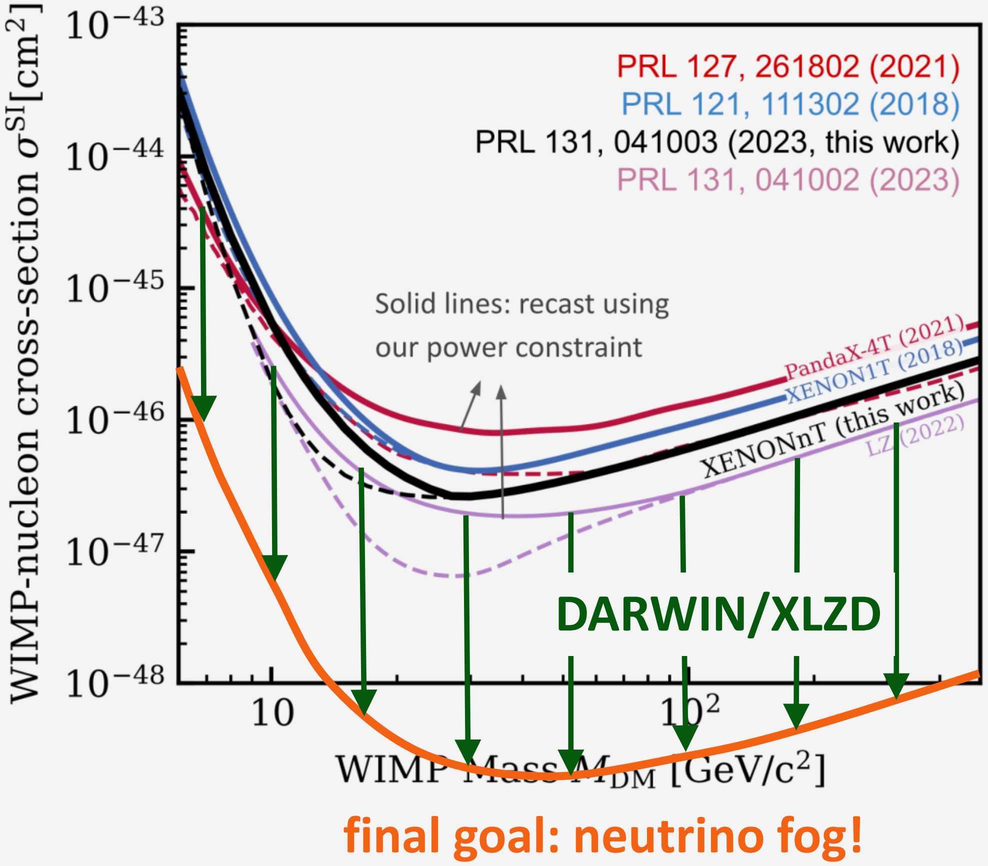
Radon distillation

- SR0 – GXe-only: 1.8 µBq/kg
- SR1 – **GXe + LXe: 0.8 µBq/kg**
  with 200 slpm GXe + 25 slpm LXe



Radon requirements for future experiments – Towards DARWIN/XLZD

- Next-generation experiments with > **50 t of LXe**
- **Multi-purpose observatory** for **dark matter**, **neutrino** and **rare events**, probing WIMPs down to the **neutrino fog**
- **Huge requirements for the ^{222}Rn concentration!**
  - ^{222}Rn continuously emanates from detector materials
  - Additional **factor 10 reduction**
  - Need combination of many Rn mitigation strategies!



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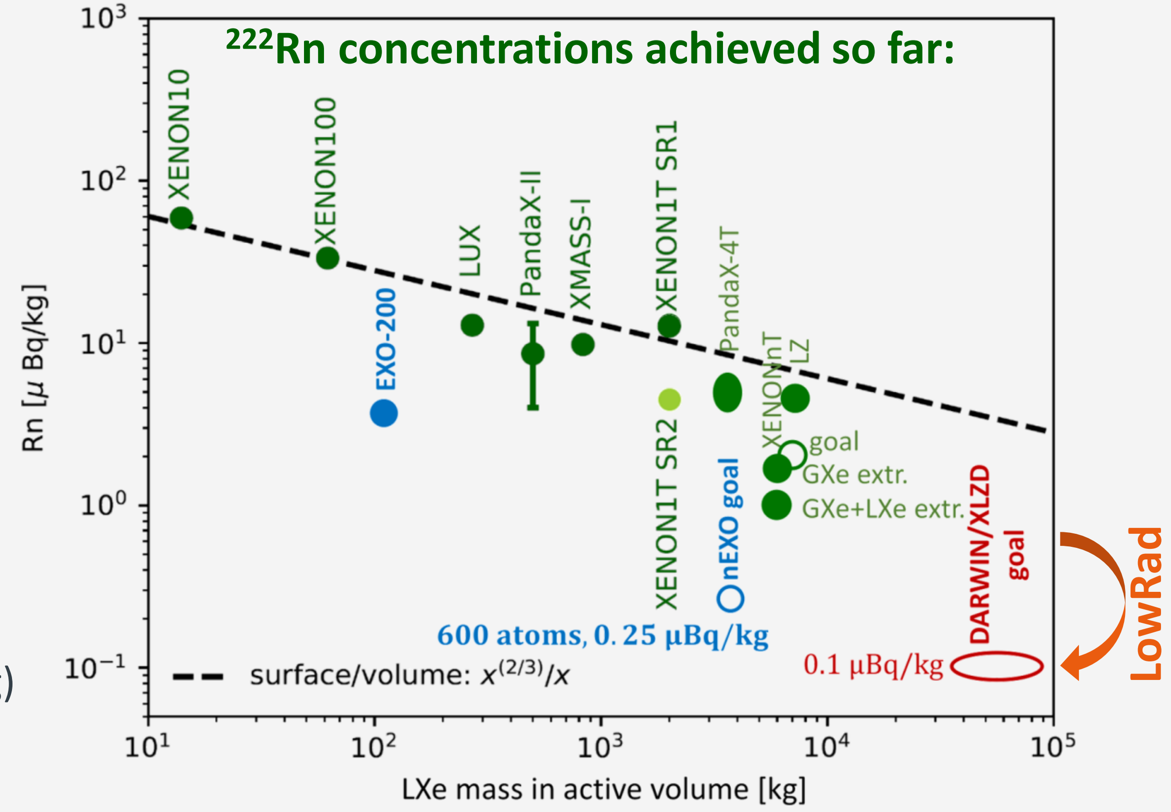
Rn background rate 10 times smaller than solar neutrino contribution



Yamashita-san plot

ERC Advanced Grant LowRad of C. Weinheimer:

- **Develop technologies** for:
  - Continuous/**online ^{85}Kr removal** (30 ppq ^{nat}Kr)
  - Another **factor 10** in **^{222}Rn reduction** (0.1 µBq/kg)
- R&D for novel purification methods
- Methods for physics searches/analyses
- Complete **purification & distillation demonstrator**

- **Reach: "less than 1 Radon atom in 100 mol of xenon"**

Key aspects of the LowRad project

How to purify 50 t of Xe from Rn in ≤ 2d ?

- **Full heat pump** to achieve enormous cooling throughput: **75 kg/h (LowRad demonstrator)**
  750 kg/h (final system)

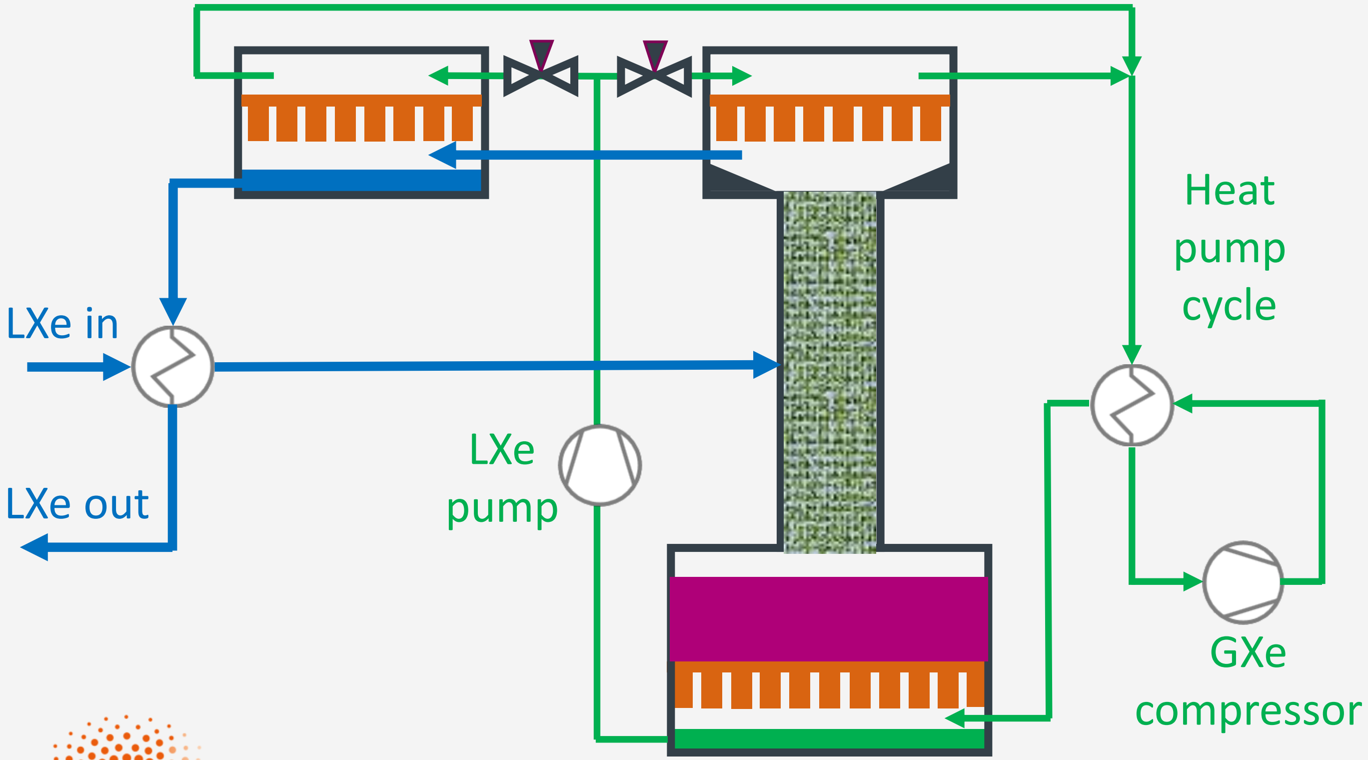
Demonstrator

- Radon-free heat exchangers
- 2nd Xe **heat pump** cycle
- With online Rn decay monitor

Final system

- Should be integrated with **purification system** for removal of electronegative impurities
- With **online Kr removal system**
- Installed in a **water shield** to avoid Xe activation

R&D and demonstrator within ERC AdG LowRad



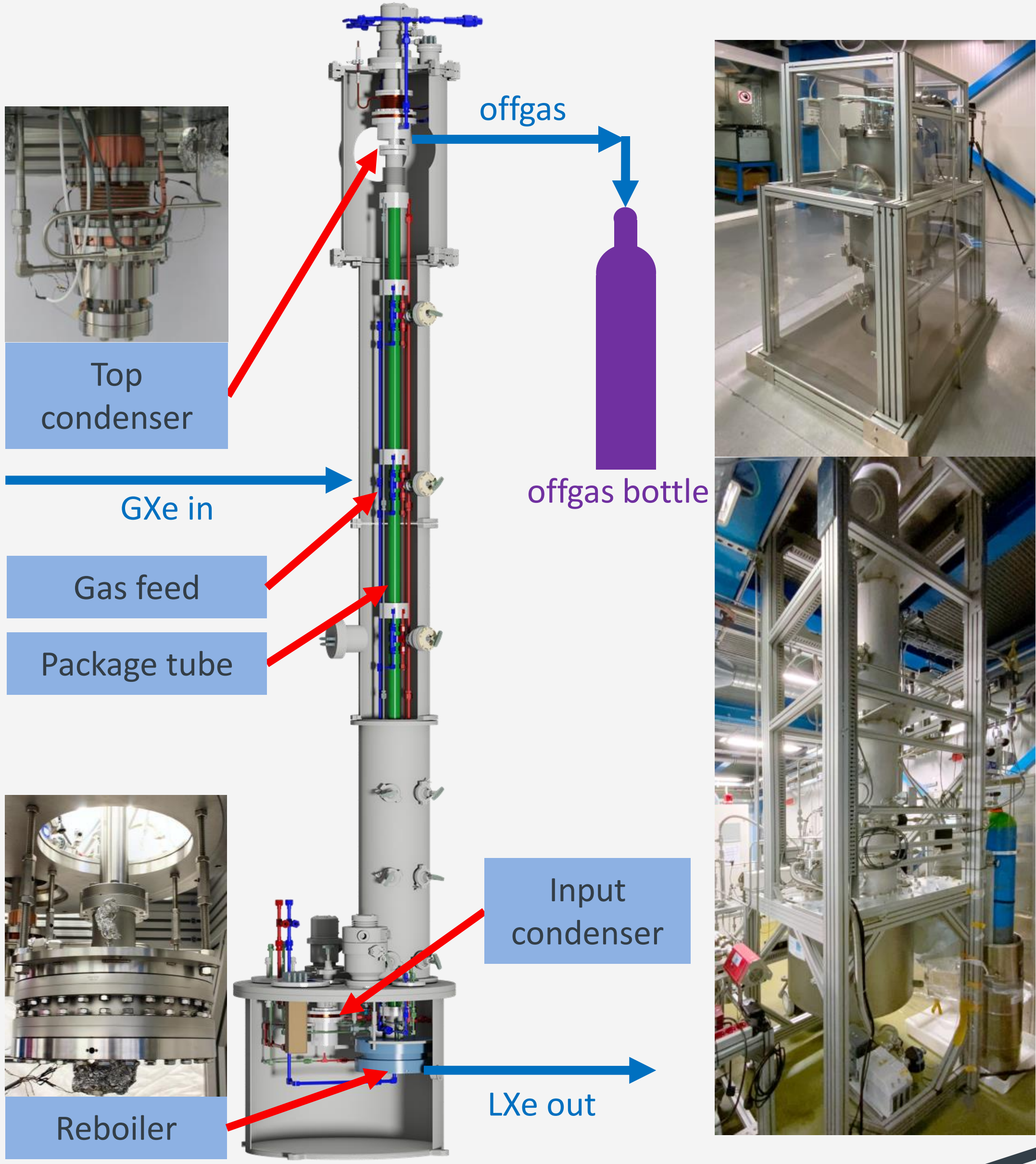
R&D currently focusing on:

- Kr-concentrator distillation system
- Demonstration of a Xe heat pump concept

Krypton distillation for XENON1T/nT

Challenges of the current system

- Initially **not designed for continuous online distillation**
- Limited to gas flows of up to 18 slpm = 6.5 kg/h
- Planned for an **offgas fraction of 1%**
- **Replacement** of the **offgas bottle** after ~ 60 days of distillation



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Online distillation for DARWIN/XLZD

Assuming a runtime of 5 years for a 50 t detector

distillation speed, offgas fraction

$$5\,\mathrm{yr} \times 8.3\,\mathrm{slpm} \times \frac{50\,\mathrm{t}}{8.6\,\mathrm{t}} \times 10^{-2}$$

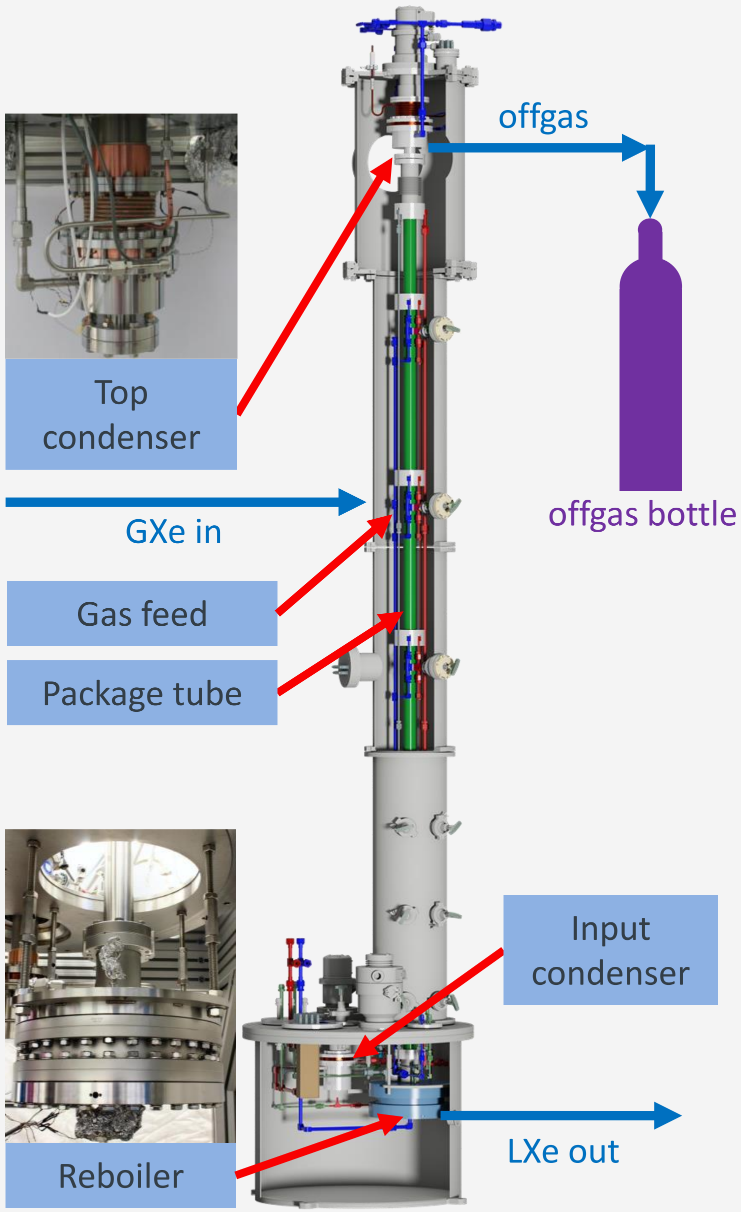
runtime, detector mass scaling

$$= 5\,\mathrm{yr} \times 3\,\mathrm{kg/h} \times 5.81 \times 10^{-2}$$

$$= 5\,\mathrm{yr} \times 0.174\,\mathrm{kg/h}$$

$$= 7620\,\mathrm{kg}$$

$\Rightarrow$ 4 kg/d offgas



Eur. Phys. J. C 77 (2017) 275

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Online distillation for DARWIN/XLZD

Assuming a runtime of 5 years for a 50 t detector

distillation speed — offgas fraction — offgas 2 fraction

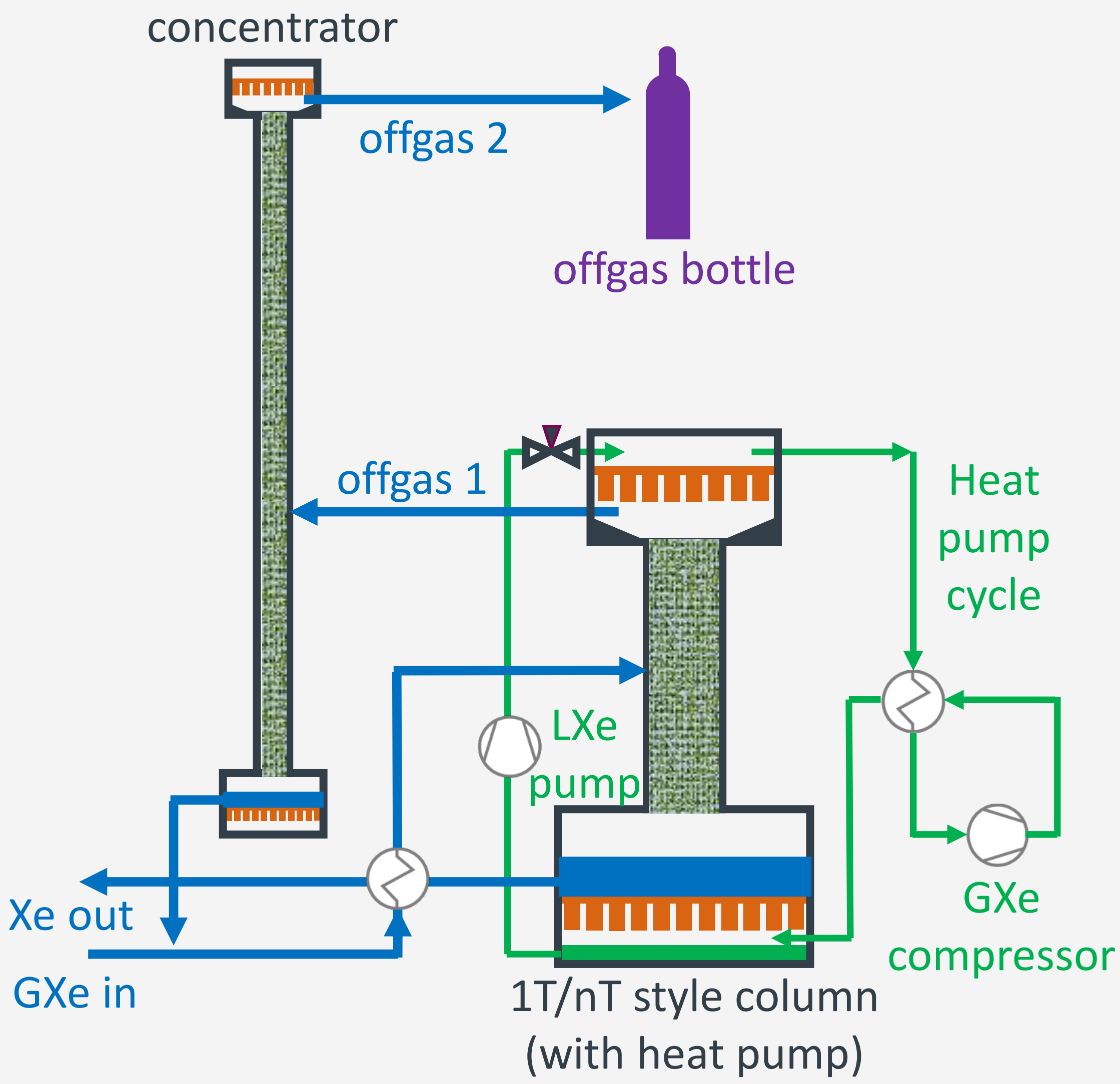
$$5\,\mathrm{yr} \times 8.3\,\mathrm{slpm} \times \frac{50\,\mathrm{t}}{8.6\,\mathrm{t}} \times 10^{-2} \times 10^{-3} = 5\,\mathrm{yr} \times 0.174\,\mathrm{g/h}$$

$$= 7.6\,\mathrm{kg}$$

runtime — detector mass scaling

⇒ 4 g/d offgas

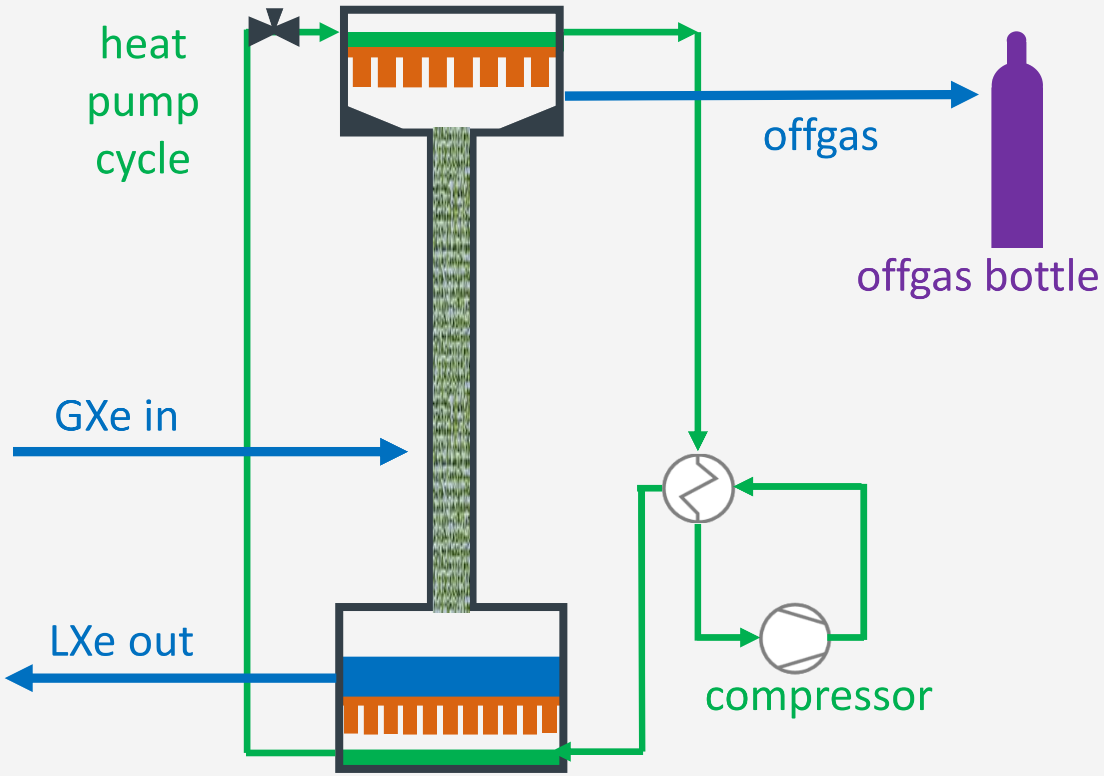
⇒ Can be easily compensated



LowRad prototype construction

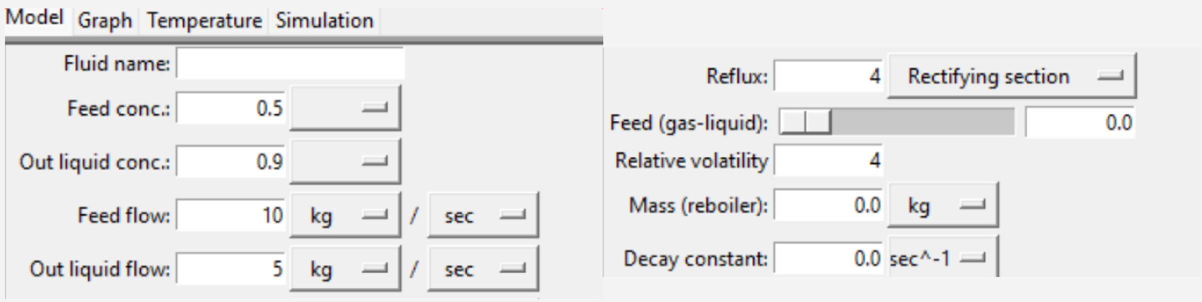
Krypton concentrator prototype

- Similar scale as the final concentrator
- Design as a tool – “as simple as possible”
- Demonstration of **high separation** and the $\mathbf{10^{-3}}$ **offgas fraction** for **low flows** (few slpm)
- Design and test of a **heat pump cycle** using a suitable process gas (design for Xe)



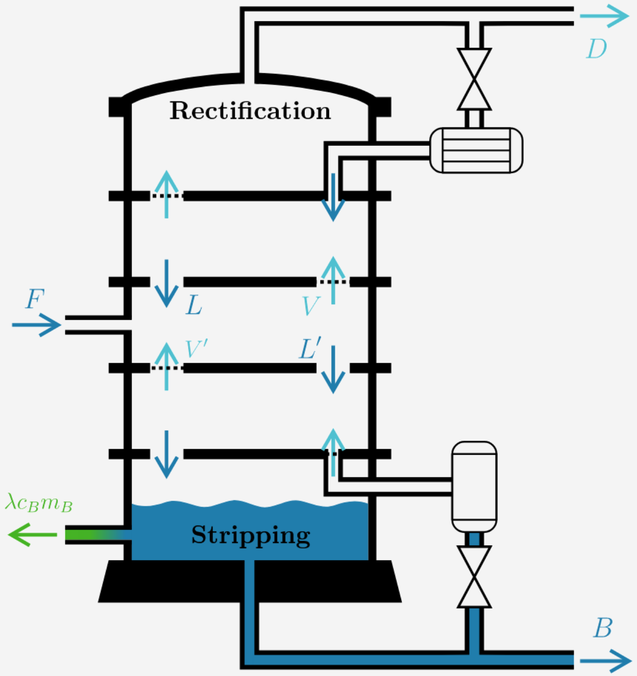
Concentrator with heat pump

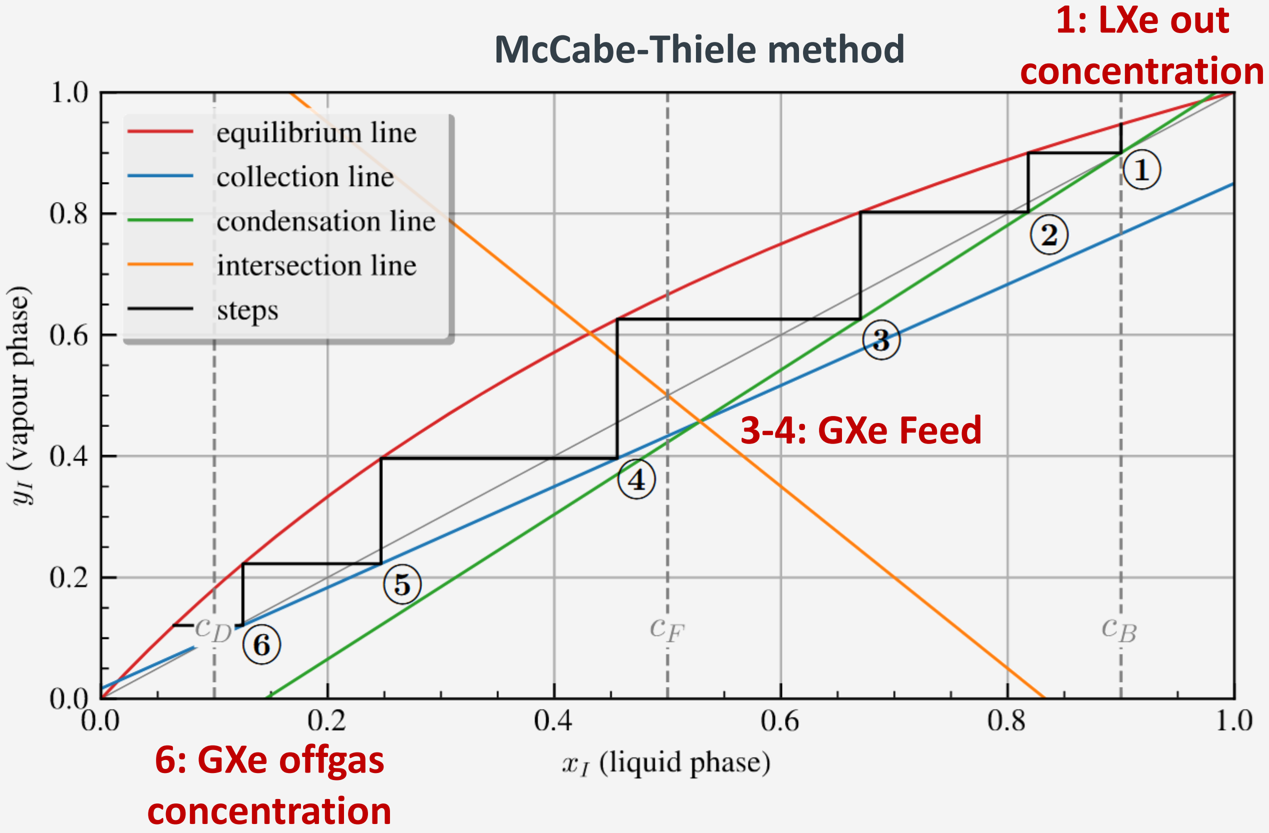
LowRad prototype construction



Development of a McCabe-Thiele calculator

- Versatile McCabe-Thiele calculator
- Estimation for **required number of distillation stages** and **expected performance**
- Arbitrary selection of conditions
- Integration of TESPy and CoolProb tools





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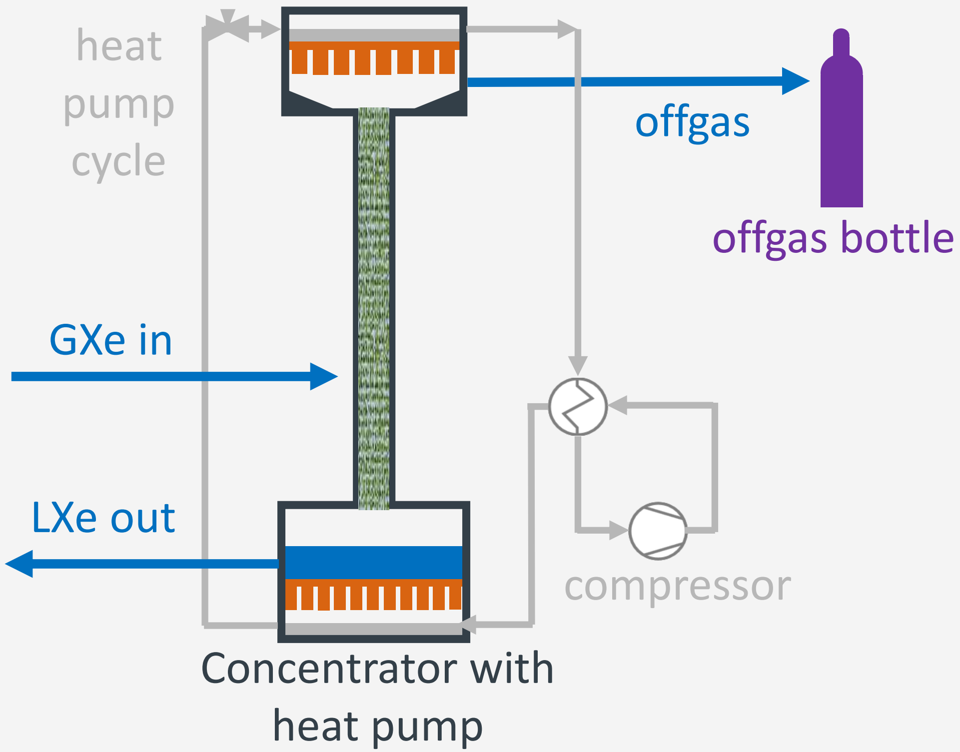
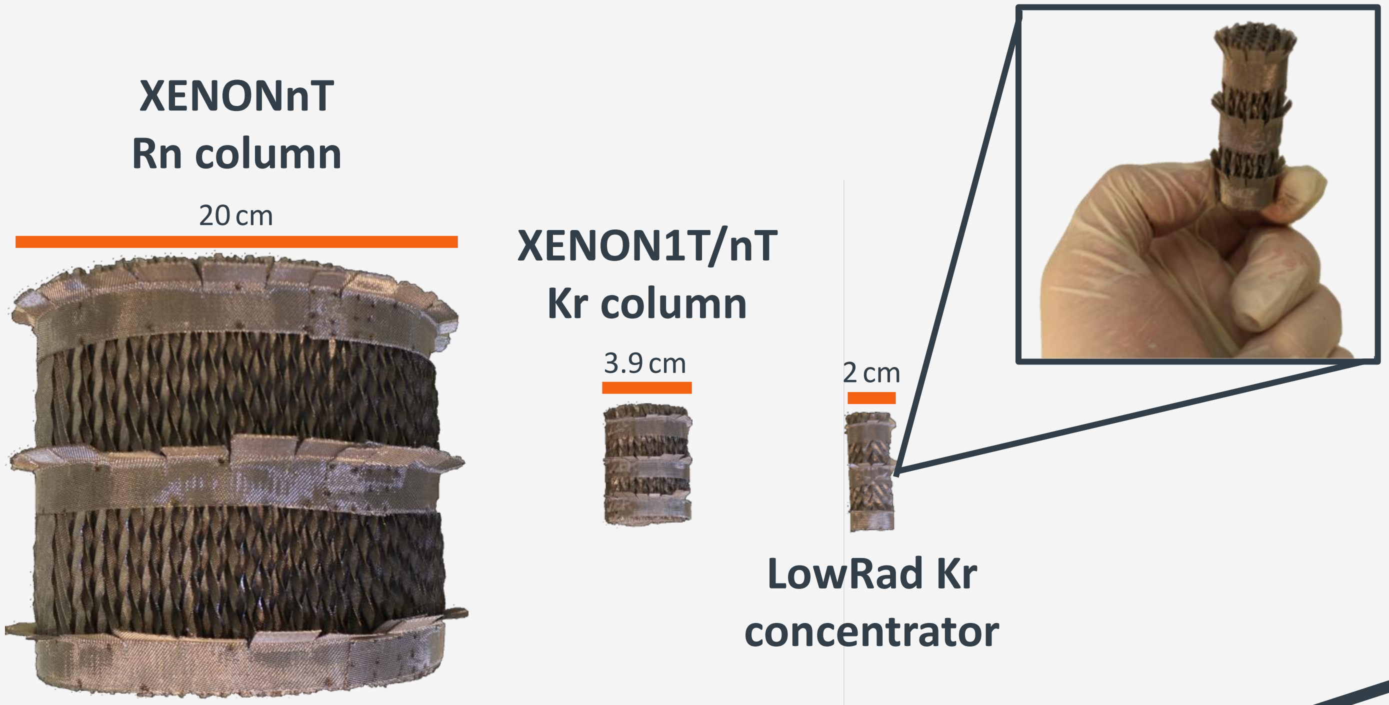
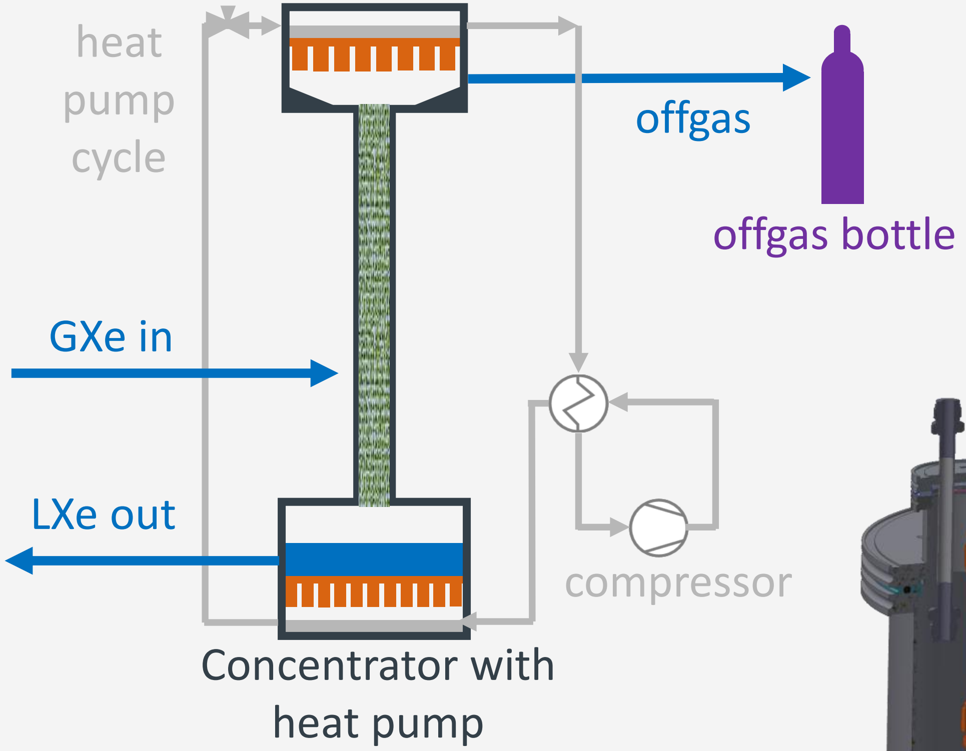


Krypton distillation column

- Size of the condenser and reboiler depend on the heat pump design
- Package height of 2.2 m (extension possible)
- Using a 2 cm diameter package material (type EX, Sulzer AG)
- Height-equivalent of one theoretical plate (HETP): 9-24 cm (PhD M. Murra)



LowRad prototype construction

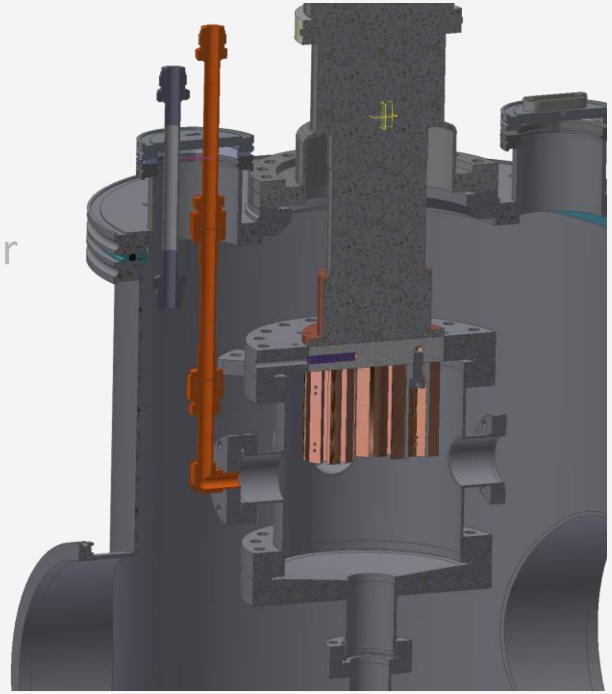


Krypton distillation column

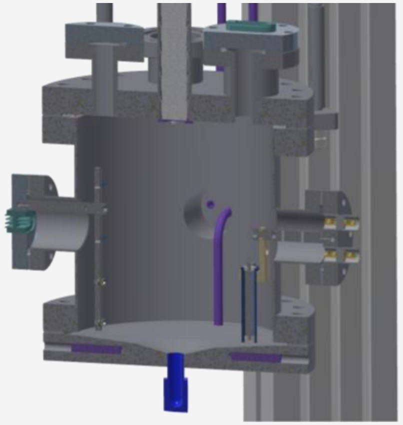
- Demonstrate performance using regular cold heads
- Modular design to switch to heat pump concept (re-evaluate performance)
- Characterize **separation** and show $\mathbf{10^{-3}}$ **offgas fraction** for **low flows** (few slpm)

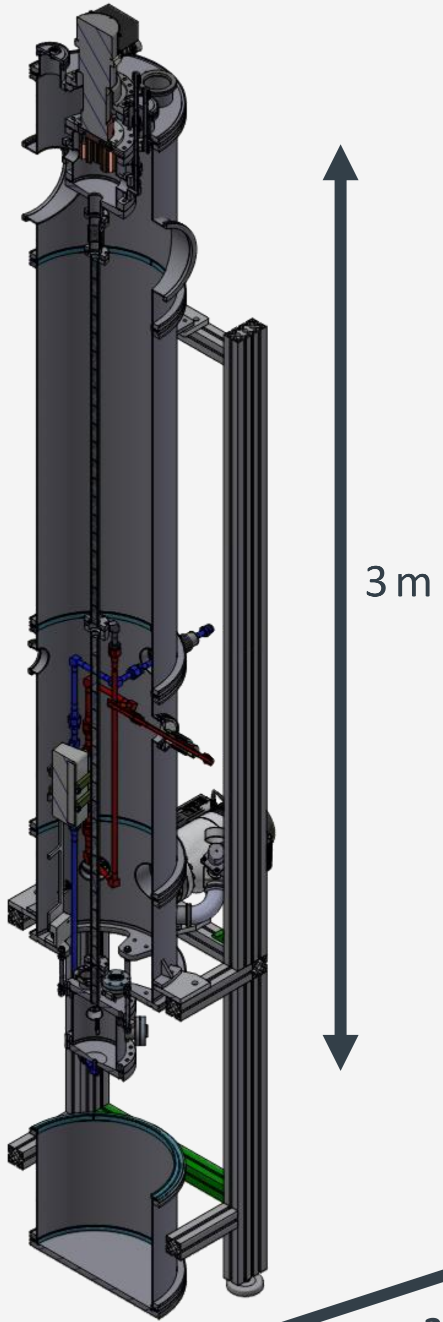
Inlet: 1 kg/h (3 slpm)
Reflux: 1200
Theoretical stages: 9

Top condenser



Bottom reboiler





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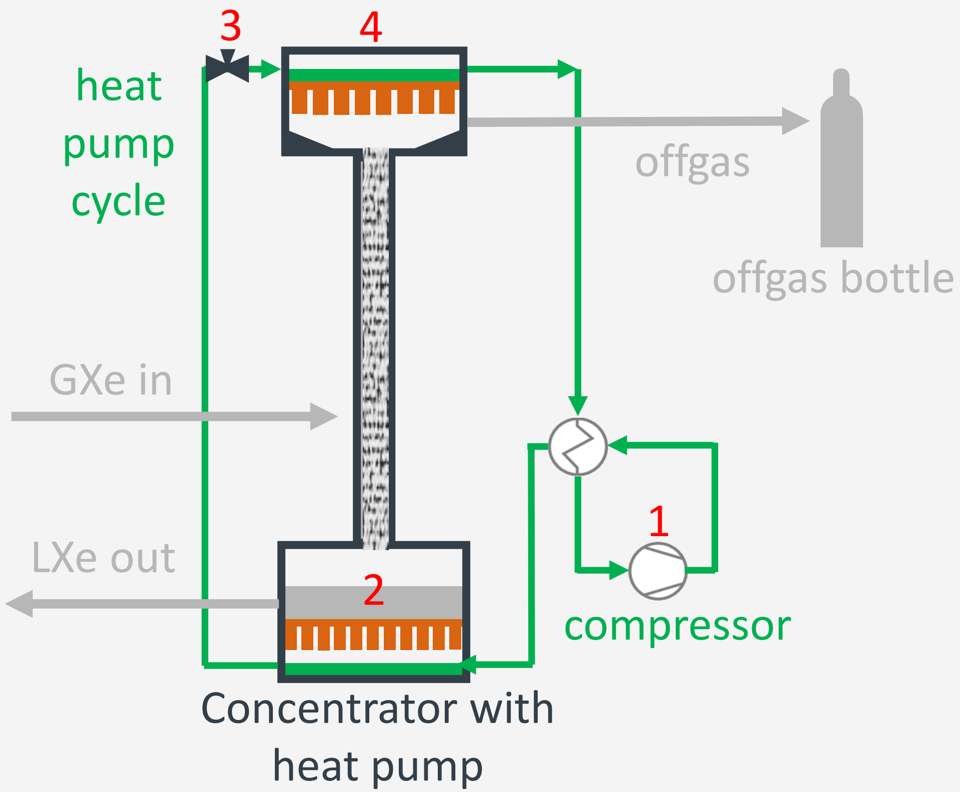
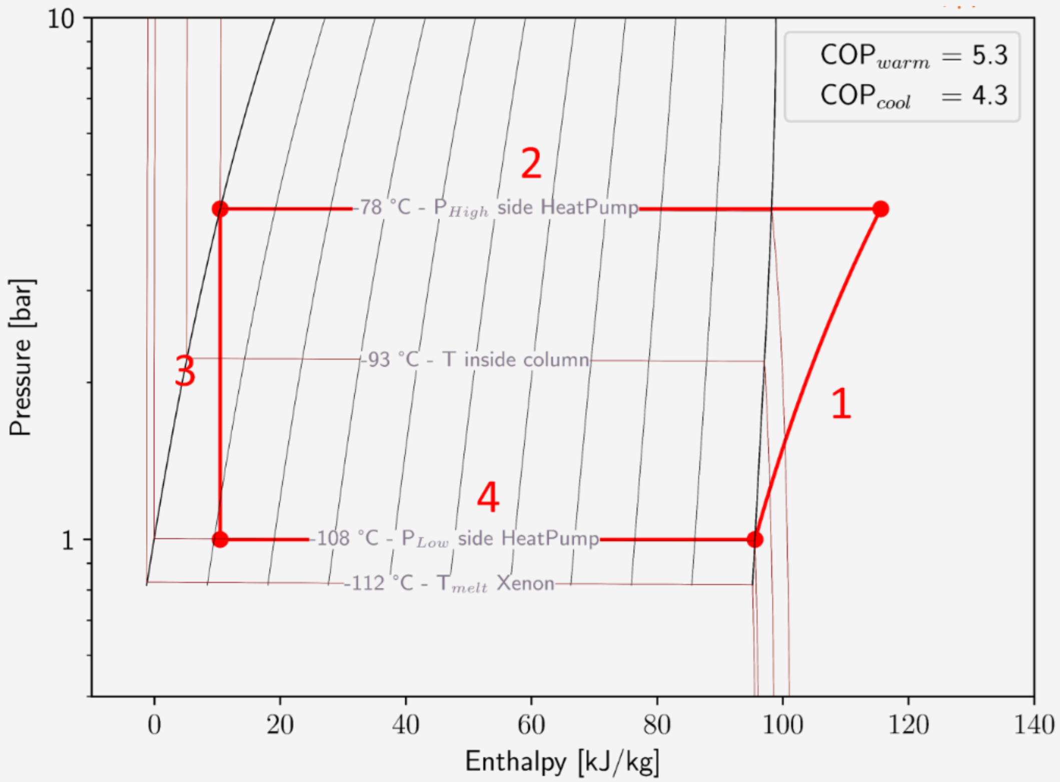


1: compression
2: condensation
3: expansion
4: evaporation

Design of a heat pump cycle

- Investigating several process gases including Xe
- Need to supply the required heating and cooling power at reboiler and condenser (roughly 10 W per 1 slpm)
- Design using custom numerical calculator for heat pump concepts/heat cycles
- Construction of first test setup started



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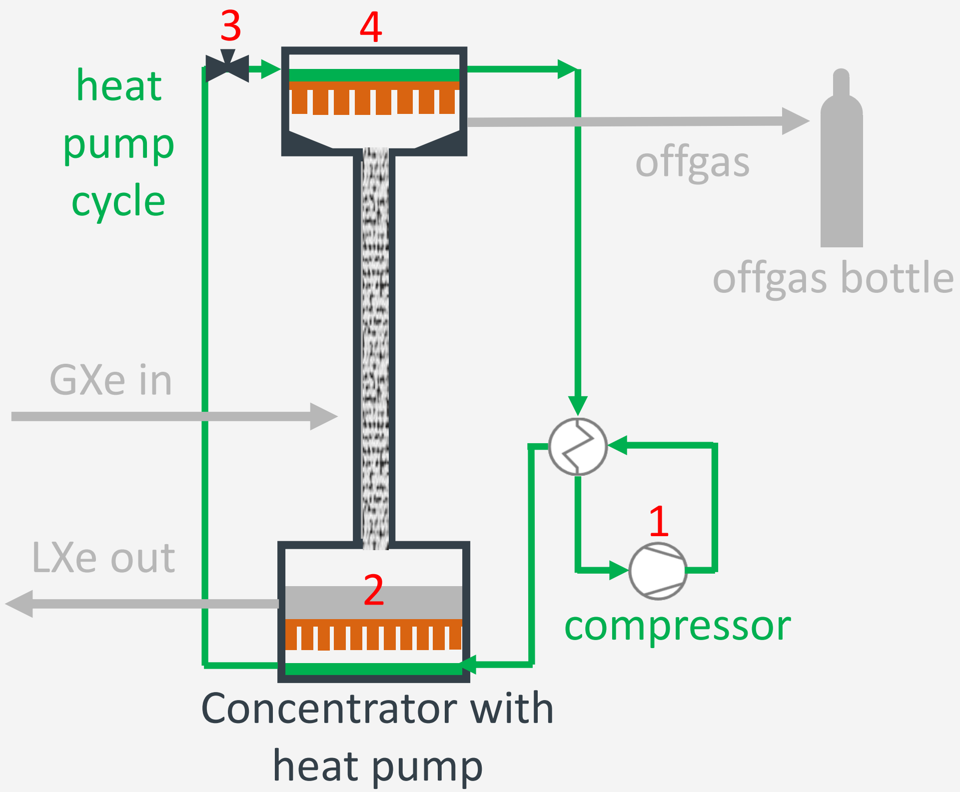

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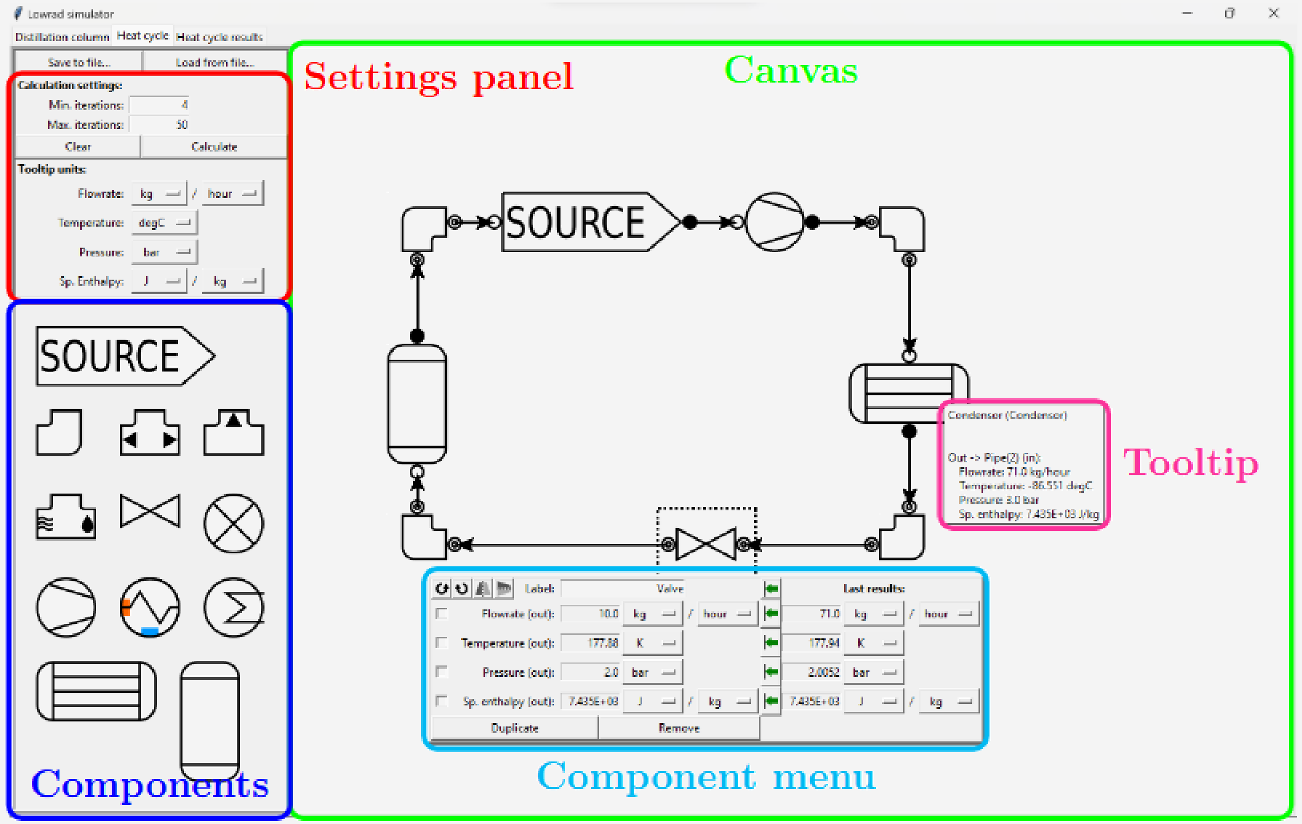

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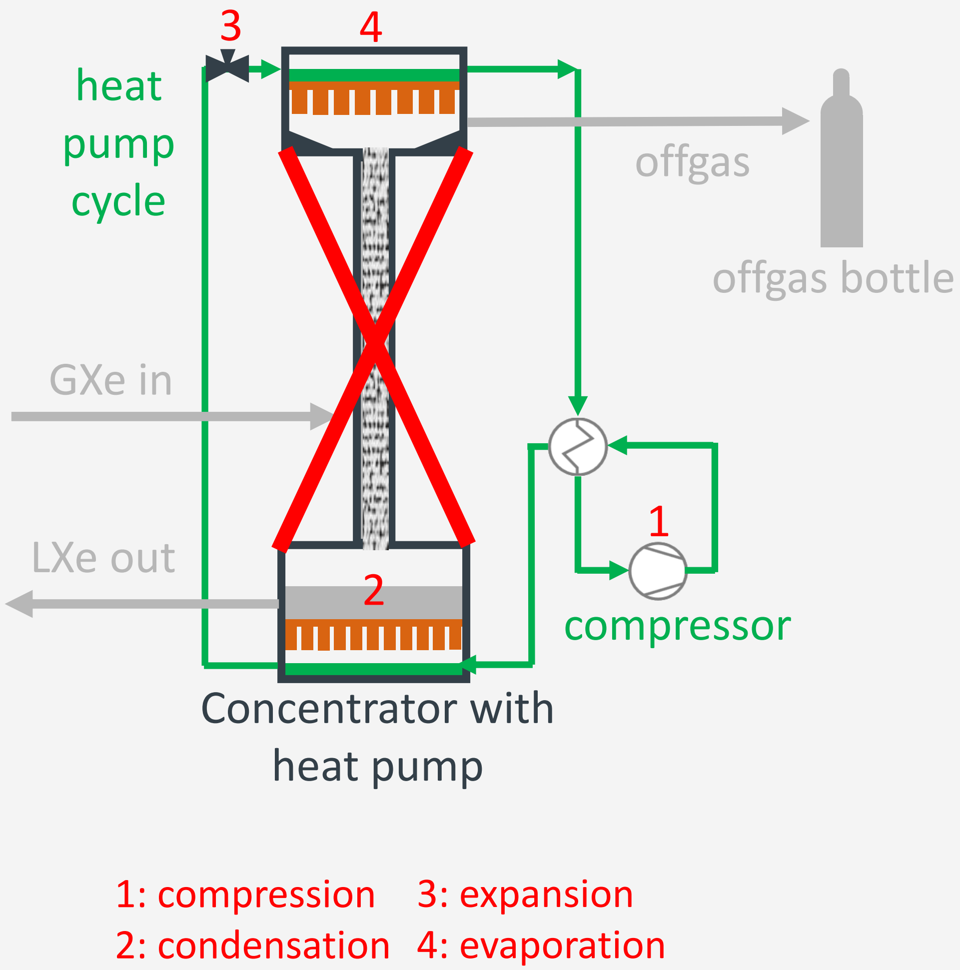

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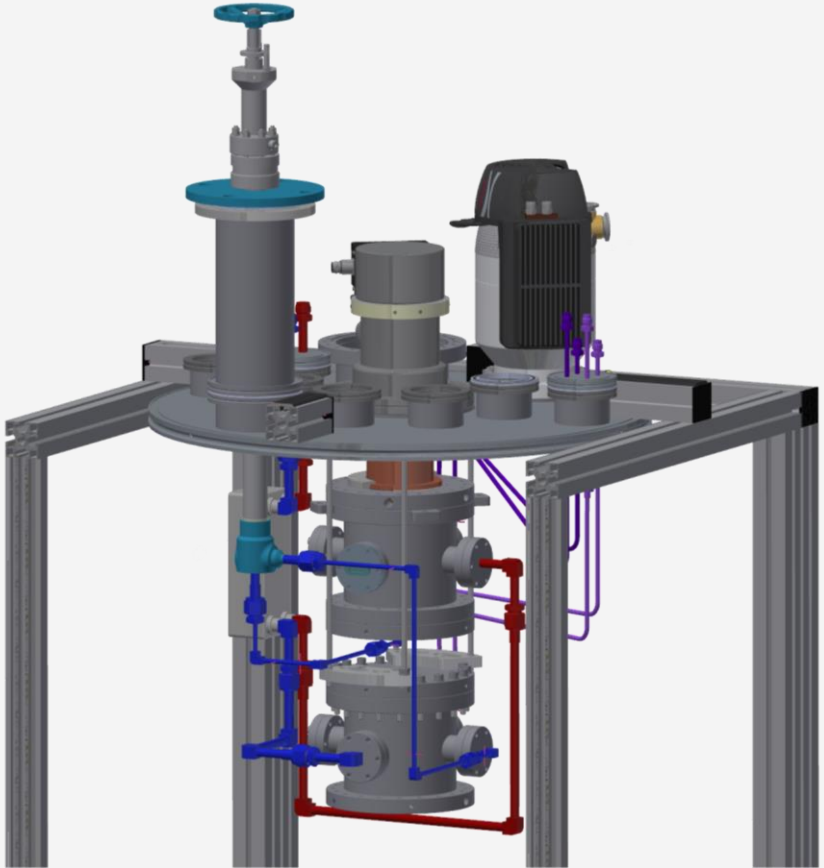
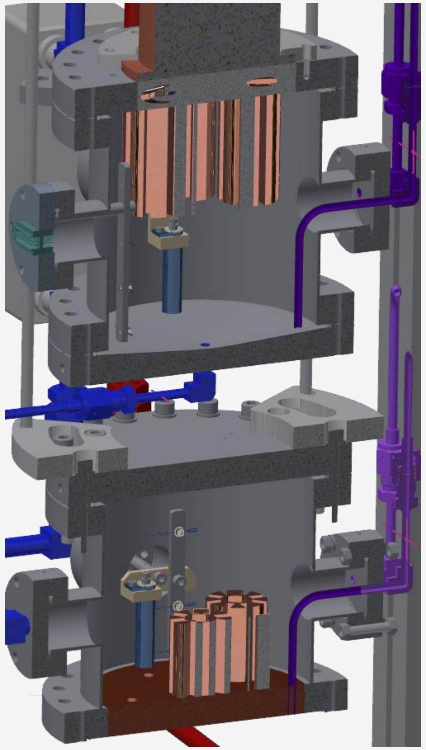


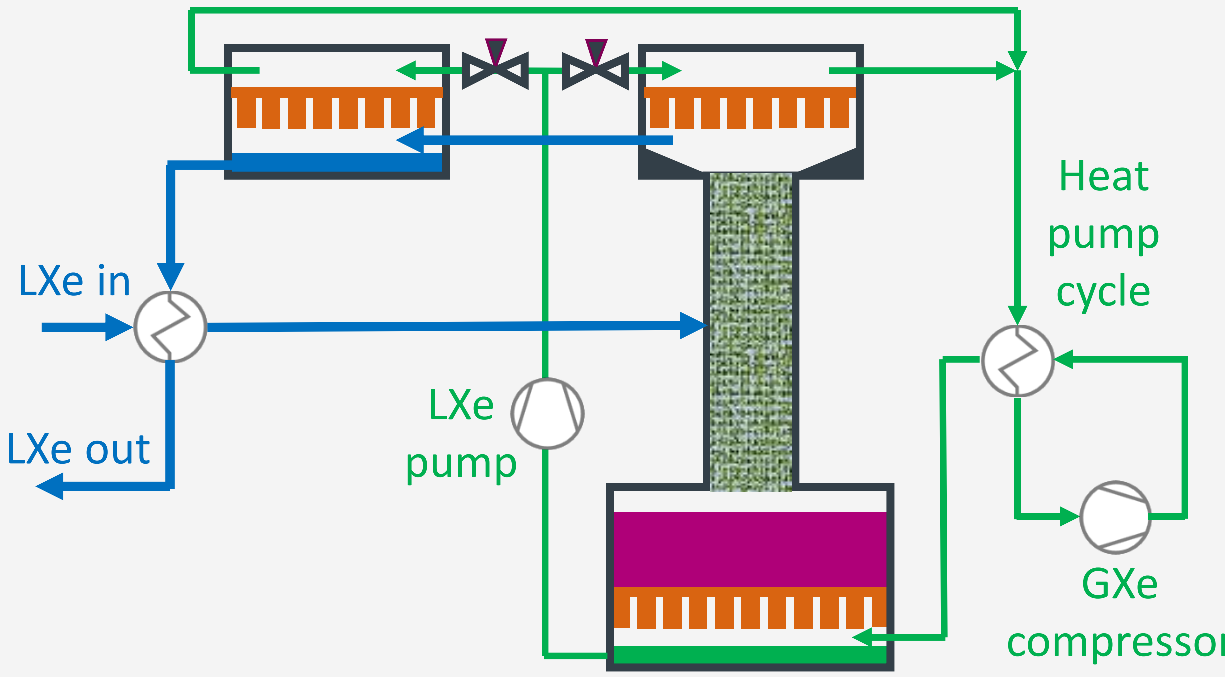


Conclusion

Classic (offline) and online **Kr removal essential** for future experiments

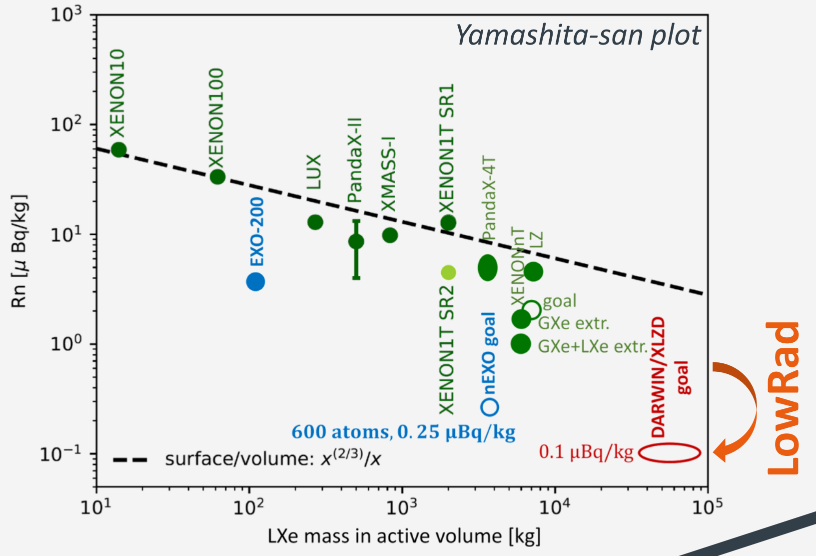
Radon mitigation needs to start with **material screening** & **selection** as well as **smart detector design**, all Xe-systems including purification system must be “**Rn emanation free**”

Online Rn removal (GXe+LXe) is the last step!



ERC AdG LowRad:

- **Develop technologies** for:
  - Continuous/**online ^{85}Kr removal** (30 ppq ^{nat}Kr)
  - Another **factor 10** in **^{222}Rn reduction** (0.1 µBq/kg)
- Complete **purification & distillation demonstrator (75 kg/h)**



This research at University of Münster is funded by

Bundesministerium für Bildung und Forschung
GRK 2149
DFG
erc

Cryogenic distillation (in the Münster context)

Cryogenic 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:

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

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

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

D. Schulte, M. Murra, P. Schulte, C. Huhmann, C. Weinheimer, **Ultra-clean radon-free four cylinder magnetically-coupled piston pump**, JINST 16 (2021) P09011

E. Aprile et al. [XENON Collaboration], **222Rn emanation measurements for the XENON1T experiment**, EPJ C 81 (2021) 337

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

E. Aprile et al. [XENON Collaboration], **Online 222Rn removal by cryogenic distillation in the XENON100 experiment**, Eur. Phys. J. C 77 (2017) 358

E. Aprile et al. [XENON Collaboration], **Removing krypton from xenon by cryogenic distillation to the ppq level**, Eur. Phys. J. C 77 (2017) 275

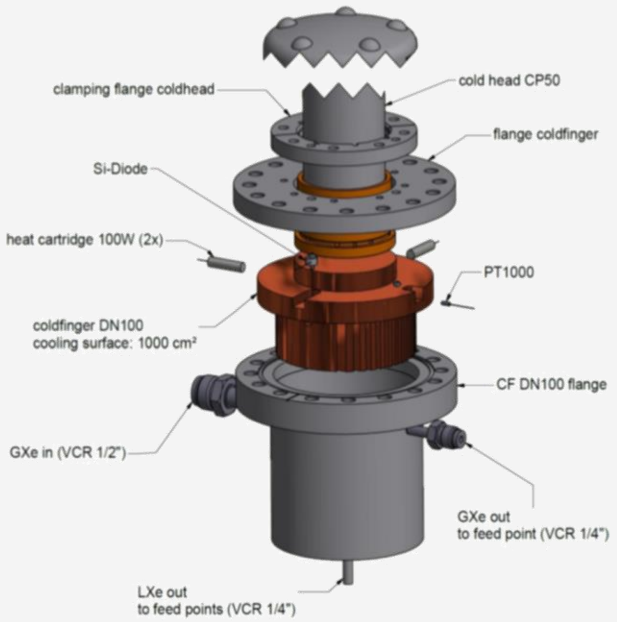
R. Rosendahl et al., **Determination of the separation efficiencies of a single-stage cryogenic distillation setup to remove krypton out of xenon by using a 83mKr tracer method**, Rev. Scient. Instr. 86 (2015) 11, 115104

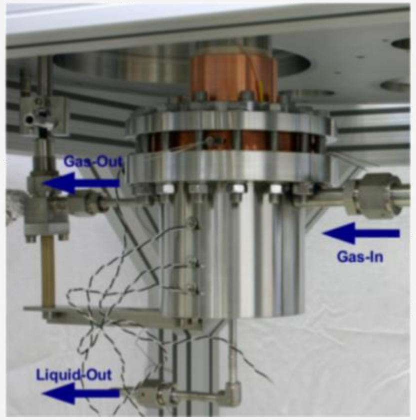
S. Rosendahl, K. Bokeloh, E. Brown, I. Cristescu, A. Fieguth, C. Huhmann, O. Lebeda, C. Levy, M. Murra, S. Schneider, D. Venos and C. Weinheimer, **A novel 83mKr tracer method for characterizing xenon gas and cryogenic distillation systems**, JINST 9 (2014) 10, P10010

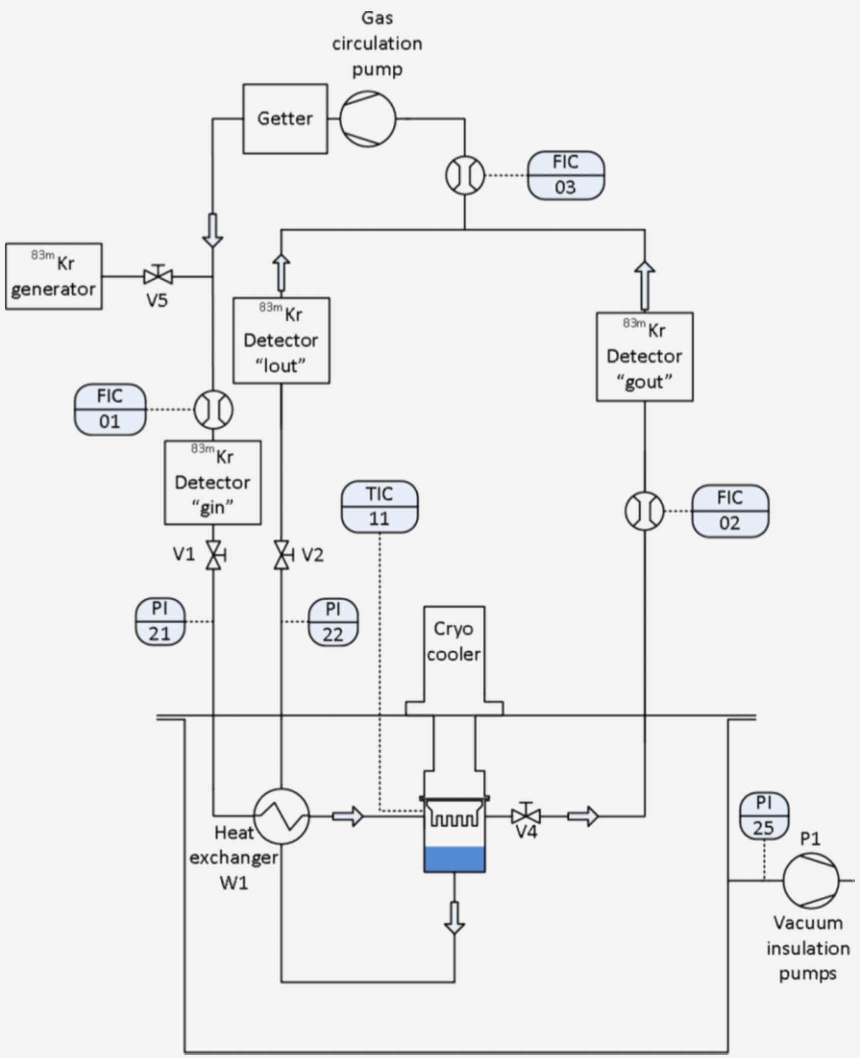
E. Brown, S. Rosendahl, C. Huhmann, C. Weinheimer and H. Kettling, **In situ measurements of Krypton in Xenon gas with a quadrupole mass spectrometer following a cold-trap at a temporarily reduced pumping speed**, JINST 8 (2013) P02011

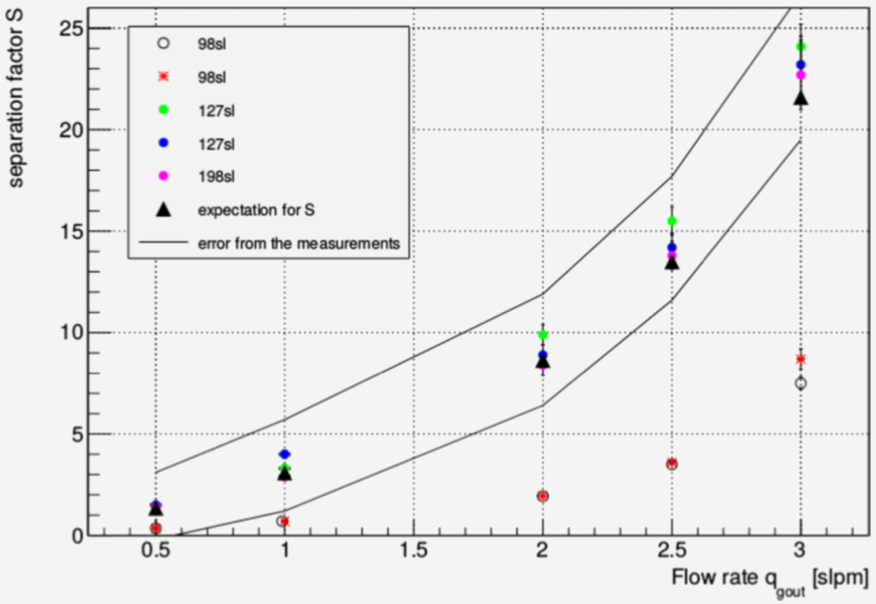
Rev Sci Instrum 86, 115104 (2015)

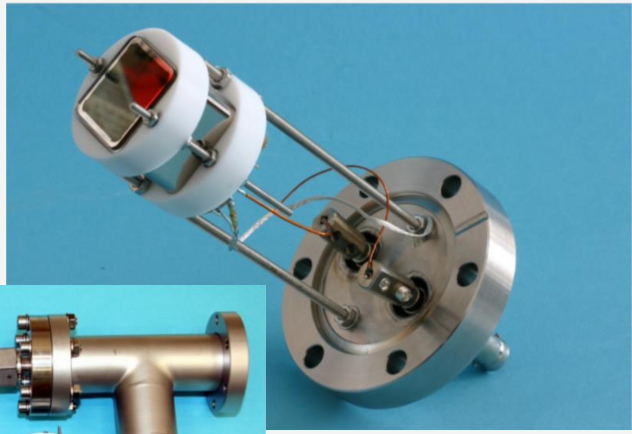
^{83m}Kr tracer method for single stage DST

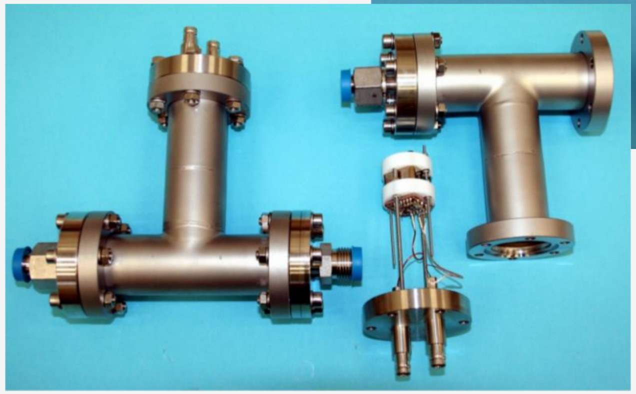












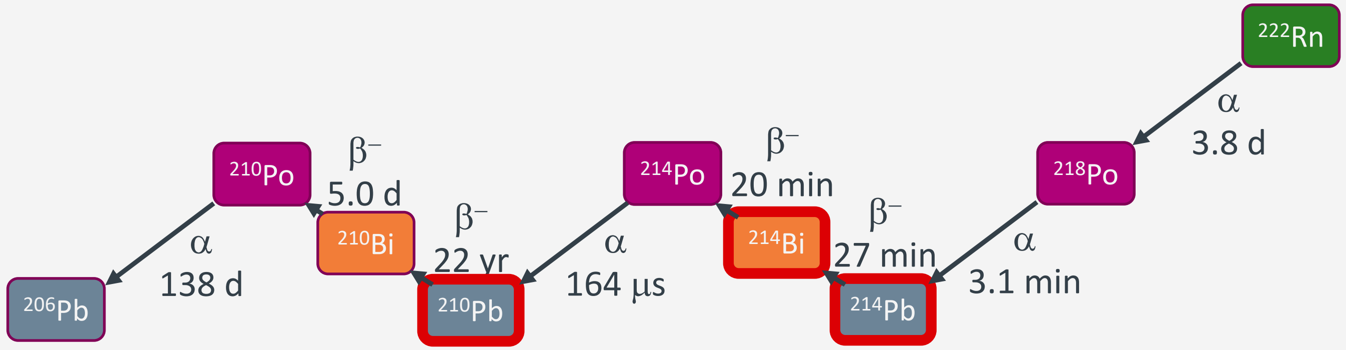
Radon mitigation strategies

I. **Screen materials** for gamma emission/Rn emanation and **selection**

II. **Avoiding Rn migration** into LXe by coating, hermetically-sealed TPCs, xenon ice, …

III. Use **larger volume-to-surface ratio** to reduce impact of Rn emanation from walls

IV. **Identify radon progeny decays** by observing co-localized progenitor or other progeny decays



^{214}Bi-^{214}Po tagging

V. **Extract** GXe from regions of **large Rn emanation**/injection (e.g. cables entering the TPC), into a **Radon Removal System** (e.g. by chromatography or distillation)

VI. **High flow LXe extraction** from the detector into a high throughput Radon Removal System

} **this talk**

EPJC 82, 599 (2022)

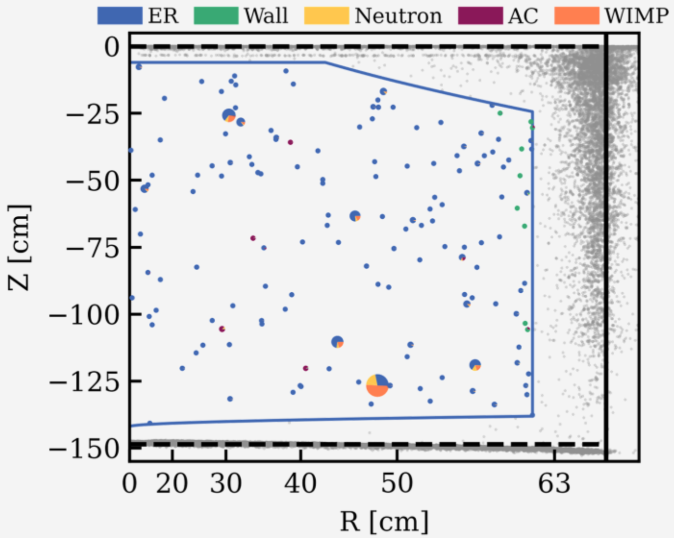
Material screening and fiducialization

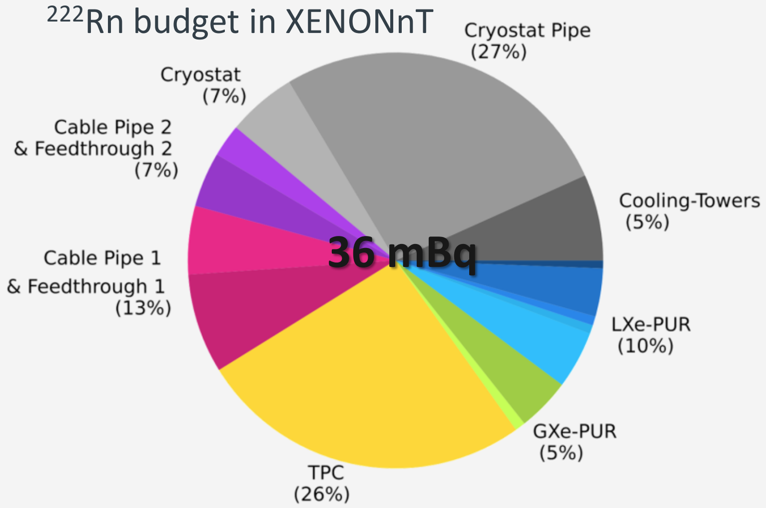
Radon prevention and radiopurity

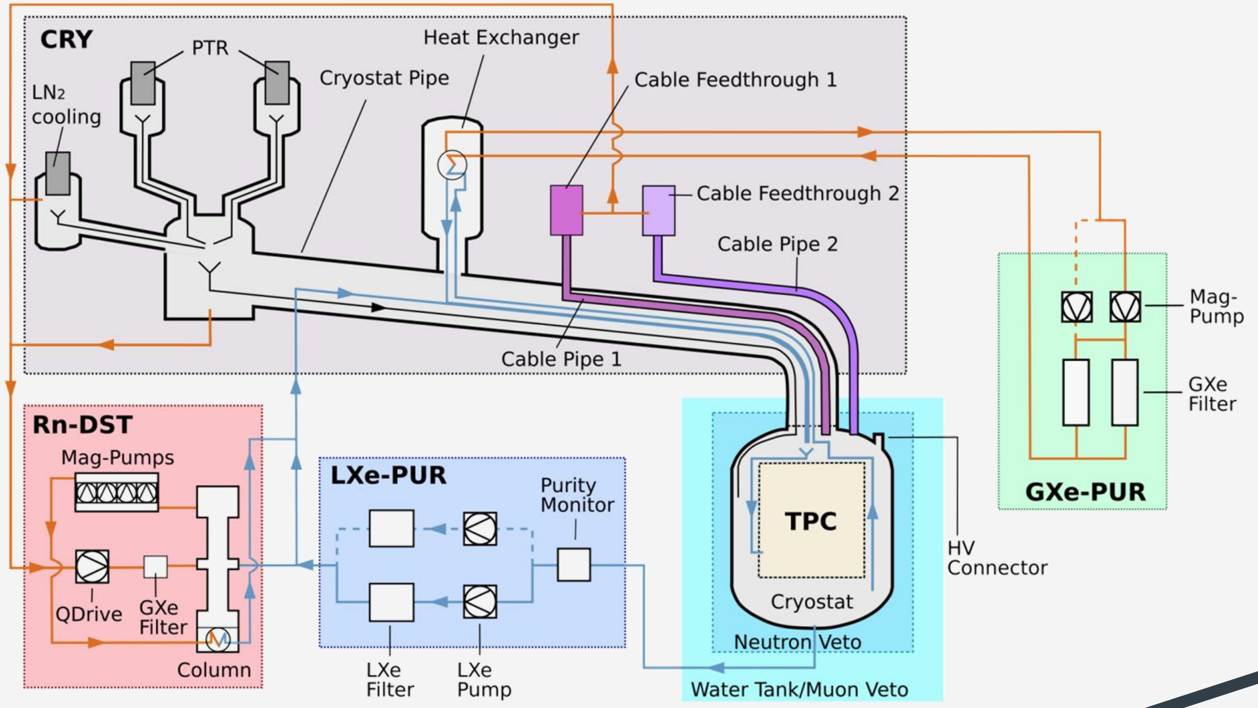
- Careful selection of materials & **screening** of all detector parts



- Total radon emanation rate: **36 mBq**
- Further reduction of material backgrounds by **fiducialization**



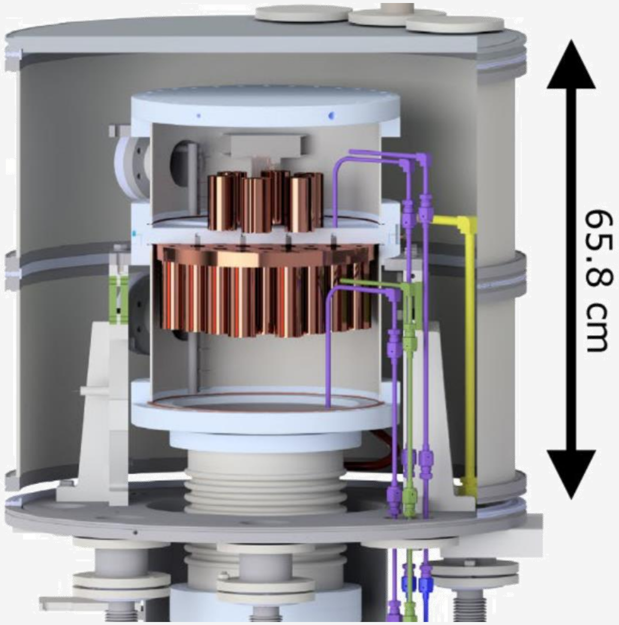


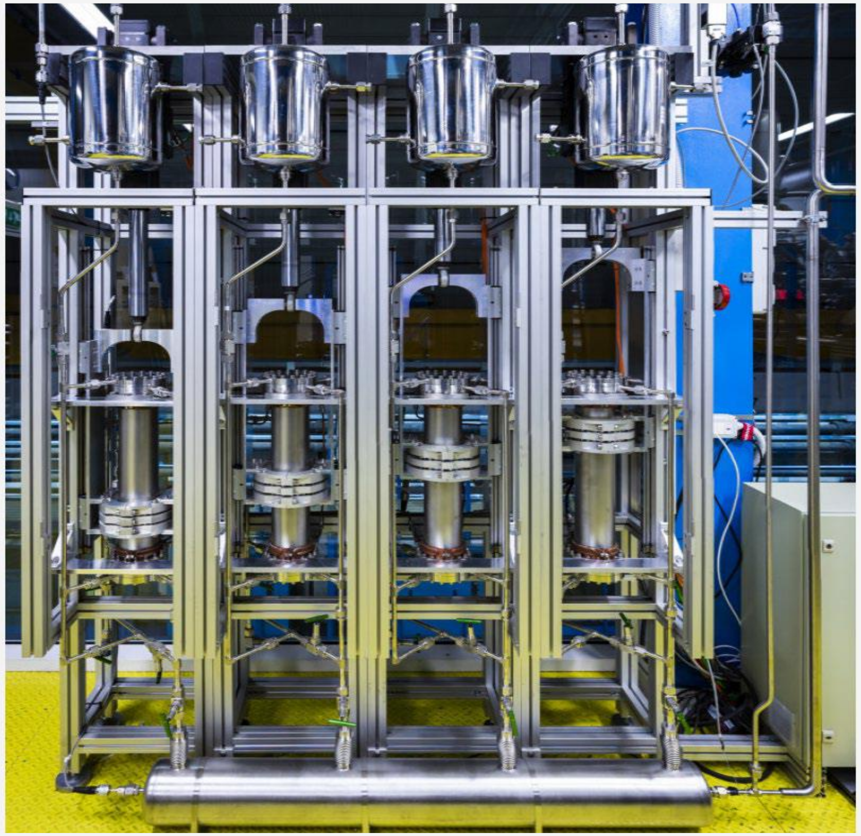


Radon removal system for XENONnT

Top condenser JINST 17 P05037 (2022)

- Custom bath-type LN_2/GXe heat exchanger
- Large surface OFHC copper fins
- **Cooling power:** > 3 kW (required 1 kW)
- **Xe liquefaction rate:** > 113 kg/h (300 slpm) (required 36 kg/h)
- **Cooling efficiency:** 0.98
- **LN_2 consumption:** 660 kg/d @ 1 kW cooling power

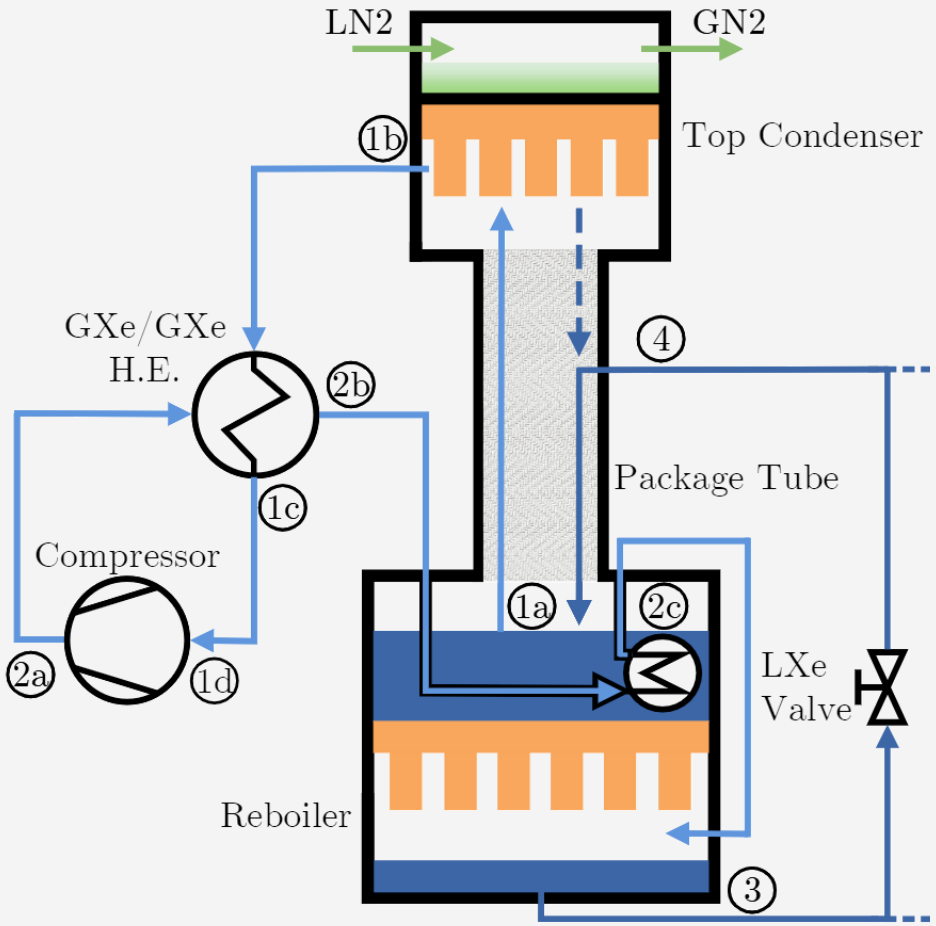




Compressor JINST 16 P09011 (2022)

- **Magnetically-coupled piston pump** based on XENON1T prototype: EPJ C78 604 (2018)
- Four piston pumps in **parallel operated phase-shifted**
- **Flow:** 170 kg/h (474 slpm) (required 72 kg/h or 200 slpm)
- **Compression:** 1.8 bar (required 1.5 bar)

Heat cycle of the XENONnT RRS



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