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Astroparticle School  
Obertrubach  
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# A cryogenic distillation column for the XENON1T experiment

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for the XENON collaboration

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living.knowledge  
WWU Münster

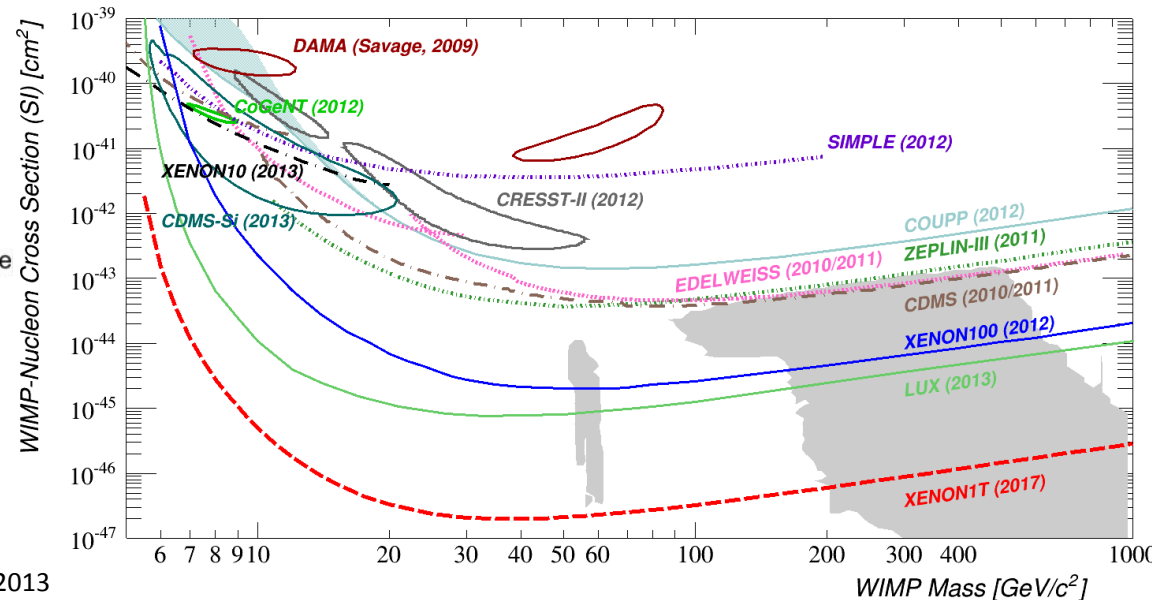
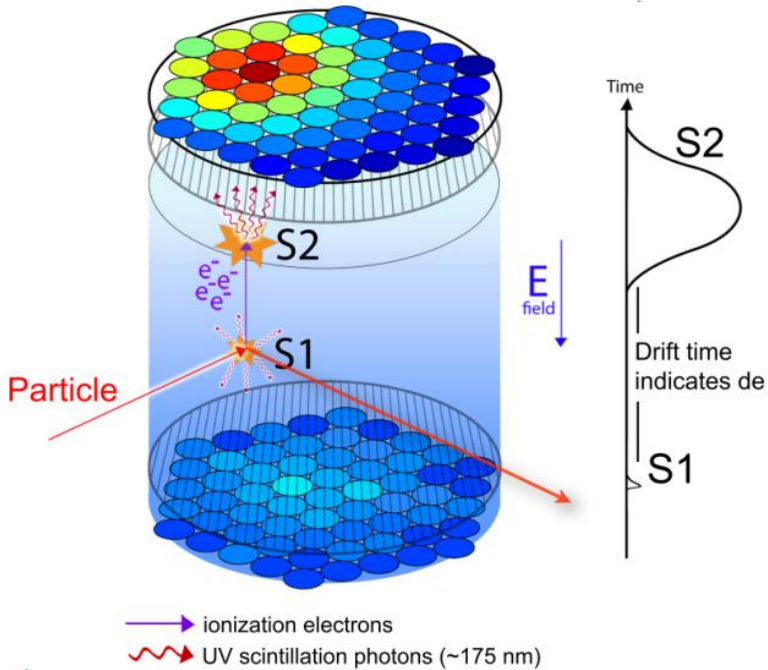
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12. October 2015

**Direct Dark Matter detection searching for WIMPs**  
(Weakly Interacting Massive Particles)

located at LNGS in Gran Sasso, Italy, at 3600 m water equivalent depth

Using dual phase xenon TPC



XENON10



2005

XENON100



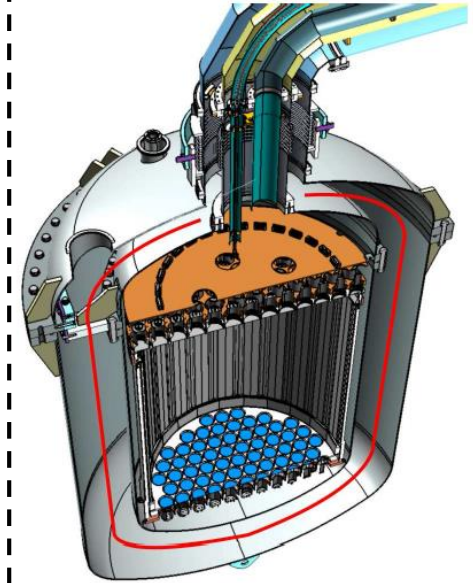
2009

XENON1T

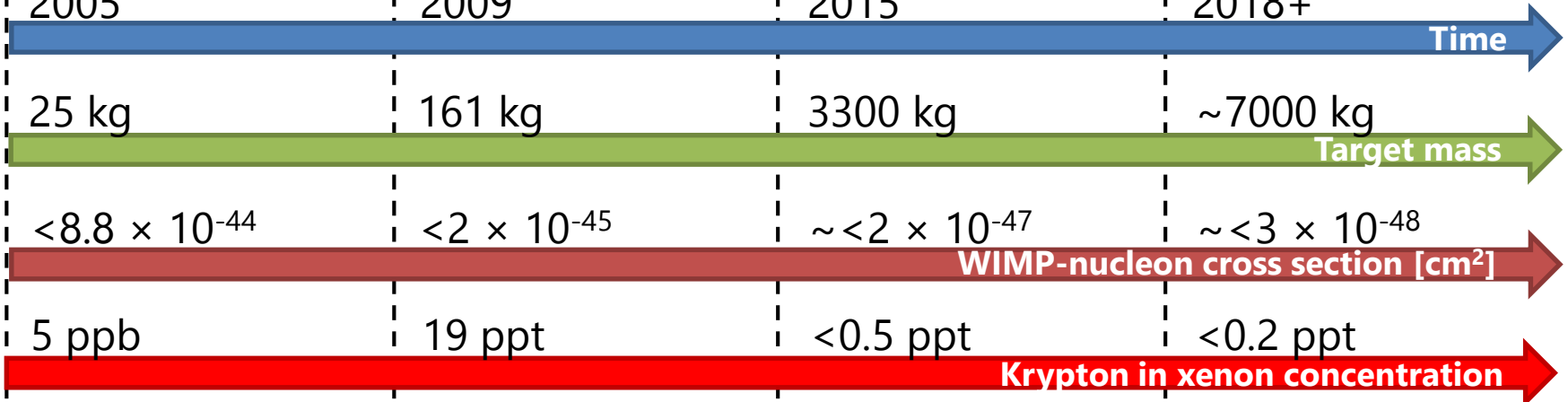


2015

XENONnT



2018+



3



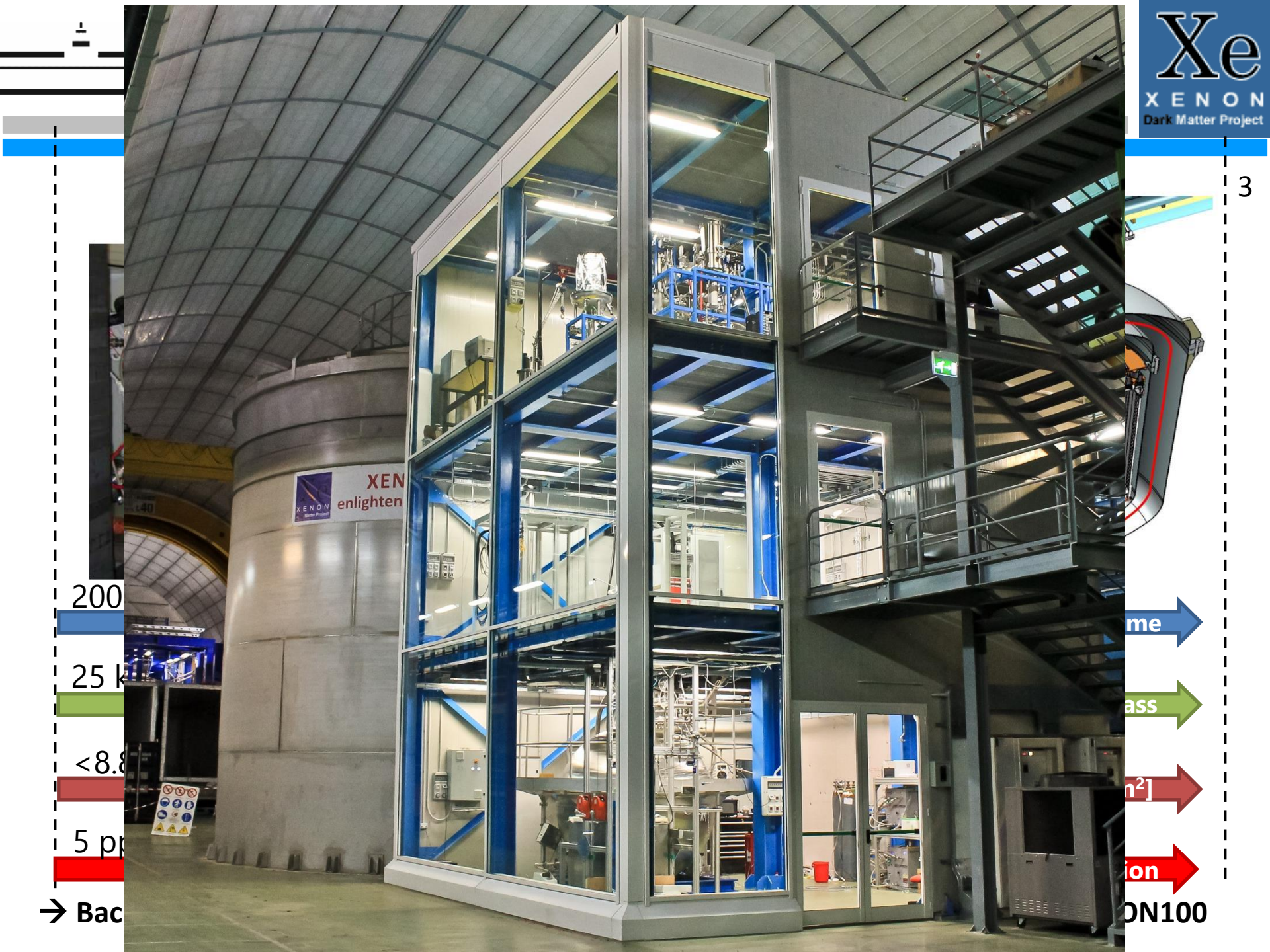
Time →

Mass →

[m<sup>2</sup>] →

Ion →

ION100



XENON enlighten

200

25 k

<8.8

5 p

→ Back

**Background for XENON1T has to be reduced by 2 orders of magnitude w.r.t. XENON100**

XENON  
enlighten

200

25 k

<8.8

5 p

→ Bac

me

ass

n<sup>2</sup>

ion

ON100



$^{85}\text{Kr}$  :  $\beta$ -emitter with endpoint energy of  $E=687.1$  keV  
and a half-life of  $t_{1/2}=10.76$  y

### intrinsic contamination of the xenon itself

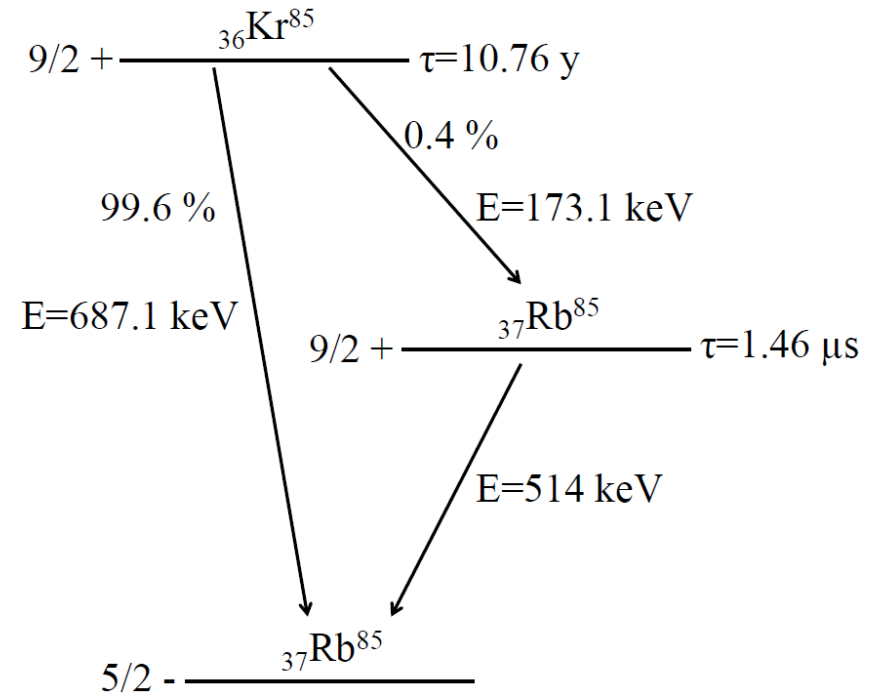
- homogeneously distributed inside the detector
- leakage events from the low energy  $\beta$ -spectrum contaminate region of interest for dark matter search

### created in nuclear fission

- homogeneously distributed in the air

commercial xenon:  $\text{natKr}/\text{Xe} \sim 10^{-9} - 10^{-6}$   
(ppb – ppm)

$^{85}\text{Kr}/\text{natKr} \sim 10^{-11}$



**For XENON1T:**

$$\text{natKr}/\text{Xe} < 5 \cdot 10^{-13} \text{ (0.5 ppt)} \quad \leftrightarrow \quad 0.5 \frac{\text{evt}}{\text{y} \cdot \text{ton}}$$

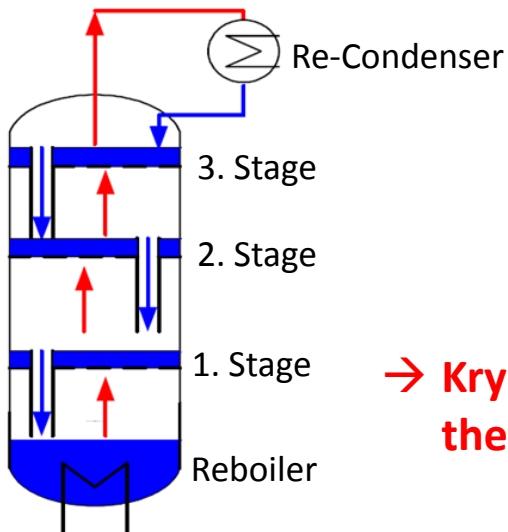
## „Multi-stage“ distillation with partial reflux:

Separation is based on the difference in vapor pressure of the two components:

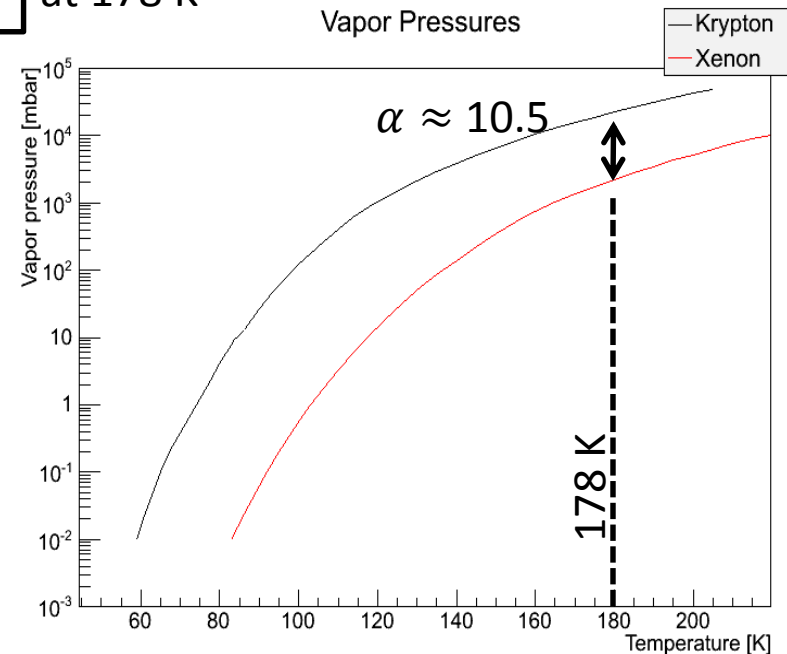
relative volatility:  $\alpha = \frac{P_{Kr}}{P_{Xe}} \approx 10.5$  at 178 K

series of distillation stages for desired purity

- Common approach is the **McCabe-Thiele** method
- Calculation of the number of theoretical stages for a idealised mixture of two gases: 9-10



→ **Krypton as more volatile gas is collected at the top, while the purified liquid xenon is enriched in the bottom**



heat- and mass transfer on huge surface: package material

**HETP:**  
Height equivalent of one theoretical plate



**Processing:**

incoming xenon is cooled, liquefied and injected to package tube

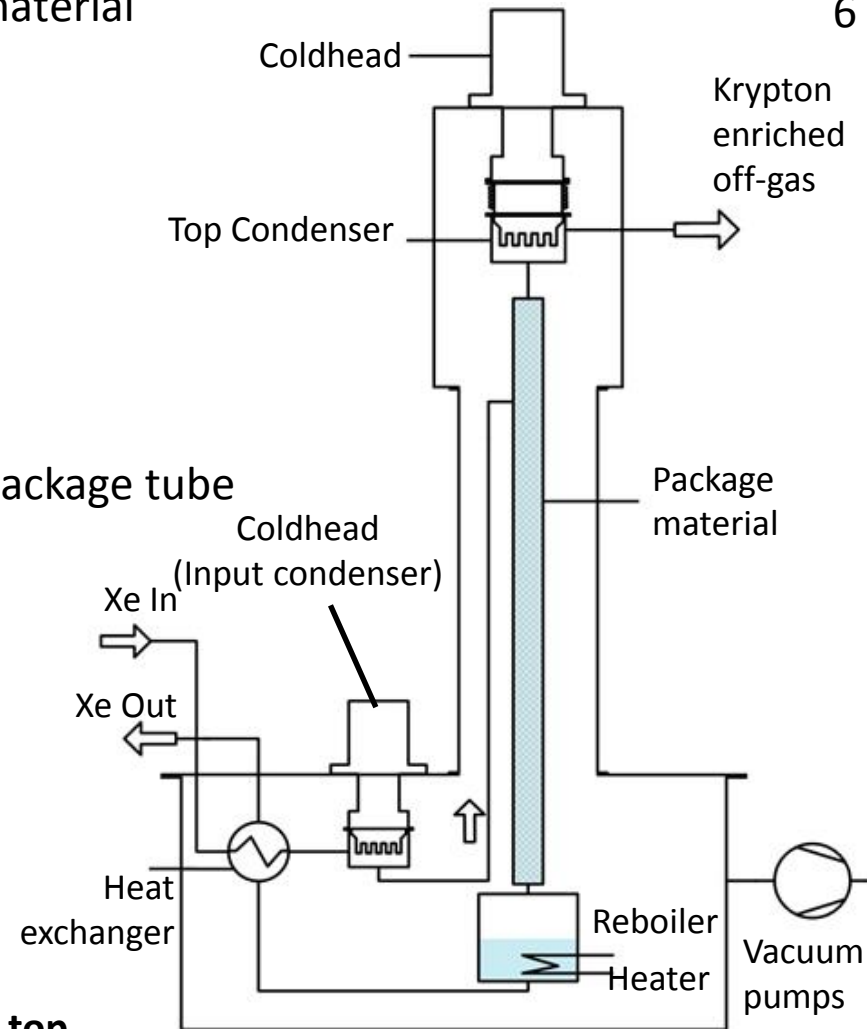
**Heater in the bottom (reboiler):**

- evaporate the mixture
- mass transfer along the package tube.
- **purified xenon extracted from the reboiler**

**Coldhead at the top (Condenser)**

- re-condense xenon and feed back to the package
- **krypton-enriched off-gas extracted from the top**

**Strategy:** Divide the project into different phases



Scheme of the package column for XENON1T

Design in collaboration with  
Dr. Ion Cristescu, KIT/ITER



## Design Parameter for XENON1T

**feeding flow rate:**

3kg/h = 8.3 SLPM

**separation factor:**  $10^4 - 10^5$

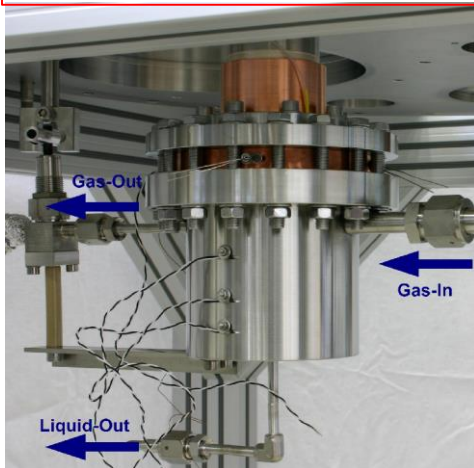
**Kr removal:**

${}^{\text{nat}}\text{Kr}/\text{Xe} < 5 \cdot 10^{-13} = 0.5 \text{ ppt}$

**Xe recovery:** 99%

**T = 178 K and p = 2 bar**

## Single distillation stage



## Design Parameter for XENON1T

feed  
3kg/  
sepa  
Kr re  
natK  
Xe re  
T = 1

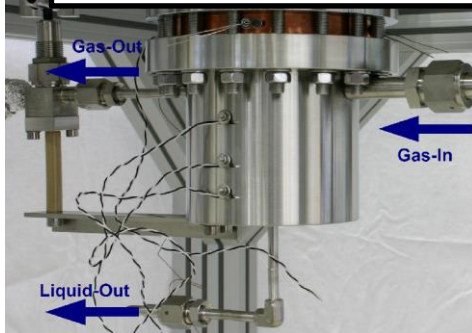
### Single distillation stage main results:

Separation factor in the order of 10 as expected ( $\alpha \approx 10$ )

**Cryogenic distillation is working in the sub-ppt concentrations  
shown with Kr-83m tracer method\***

\*See talk by Christian Wittweg

Tracer method concept: S. Rosendahl et al, JINST 9 P10010 (2014)  
Single stage measurements submitted to Review of Scientific Instruments



## Design Parameter for XENON1T

**feeding flow rate:**

3kg/h = 8.3 SLPM

**separation factor:**  $10^4 - 10^5$

**Kr removal:**

${}^{\text{nat}}\text{Kr}/\text{Xe} < 5 \cdot 10^{-13} = 0.5 \text{ ppt}$

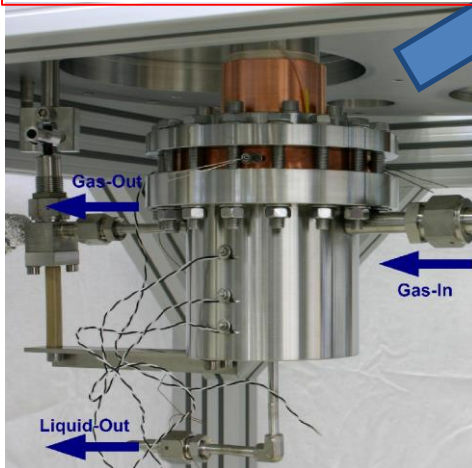
**Xe recovery:** 99%

**T = 178 K and p = 2 bar**

Phase 1 distillation column



Single distillation stage



## Design Parameter for XENON1T

feed  
3kg/  
sepa  
Kr re  
natKr  
Xe re  
T = 1

### Phase-1 main results:

(S. Rosendahl, Dissertation, 2015)

Thermodynamic stability under design parameters

Separation factor > 5.000

**Purified liquid out:  ${}^{\text{nat}}\text{Kr}/\text{Xe} < 26 \cdot 10^{-15} = 26 \text{ ppq}$  (90% c.l.)**

With GC-RGMS\* system in Heidelberg only a limit could be set!

→ **Lowest concentration of  ${}^{\text{nat}}\text{Kr}/\text{Xe}$  measured so far (to our knowledge)**

Factor  $\approx 20$  better than required for XENON1T!!

\*(GC-RGMS paper from MPIK claiming sup-ppt sensitivity: S. Lindemann and H. Simgen Eur.Phys.J. C74 (2014) 2746)

# Different phases of distillation project

## Design Parameter for XENON1T

feeding flow rate:

3kg/h = 8.3 SLPM

separation factor:  $10^4 - 10^5$

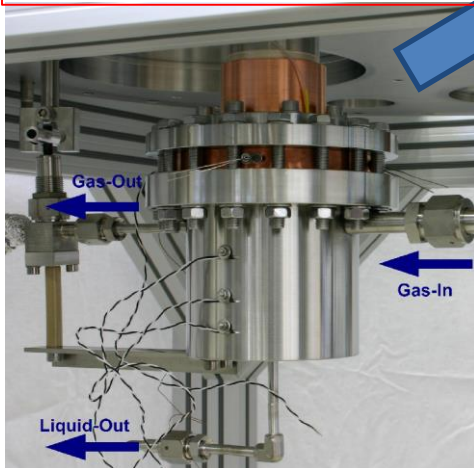
Kr removal:

${}^{\text{nat}}\text{Kr}/\text{Xe} < 5 \cdot 10^{-13} = 0.5 \text{ ppt}$

Xe recovery: 99%

T = 178 K and p = 2 bar

Single distillation stage



Phase 2 distillation column

Phase 1 distillation column



# Different phases of distillation project



## Design Parameter for XENON1T

feed  
3kg/  
sepa  
Kr re  
natKr  
Xe re  
T = 1

### Phase-2 main results:

Thermodynamic stability for flows up to 18 slpm

Measurements of the absolute concentration in the gas-in line and the purified liquid-out line:

RGA with cold-trap enhanced sensitivity\* (→ See talk by Alex Fieguth)

gas-in concentration: 25 ppm  
liquid-out concentration: < 200 ppt

→ Sensitivity limit of RGA method 200 ppt

**Separation factor  $^{\text{nat}}\text{Kr}/\text{Xe} > 125.000$**

\*E. Brown et al, 2013\_JINST\_8\_P02011 (2013)



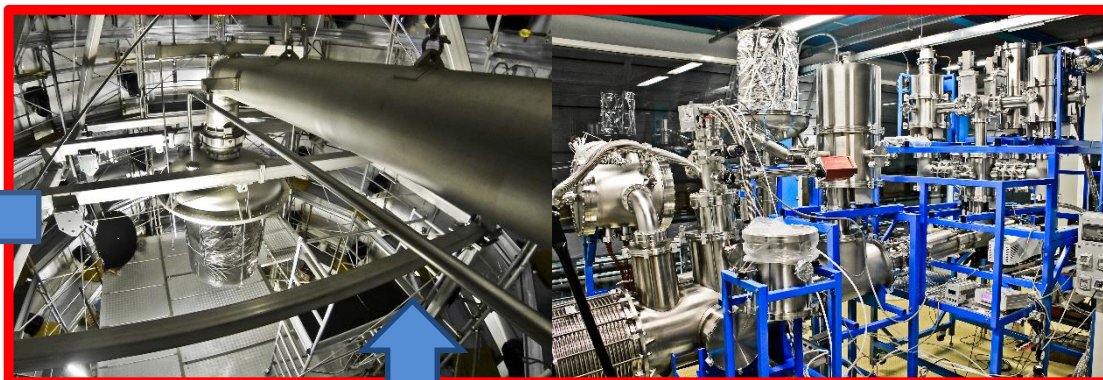
# Commissioning at XENON1T

## RESTOX:

Storage vessel for up to 7 tons of xenon



## CRYOSTAT + CRYOGENICS: Housing of TPC + Liquefaction of xenon



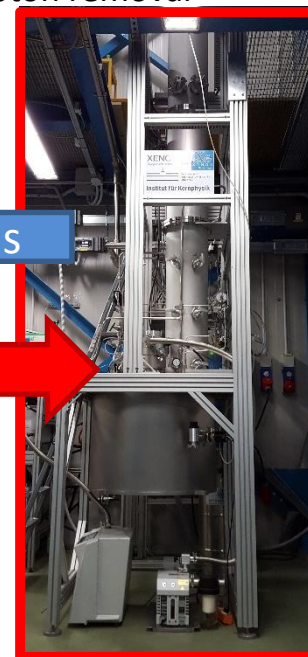
10

store

distribute

## DISTILLATION COLUMN:

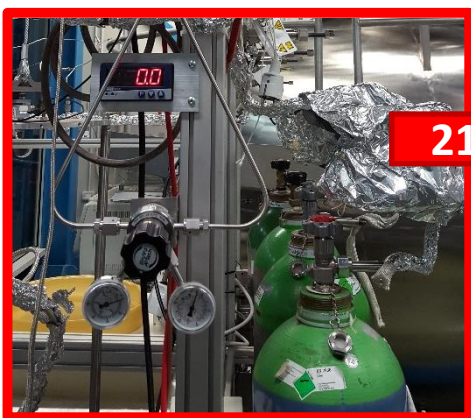
Krypton removal



purified gas

## BOTTLE RACK:

Fill xenon from bottles  
+ analyse impurities



210 kg xenon

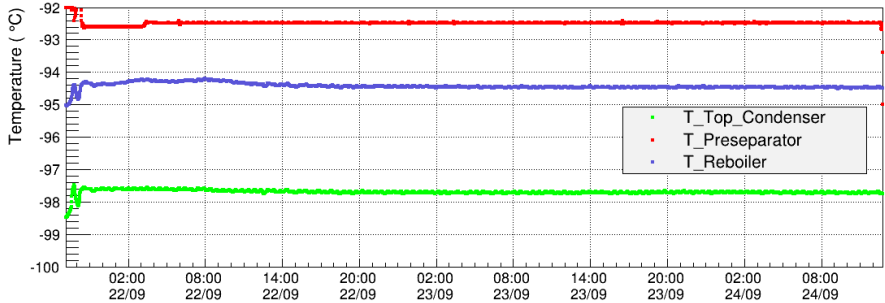
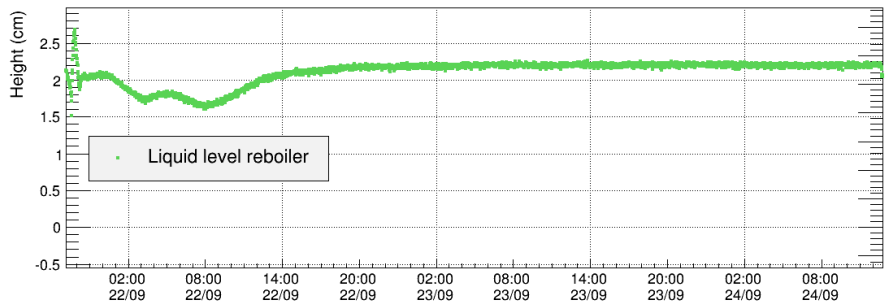
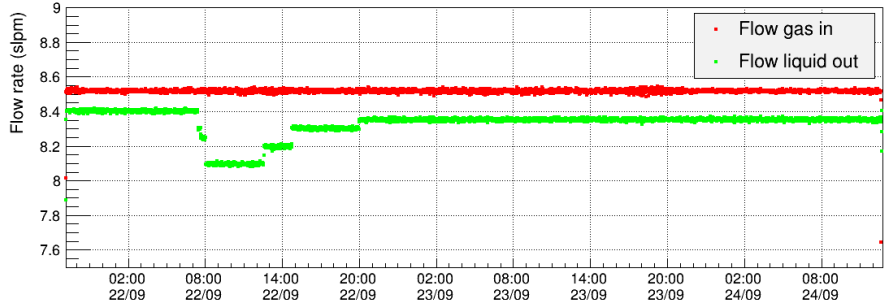
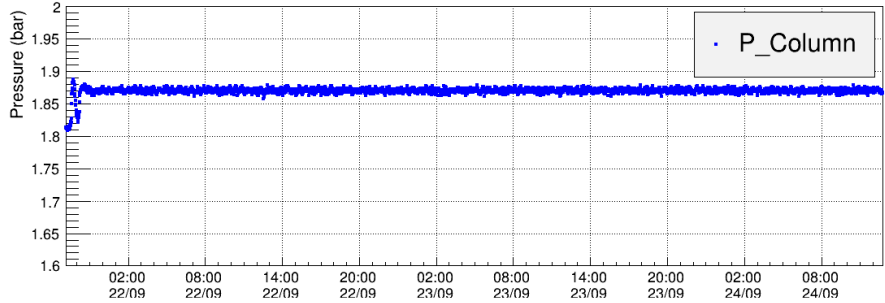
raw gas



## PURIFICATION SYSTEM:

Gas distribution + hot getter purification

# g at XENON1T



• **CRYOGENICS:** Housing of TPC + Liquefaction of xenon



**Processing 210 kg of commercial xenon**

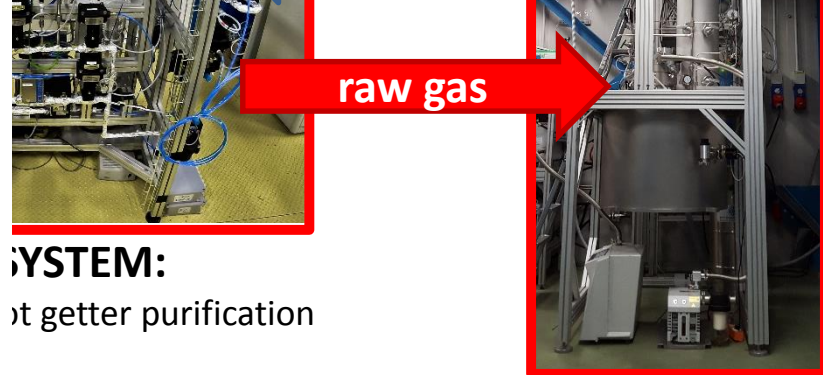
**Flow rate of 8.5 slpm (3.1 kg/h)**

**~ 3 days of continuous distillation**

**Gas samples for the characterisation of the column performance have been taken for RGMS measurements**

10

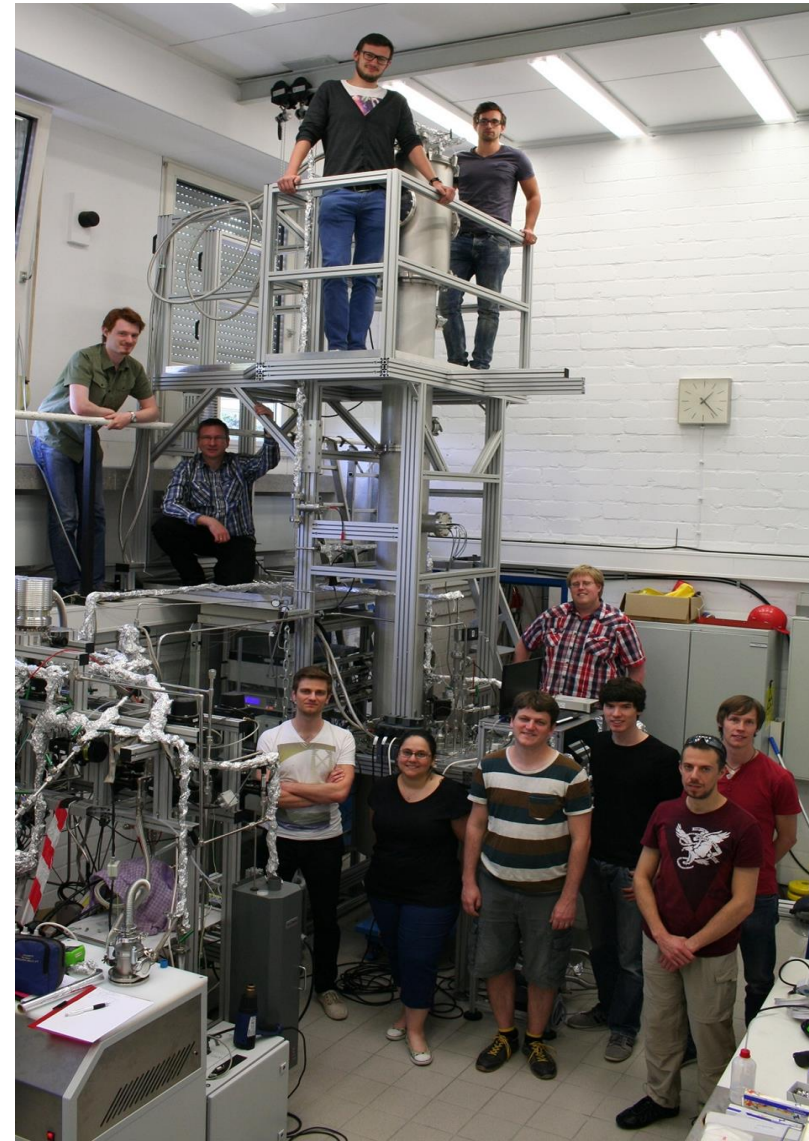
JMN:



**SYSTEM:**  
not getter purification



- XENON1T requests a high throughput and ultra-clean new distillation column
- Package column based on McCabe-Thiele method
- Thermodynamic stability at designed flow rate of 8.3 slpm (3kg/h) for different operation modes demonstrated
- **RGMS system at MPIK shows <26 ppq (0.026 ppt) with 90% confidence level**
- **Separation factor > 125.000**  
→ **XENON1T requirements more than fulfilled**
- Installation and commissioning of Phase-2 at XENON1T experiment at LNGS has been done in September 2015
- **Ready for purification of 3.3 tons (~7 weeks of distillation) of xenon**



# Thank you for your attention!

This project is supported by DFG Großgeräte (INST 211/528-1 FUGG, funded together with the state NRW and University of Münster) and by BMBF (05A11 PM1). Some aspects of the R&D for this project are funded by DFG (WE 1843/7-1) and by Helmholtz Alliance of Astroparticle Physics HAP.

Deutsche  
Forschungsgemeinschaft  
**DFG**

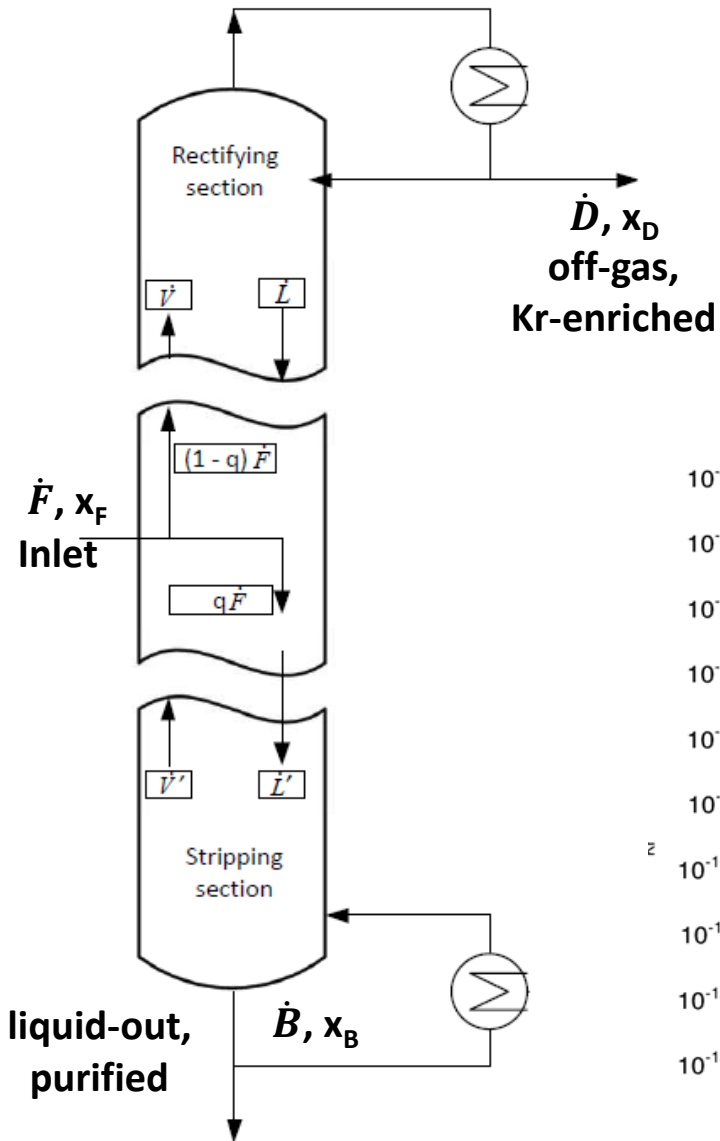


HELMHOLTZ  
ASSOCIATION  
Alliance for Astroparticle Physics



Bundesministerium  
für Bildung  
und Forschung

# McCabe-Thiele calculation for the XENON1T column



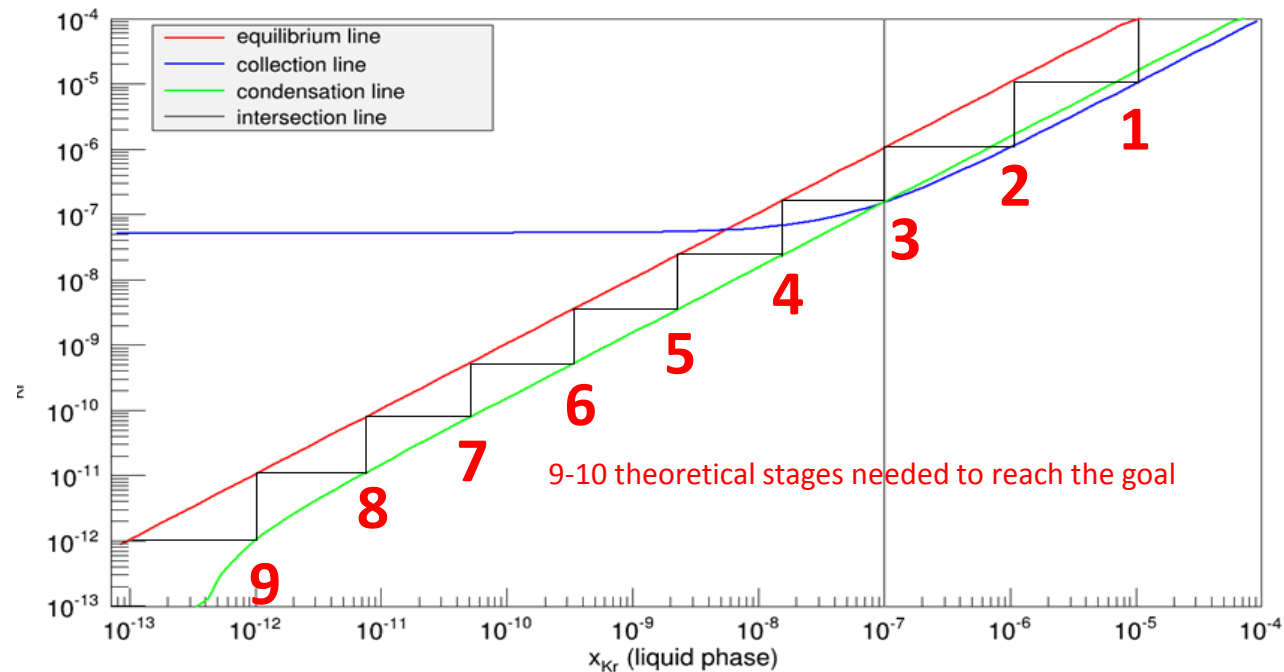
Krypton particle mass balance: Input = Output 8

„Flow · Concentration“

$$\dot{F} \cdot x_F = \dot{D} \cdot x_D + \dot{B} \cdot x_B$$

→ Calculation for a series of distillation stages leads to McCabe –Thiele diagramm

McCabe-Thiele-diagram for liquid feed



## Distillation Column of XENON100:

7

- throughput: 0.6 kg/h

→  $^{\text{nat}}\text{Kr}/\text{Xe} = (19 \pm 4)$  ppt

E. Aprile et al, arXiv: 1207.5988v1

XMASS achieved  $^{\text{nat}}\text{Kr}/\text{Xe} = 3$  ppt

K. Abe et al, arXiv: 0809.4413v3

Panda X achieved  $^{\text{nat}}\text{Kr}/\text{Xe} = (21 \pm 3)$  ppt  
(with a 5m tall column)

Z. Wang et al, 2014 JINST 9 P11024

## Design Parameters for XENON1T:

- feeding flow rate: 3kg/h = 8.3 SLPM
- separation factor:  $10^4 - 10^5$
- Kr removal:  $^{\text{nat}}\text{Kr}/\text{Xe} < 0.5$  ppt
- Xe recovery: 99%
- $T = 178$  K and  $p = 2$  bar

LUX achieved  $^{\text{nat}}\text{Kr}/\text{Xe} = (4 \pm 1)$  ppt  
(with charcoal chromatography)

Lux Collaboration, arXiv:1403.1299v1

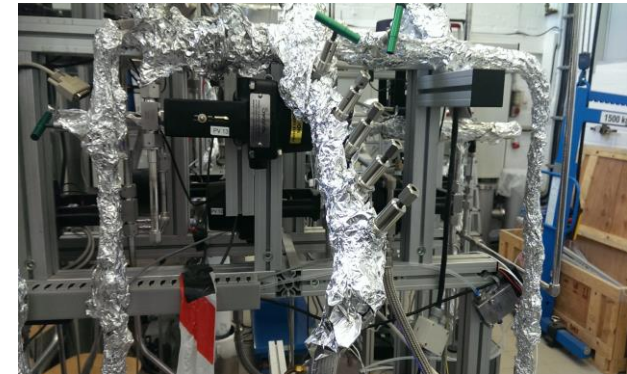
# Determination of column performance using RGMS at MPIK, Heidelberg

Measurements of krypton in xenon to the ppq level should be possible with RGMS

Mounting of ultra-clean pipettes from MPIK to the distillation column

Extensive pumping and baking procedure to avoid contaminations of the samples

Taking several samples from the distillation column, during distillation run at 8.5 slpm



**Main Result:**

**Purified liquid out:  ${}^{\text{nat}}\text{Kr}/\text{Xe} < 26 \cdot 10^{-15} = 26 \text{ ppq}$  (90% c.l.)**

With RGMS system in Heidelberg only a limit could be set!

→ **Lowest concentration of  ${}^{\text{nat}}\text{Kr}/\text{Xe}$  measured so far (to our knowledge)**

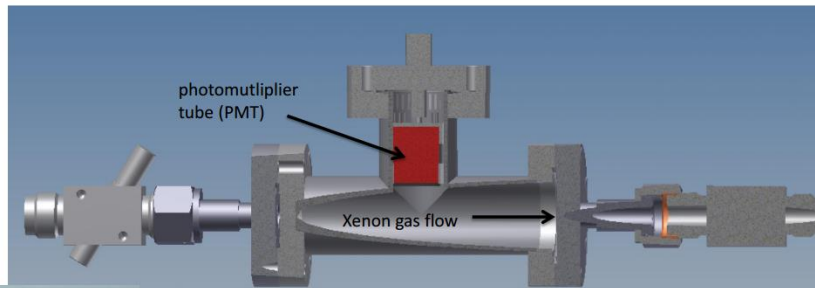
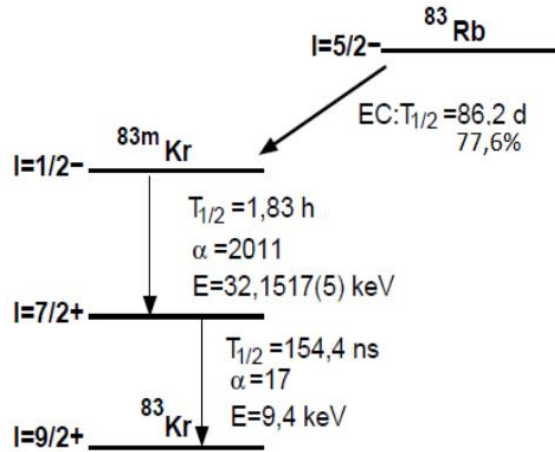
Factor  $\sim 20$  better than required for XENON1T!!



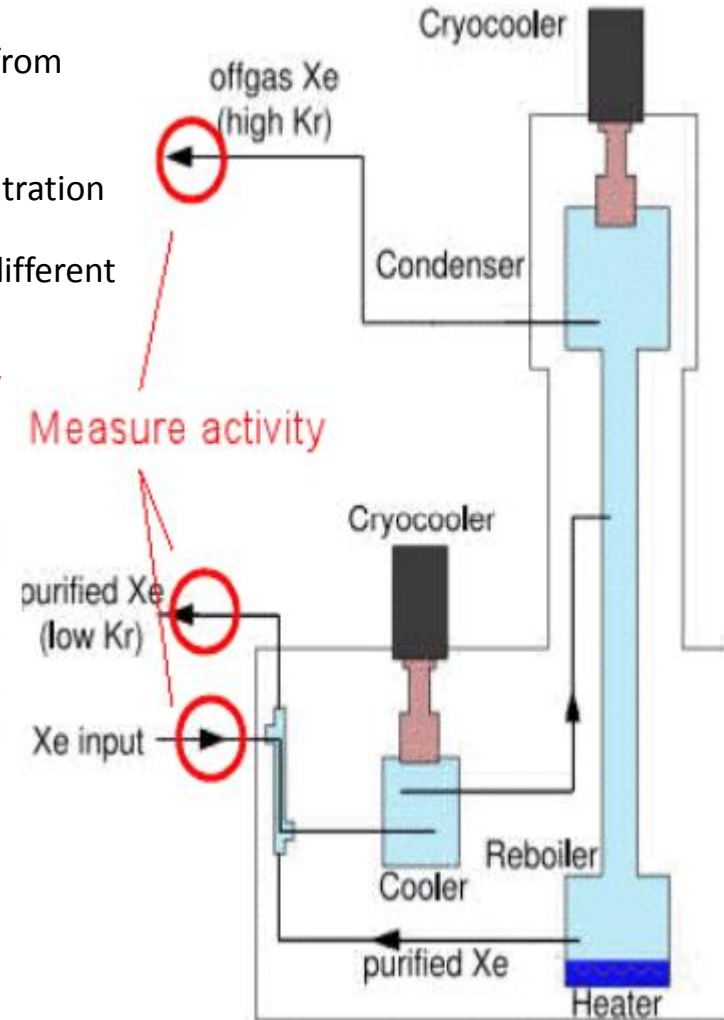
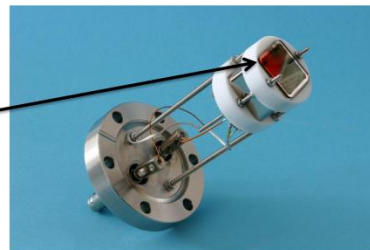
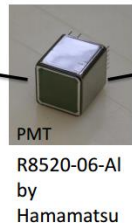
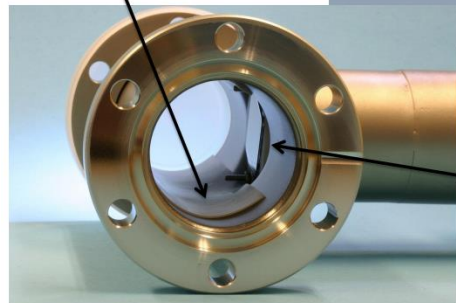
## $^{83m}\text{Kr}$ Monitors

- doping Xe gas with  $^{83m}\text{Kr}$  as a tracer, which has same chemical properties as  $^{85}\text{Kr}$
- measuring the Xe scintillation light from  $^{83m}\text{Kr}$  decays
- signal rate is proportional to concentration
- monitor relative concentrations of different points in the column

→ determine separation efficiency



PTFE VUV light reflecting foil



# Gas diagnostics using RGA with cold-trap enhanced sensitivity

## System is equipped with a RGA setup

- Custom made differential pumping sections and a liquid nitrogen coldtrap allows to freeze out the xenon while the krypton passes nearly unattached
- Custom made butterfly valve allows for reduced pumping speed which further increase the sensitivity
- Increased sensitivity down to  $\sim 40$ ppt possible

