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Review on high purity nitrogen technology and special techniques

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Outline

- High purity nitrogen
 - BOREXINO
 - nitrogen purity requirements
 - nitrogen purification
 - development of LAKN delivery chain
 - production of synthetic air
 - Purification of nitrogen for JUNO
 - Removal of ^{222}Rn
 - Removal of Ar and Kr
- ^{222}Rn -free clean rooms and ^{222}Rn monitoring
- ^{222}Rn diffusion and emanation studies
- Conclusions



BOREXINO radiopurity

In a nutshell: the radio-purest detector ever built

Isotope	Specification for LS	Phase I	Phase II
^{238}U	$\leq 10^{-16}$ g/g	$(1.7 \pm 0.1) \cdot 10^{-17}$ g/g	$< 0.9 \cdot 10^{-19}$ g/g
^{232}Th	$\leq 10^{-16}$ g/g	$(4.6 \pm 0.8) \cdot 10^{-18}$ g/g	$< 0.7 \cdot 10^{-18}$ g/g
$^{14}\text{C}/^{12}\text{C}$	$\leq 10^{-18}$	$(2.69 \pm 0.06) \cdot 10^{-18}$ g/g	unchanged
^{40}K	$\leq 10^{-18}$ g/g	$\leq 0.4 \cdot 10^{-18}$ g/g	unchanged
^{85}Kr	≤ 1 cpd/100 t	(30 ± 5) cpd/100 t	≤ 5 cpd/100 t
^{39}Ar	≤ 1 cpd/100 t	$\ll ^{85}\text{Kr}$	$\ll ^{85}\text{Kr}$
^{222}Rn	≤ 1 cpd/100 t	see ^{238}U	see ^{238}U
^{210}Po	not specified	$\sim (70)$ 1 dpd/100 t	unchanged
^{210}Bi	not specified	(20) 70 dpd/100 t	(11.5 ± 1.3) cpd/100 t

BX nitrogen

Requirements

Purification

LAKN development

Synthetic air

JUNO nitrogen

^{222}Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

Conclusions



Nitrogen gas in BOREXINO

Nitrogen of different grades was used in BOREXINO for:

- Cleaning/drying (flushing/purging vessels)
- Blanketing (CTF)
→ **RPN**
- Stripping (purification of water)
- Radon emanation measurements
- Production of synthetic air (inner vessel inflation)
→ **HPN**
- Scintillator purification: stripping of the LS was the last purification step in the detector filling process
→ **LAKN**

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Available nitrogen qualities

- Regular Purity Nitrogen (RPN)

Nitrogen evaporated from the technical quality of LN₂ (4.6, now 5.0)

Production rate up to 100 m³/h (STP)

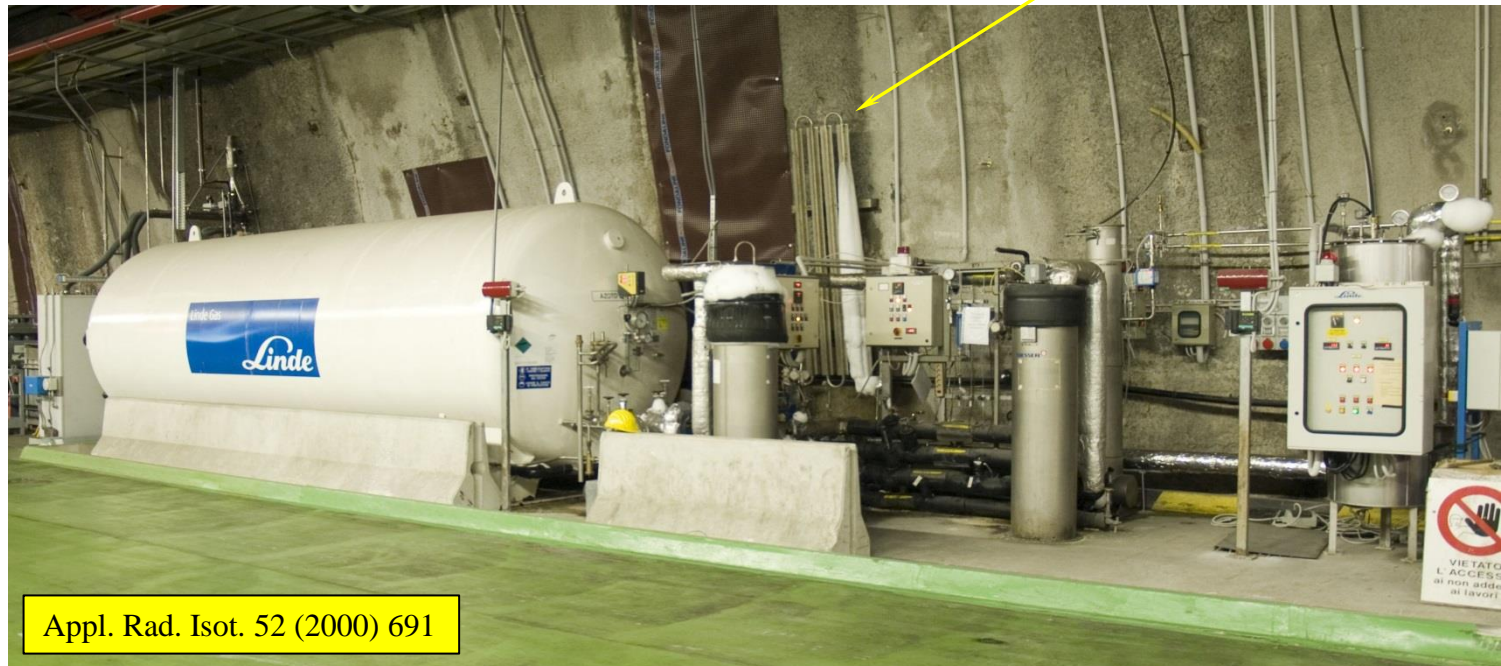
Used for: water stripping, cleaning, blanketing

$$C_{\text{Rn}} \sim 50 \mu\text{Bq}/\text{m}^3$$

$$C_{\text{Kr}} \sim 30 \text{ ppt}$$

$$C_{\text{Ar}} \sim 10 \text{ ppm}$$

Atmospheric
evaporator



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Available nitrogen qualities

- High Purity Nitrogen (HPN)

Liquid nitrogen purified in a charcoal-filled column (LTA)

LTA: ~2 kg of HP AC, LN₂ cooling, built-in E-heater for regeneration

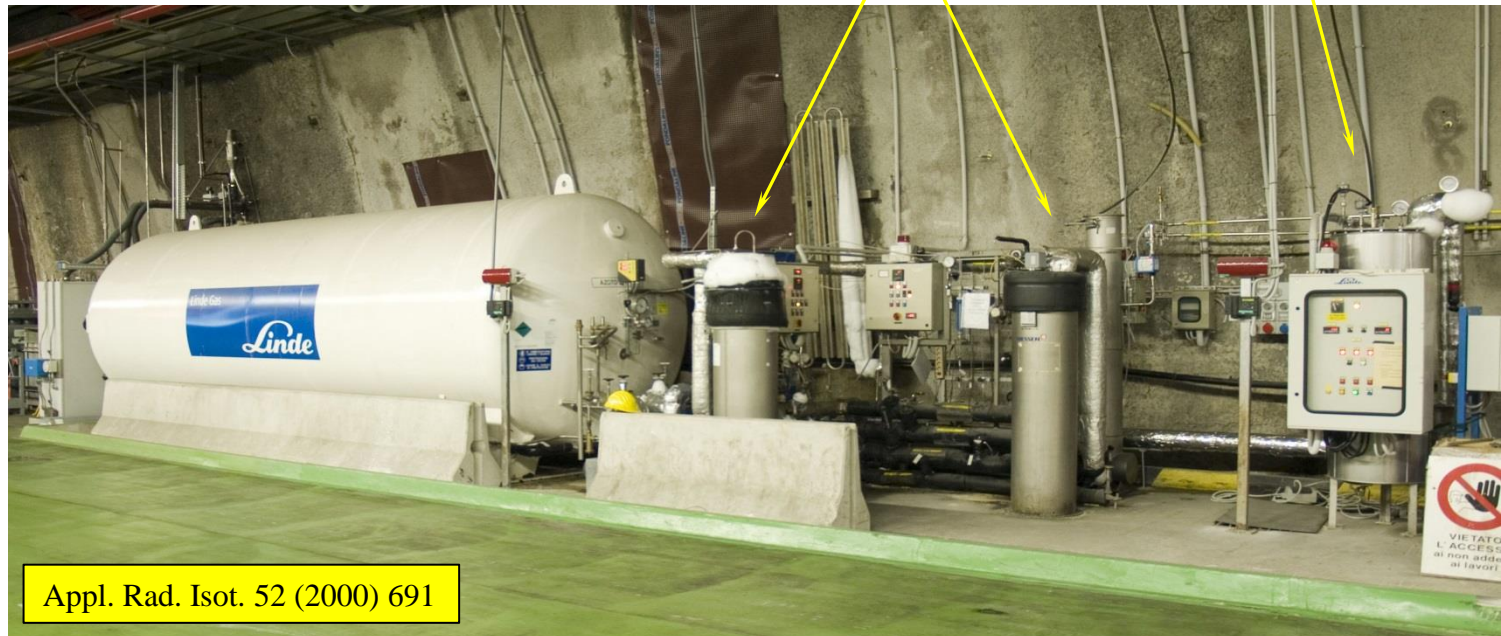
LN₂ evaporated after purification, N₂ flow up to 100 m³/h (STP)

Used for: stripping, cleaning, blanketing, ²²²Rn measurements,
production of synthetic air

$$C_{Rn} < 0.5 \mu\text{Bq}/\text{m}^3$$

Electric
evaporator

LTA columns



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LN₂ evaporated after purification, N₂ flow up to 100 m³/h (STP)

Used for: stripping, cleaning, blanketing, ²²²Rn measurements,
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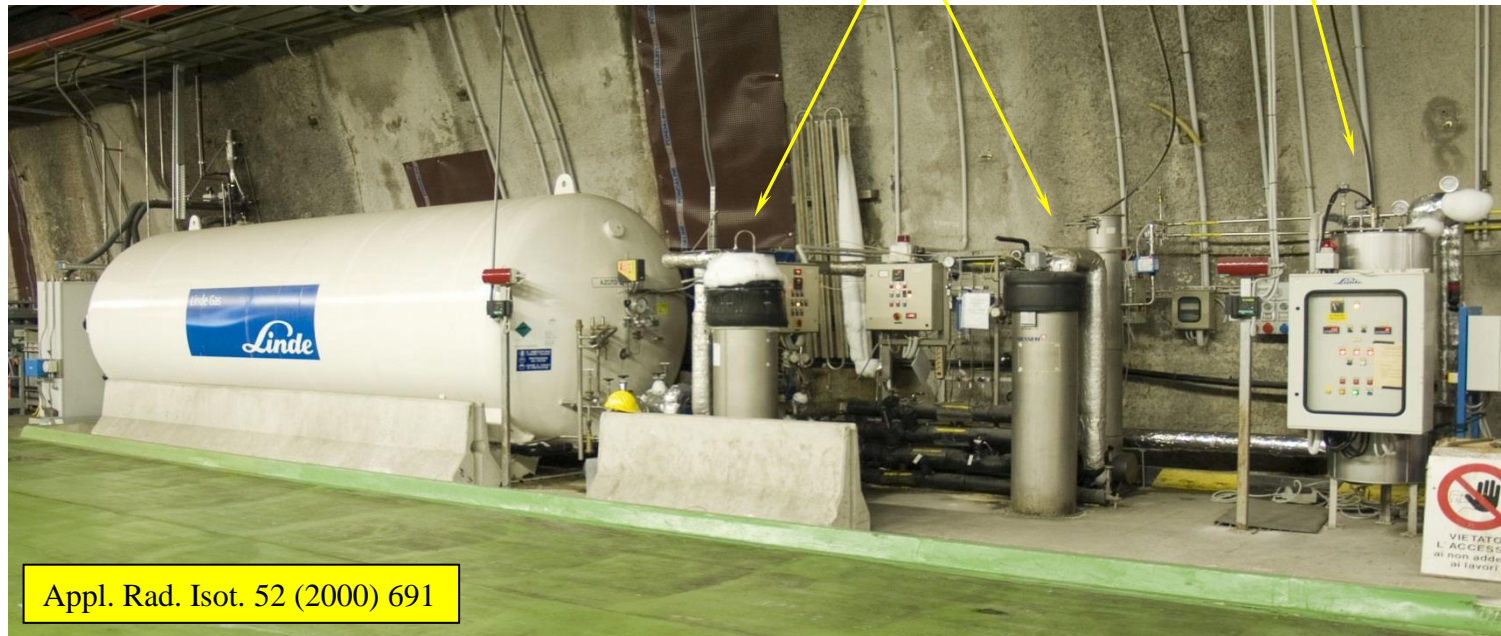
$$C_{\text{Rn}} < 0.5 \mu\text{Bq}/\text{m}^3$$

$$C_{\text{Kr}} \sim 30 \text{ ppt}$$

$$C_{\text{Ar}} \sim 10 \text{ ppm}$$

LTA columns

Electric
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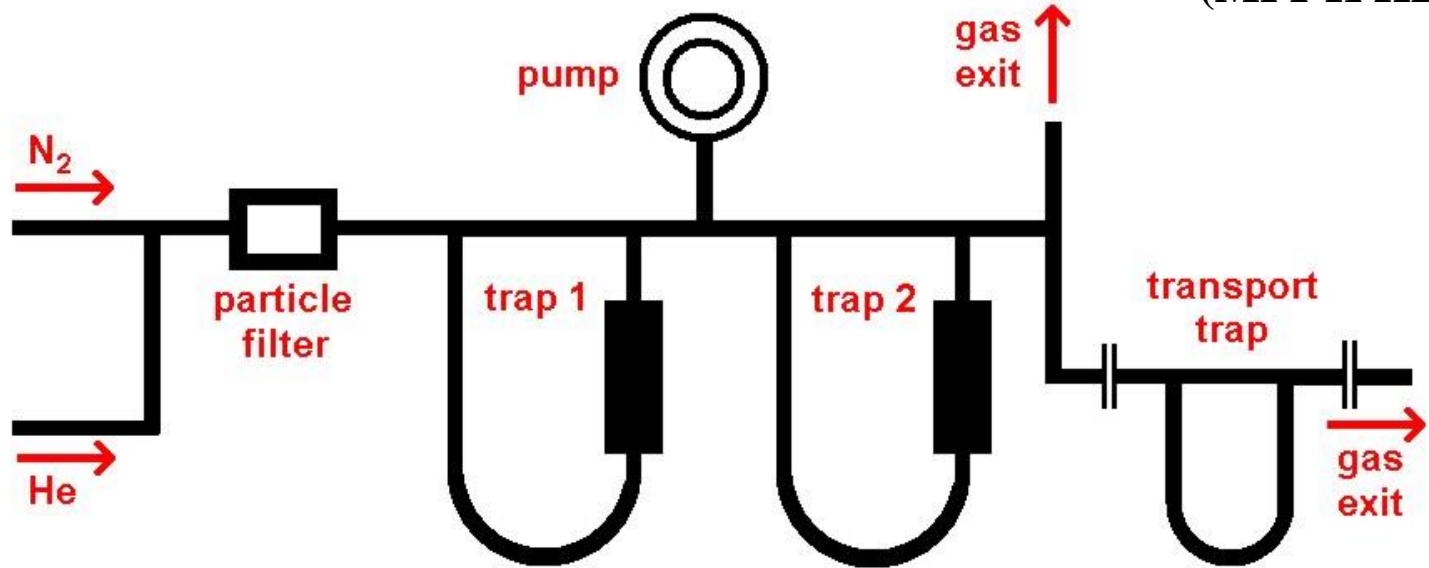
Rn diff/emanation

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Detection of ^{222}Rn – MoREx

(MPI-K HD)



BX nitrogen

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Conclusions

^{222}Rn in gases (N_2/Ar)

- ^{222}Rn adsorption on activated carbon
- several AC traps available (MoREx/MoRExino)
- pre-concentration from 100 – 200 m^3
- purification is possible (LTA)

^{222}Rn detection limit:
 $\sim 0.5 \mu\text{Bq}/\text{m}^3$ (STP)
[1 atom in 4 m^3]



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Detection of ^{222}Rn – MoREx

BX nitrogen

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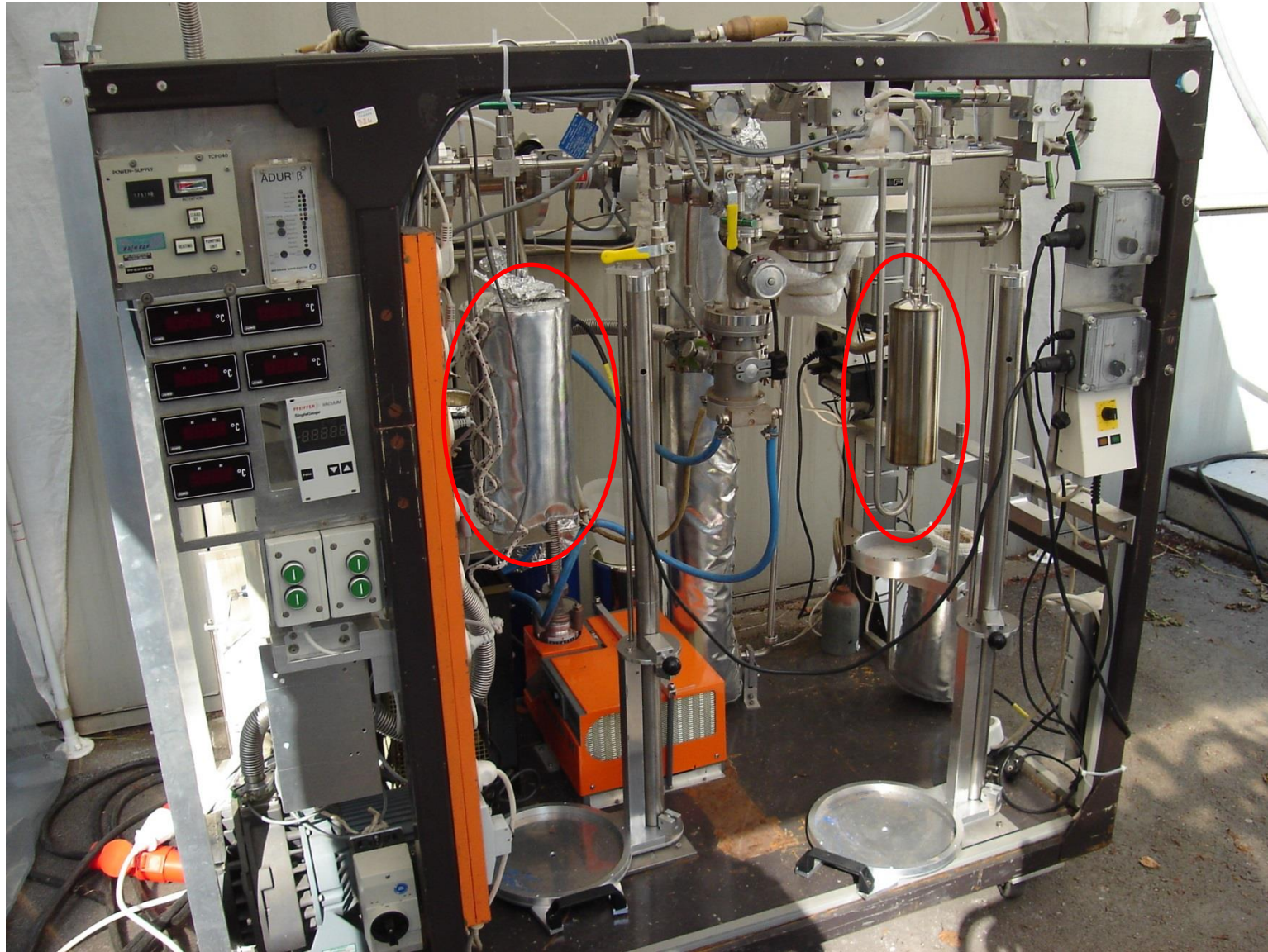
^{222}Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

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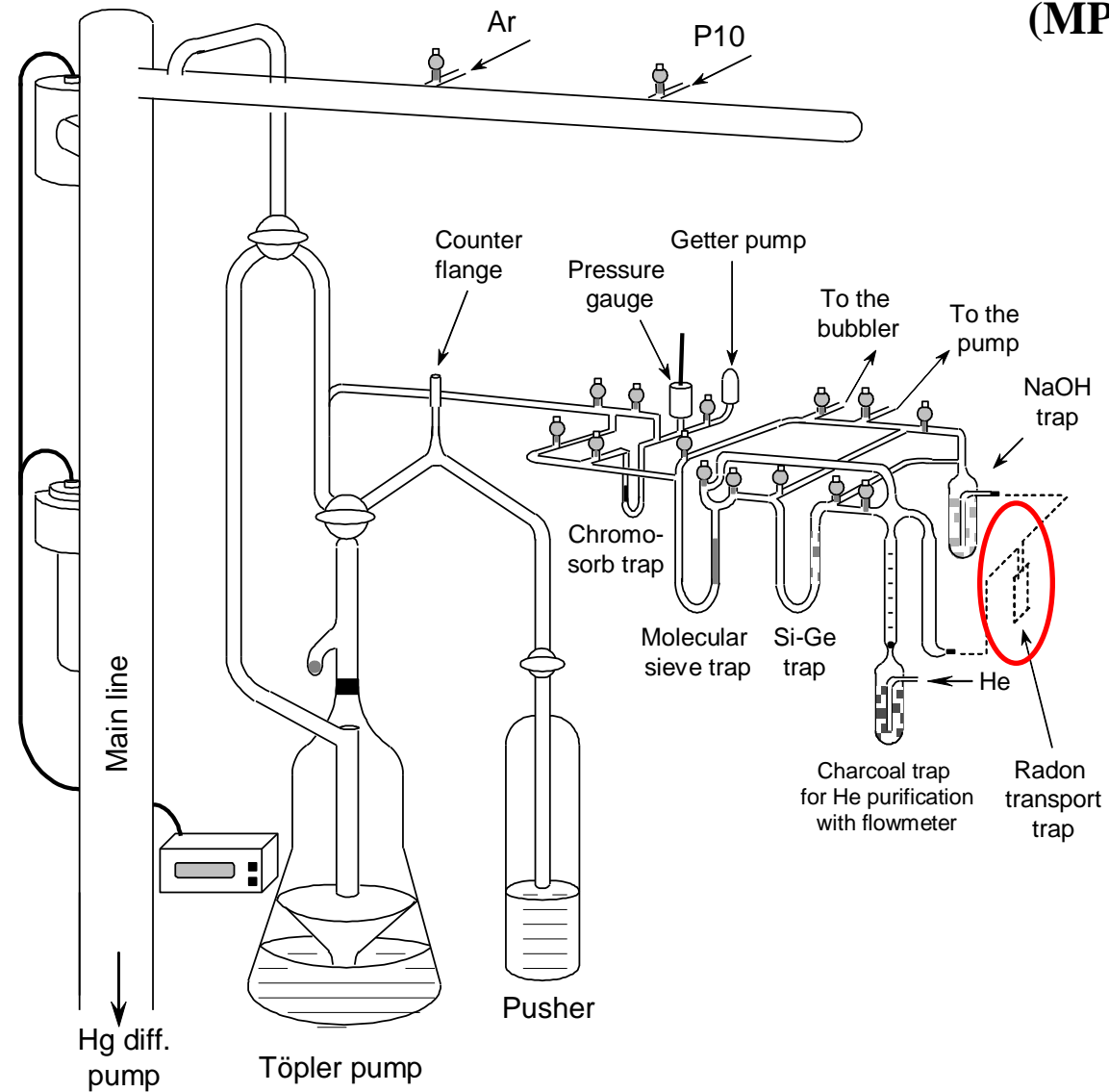




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Detection of ^{222}Rn – filling line

(MPI-K HD)



BX nitrogen

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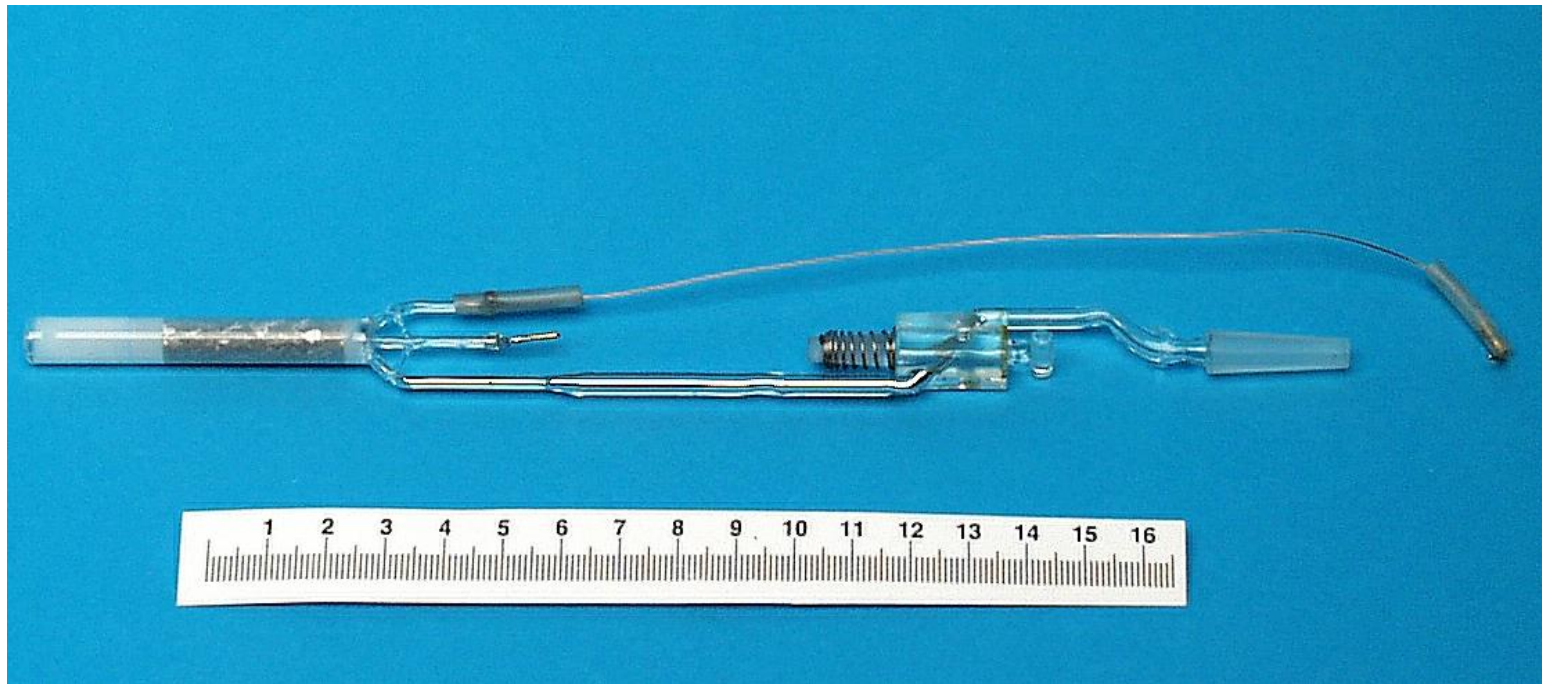
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Detection of ^{222}Rn – counters



BX nitrogen

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Conclusions

- Developed for the GALLEX/GNO experiment
- Hand-made at MPI-K ($\sim 1 \text{ cm}^3$ active volume)
- In case of ^{222}Rn only α -decays are detected
- 50 keV threshold
 - bcg: 0.1 – 2 cpd
 - total detection efficiency of ~ 1.5
- **Absolute detection limit $\sim 30 \mu\text{Bq}$ (15 atoms)**

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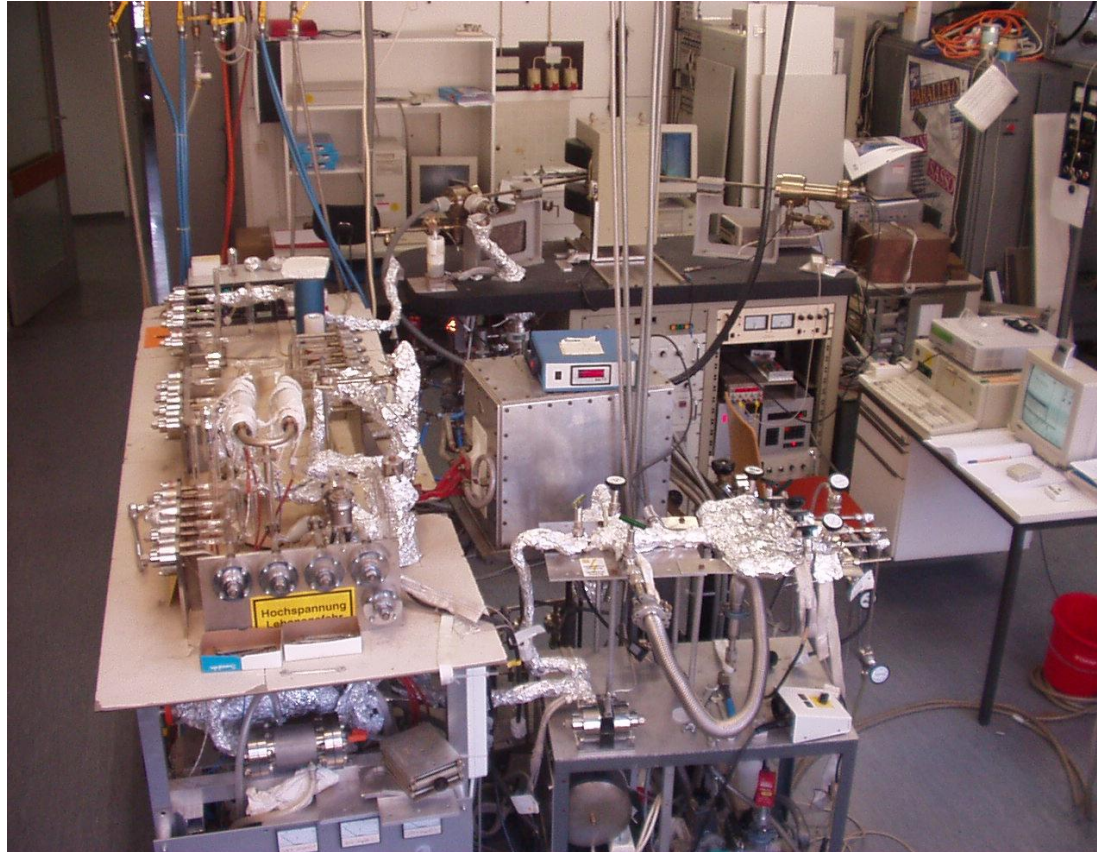
Rn-free clean room

Rn diff/emanation

Conclusions

Detection of Ar and Kr

(MPI-K HD)



NG VG 3600
magnetic sector field
spectrometer.

Used to investigate
noble gases in the
terrestrial and extra-
terrestrial samples.

Adopted to test the
nitrogen purity and
purification methods.

**Detection limits: Ar: 10^{-9} cm^3
Kr: 10^{-13} cm^3**

$V_s \sim 1 \text{ cm}^3$

**Ar: $\sim 1 \text{ ppb (STP)}$
Kr: $\sim 0.1 \text{ ppt (STP)}$**

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Requirements for LAKN purity

1 cpd in the FV from ^{39}Ar , ^{85}Kr and ^{222}Rn (100 nBq/m³ LS)



Solubilities Ar: $\text{N}_2/\text{PC} = 4.4$ ($\text{N}_2/\text{H}_2\text{O} = 37$)

Kr: $\text{N}_2/\text{PC} = 1.4$ ($\text{N}_2/\text{H}_2\text{O} = 21$)

Rn: $\text{N}_2/\text{PC} = 0.09$ ($\text{N}_2/\text{H}_2\text{O} = 4$)



0.5 $\mu\text{Bq } ^{39}\text{Ar}/\text{m}^3 \text{N}_2$

0.2 $\mu\text{Bq } ^{85}\text{Kr}/\text{m}^3 \text{N}_2$

0.01 $\mu\text{Bq } ^{222}\text{Rn}/\text{m}^3 \text{N}_2$



Specific Activities: 1.4 Bq/m³ (^{39}Ar in Ar)

1 MBq/m³ (^{85}Kr in Kr)

α/β , Bi-Po's: ^{222}Rn decays



0.4 ppm Ar in N₂

0.14 ppt Kr in N₂

7 $\mu\text{Bq } ^{222}\text{Rn}/\text{m}^3 \text{N}_2$

BX nitrogen

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Options considered to get LAKN

1. Additional purification of HPN

$C_{\text{Kr}} \sim 30 \text{ ppt} \rightarrow 0.2 \text{ ppt}$ (needed reduction factor = 150)

$C_{\text{Ar}} \sim 10 \text{ ppm} \rightarrow 0.4 \text{ ppt}$ (needed reduction factor = 25)

Distillation → efficient but difficult. Requires expertise, resources, new developments (time), new equipment, manpower etc.

Low temperature adsorption → only gas phase considered (liquid phase not effective), needed adsorber with optimized pore size distribution ($\sim 7 \text{ \AA}$ for Kr adsorption)

2. Delivery of pure enough gas to the experimental site

Nitrogen produced by distillation of air
→ can it be pure enough ?

How to deliver it to the experimental site
→ avoiding contamination during refilling and transportation

Can ^{222}Rn level (emanation) be low enough?



BX nitrogen

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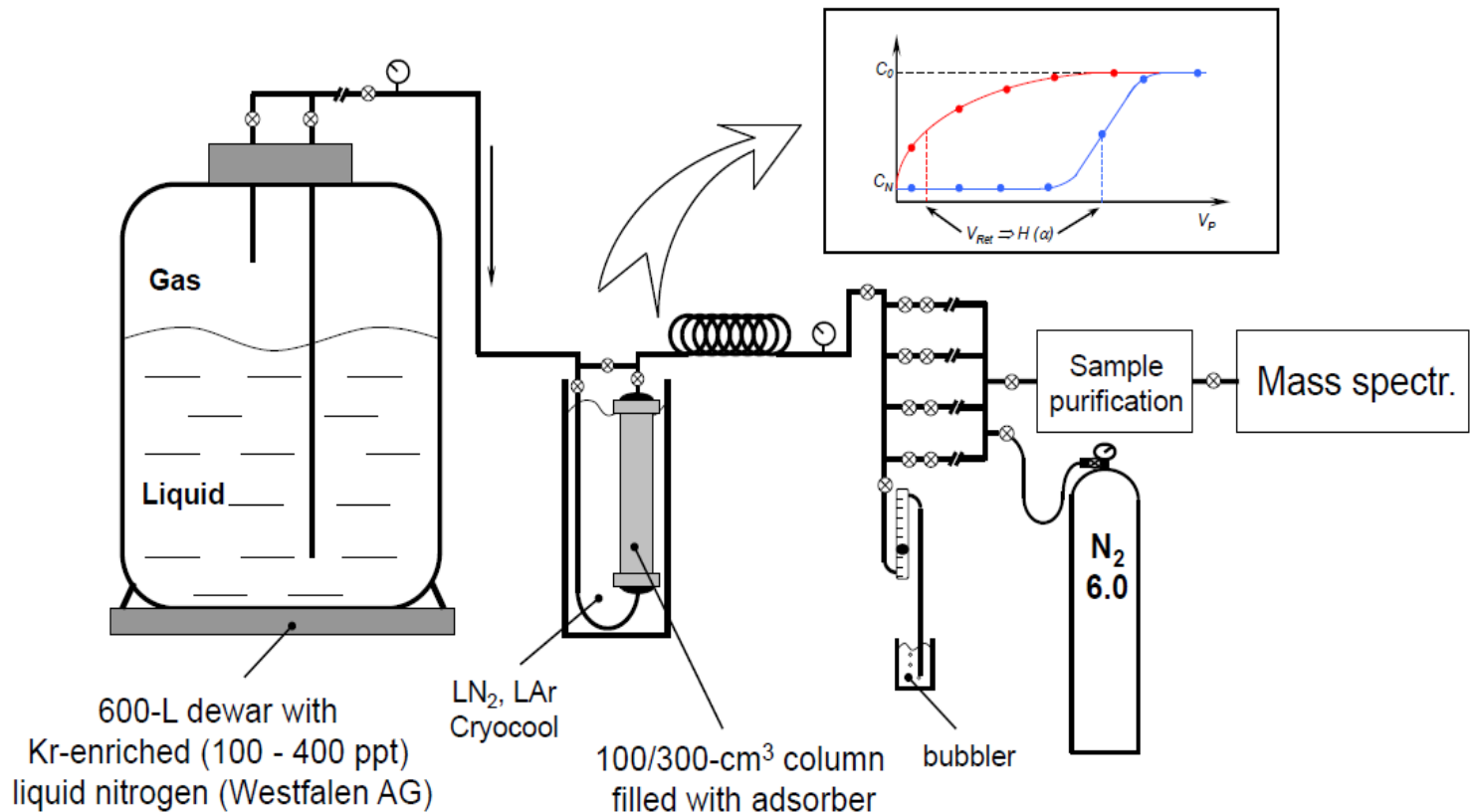
Conclusions



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Kr removal from nitrogen

Experimental setup used to study Kr removal from nitrogen gas by cryo-adsorption. The column filled with adsorber can be cooled by LN₂ (liquid phase adsorption) or LAr (gas phase adsorption).



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- BX nitrogen
- Requirements
- Purification
- LAKN development
- Synthetic air
- JUNO nitrogen
- ²²²Rn removal
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Kr removal from nitrogen

Main properties of the investigated carbon-based adsorbers. The first three products are "standard" activated carbons with wide pore size distributions. The other two are special products with narrow pore size distributions.

Product	F3/F4	C 38/2	FM 5-250	Carbosieve SIII	Skeleton 7Å
Manufacturer	CarboAct	CarboTech	Charcoal Cloth Int.	Supelco	Skeleton Technologies
Pore size distribution	unknown (wide distribution)	wide distribution, enhanced fraction around ~7 Å ~20 Å		narrow distribution (15 – 40 Å)	narrow distribution (7 Å)
Composition of product	grains	pressed cylinders	fabric	grains	grains
Size	0.85 – 2.8 mm	Length: 2 – 7 mm Diam.: 2 mm	Thickness: 1.2 – 1.4 mm	60 – 80 mesh	~70 μm
Density	~0.1 g/cm ³	0.65 g/cm ³	~210 g/m ²	0.61 g/cm ³	~0.6 g/cm ³
Surface area	1200 m ² /g	1300 m ² /g	1000 – 1200 m ² /g	820 m ² /g	1270 m ² /g

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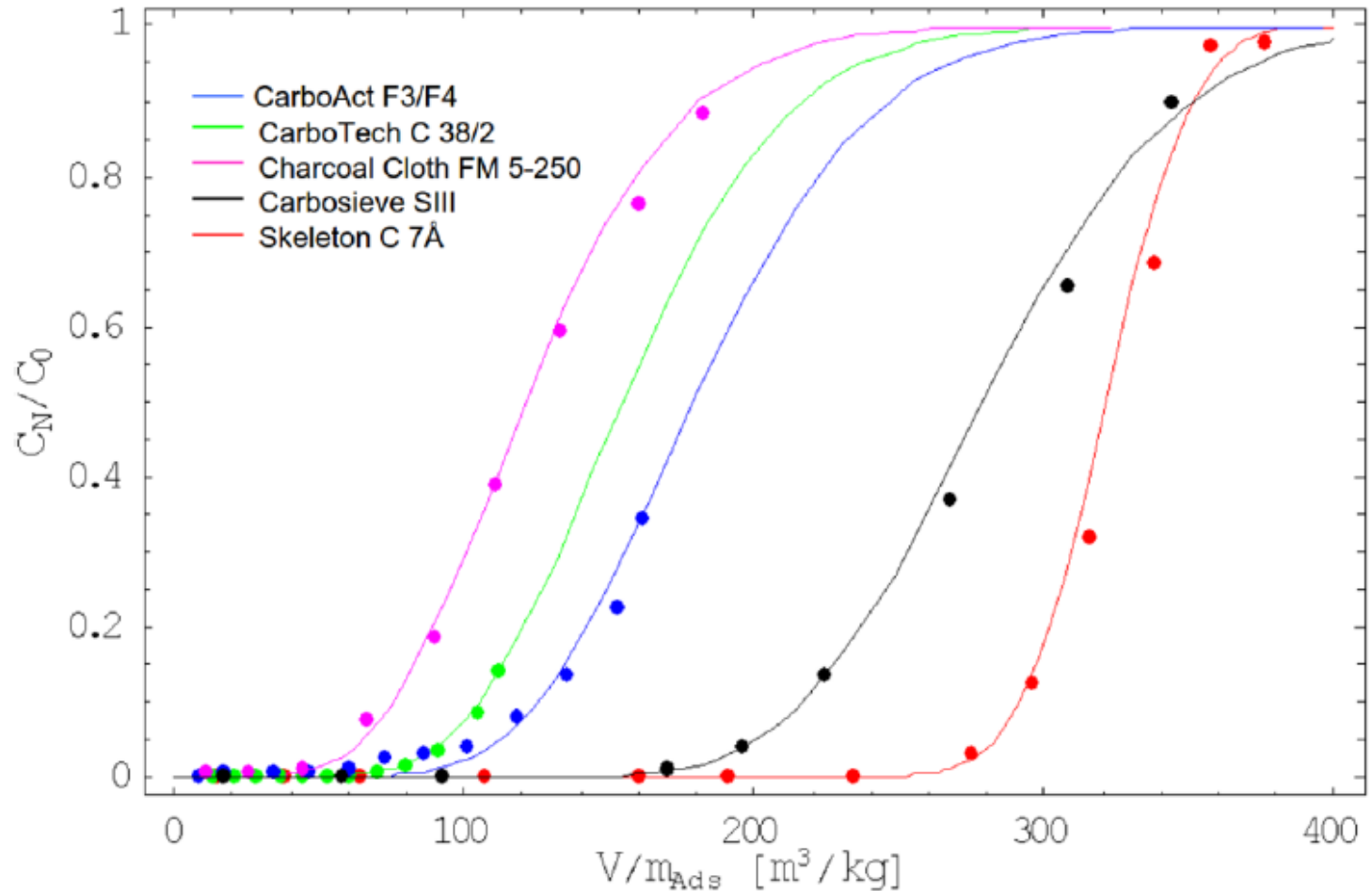
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Kr removal from nitrogen

Breakthrough curves for Kr adsorption from nitrogen on different carbon-based at the liquid argon temperature (gas phase adsorption).



BX nitrogen

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Kr removal from nitrogen

Results of Kr adsorption from gaseous nitrogen on different types of carbon-based adsorbers cooled by LN₂.

Adsorber	<i>HETP</i> [mm]	<i>H</i> [10 ⁻² mol/Pa/kg]	<i>V_{Ret}/m_{Ads}</i> [m ³ /kg]
CarboAct [®]	275	2.4	17
CarboTech C38/2	34	1.6	12
Carbon Cloth	21	2.9	21
Carbosieve SIII	110	8.3	60
Skeleton 7Å	55	10	73

Results of Kr adsorption from gaseous nitrogen on different types of carbon-based adsorbers cooled by LAr.

Adsorber	<i>HETP</i> [mm]	<i>H</i> [10 ⁻² mol/Pa/kg]	<i>V_{Ret}/m_{Ads}</i> [m ³ /kg]
CarboAct [®]	15	21	183
CarboTech C38/2	17	19	158
Carbon Cloth	24	16	125
Carbosieve SIII	10	34	282
Skeleton 7Å	1.6	37	321

BX nitrogen

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Rn-free clean room

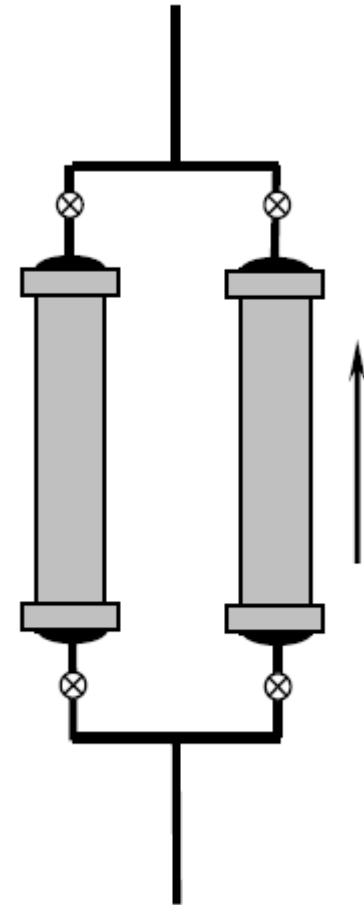
Rn diff/emanation

Conclusions



Kr removal from nitrogen - summary

- The BOREXINO experiment required krypton-free nitrogen at a flow rate of 30 m³/h.
- Assuming a column filled with 10 kg of Skeleton 7 Å, a permanent operation for about 4 d would be possible
- 4 d would be sufficient to regenerate a second identical column
- 20 kg of the adsorber would be needed to guarantee a continuous supply of purified nitrogen (a system of two parallel columns)



→ Ar not removed, high costs of the adsorber

BX nitrogen

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Development of LAKN delivery chain

Survey of the nitrogen available on the European market

Supplier	Gas quality	C_{Ar} [ppm]	C_{Kr} [ppt]
Messer Griesheim, Oberhausen	4.0	200 ± 30	1680 ± 240
Air Liquide, Terni	4.0	11.0 ± 1.3	40 ± 5
Air Liquide, Terni	4.0 (after) ^{222}Rn removal ³)	10.4 ± 1.2	33 ± 4
Linde AG, Worms	7.0 (LIPUR)	0.060 ± 0.002	0.06 ± 0.01
SOL, Mantova	6.0	0.063 ± 0.006	0.04 ± 0.01
Westfalen AG, Hörstel	6.0	$(5.0 \pm 0.8) \times 10^{-4}$	0.06 ± 0.02
Goal		≤ 0.4	≤ 0.14

- Nitrogen taken directly from production plants as liquid into a dedicated transport dewar (e.g. no pressure build-up system)
- Filling done after a careful selection of ports and after a long flushing of the connecting tubes and the dewar
- Close cooperation with companies was necessary
- Dewar with LN_2 connected to the NG mass spectrometer

BX nitrogen

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Development of LAKN delivery chain

Linde provided a transportable storage tank filled with LAKN and brought to Heidelberg for further tests: Rn, Ar and Kr measurements (vs. filling level)

BX nitrogen

Requirements

Purification

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JUNO nitrogen

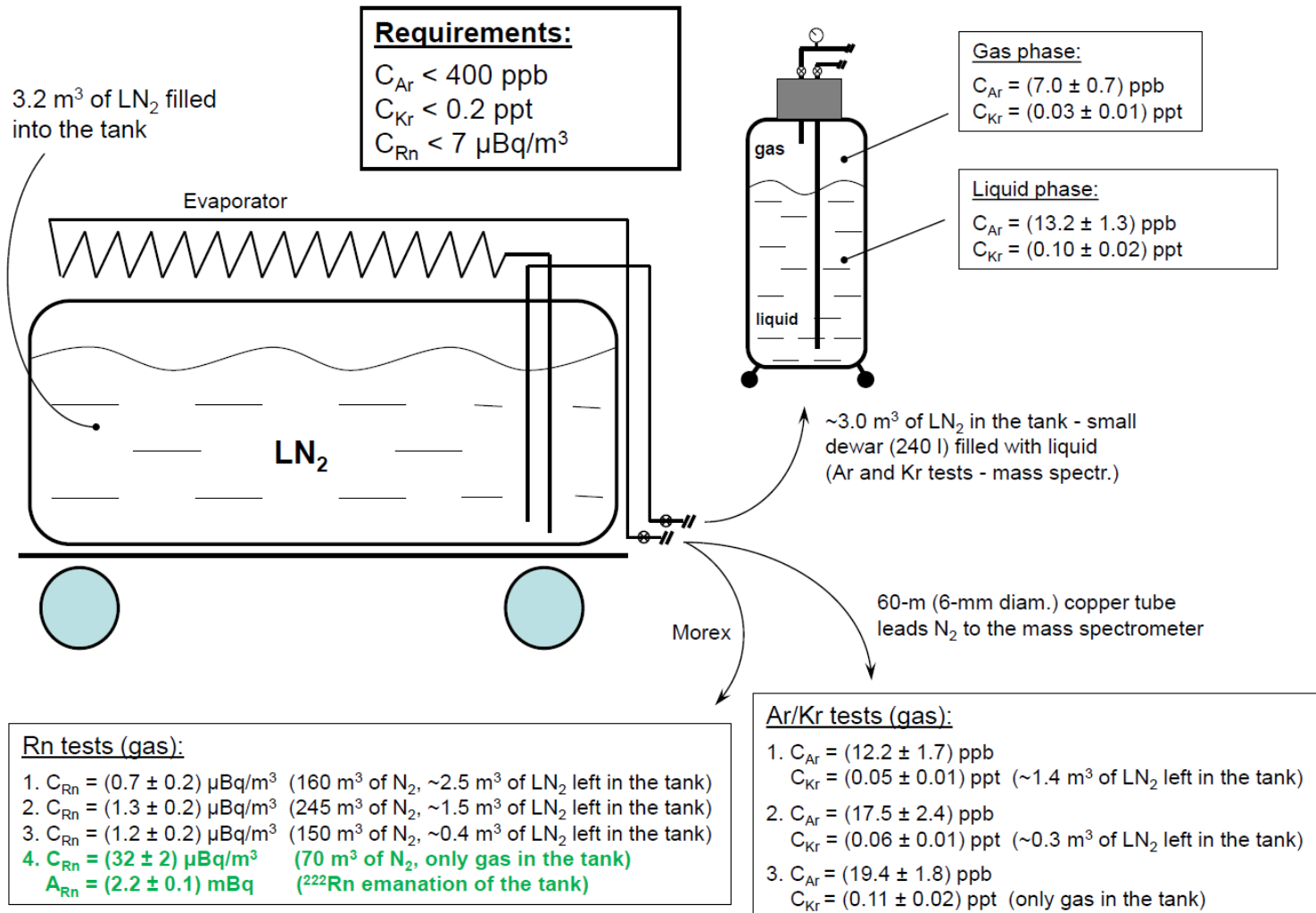
^{222}Rn removal

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Development of LAKN delivery chain

SOL built a dedicated LAKN storage tank (all-metal sealed, special cleaning) with electric evaporator, all installed on a transportable platform

BX nitrogen

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BX nitrogen

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Development of LAKN delivery chain

- Tank delivered cold, with 250 L of LAKN inside for some initial tests, like its ^{222}Rn emanation (60 mBq but still fine, $C_{\text{Rn}} \sim 5 \mu\text{Bq}/\text{m}^3$).
- Tank connected directly to the mass spectrometer (analysis of Ar and Kr) and to MoREx (analysis of ^{222}Rn)
- In the next step the the tank filled from a dedicated tanker by a dedicated driver according to the procedure worked out with the company (LAKN produced directly into the tanker, extended purging with warm gas of the connecting tubes/hoses etc.)



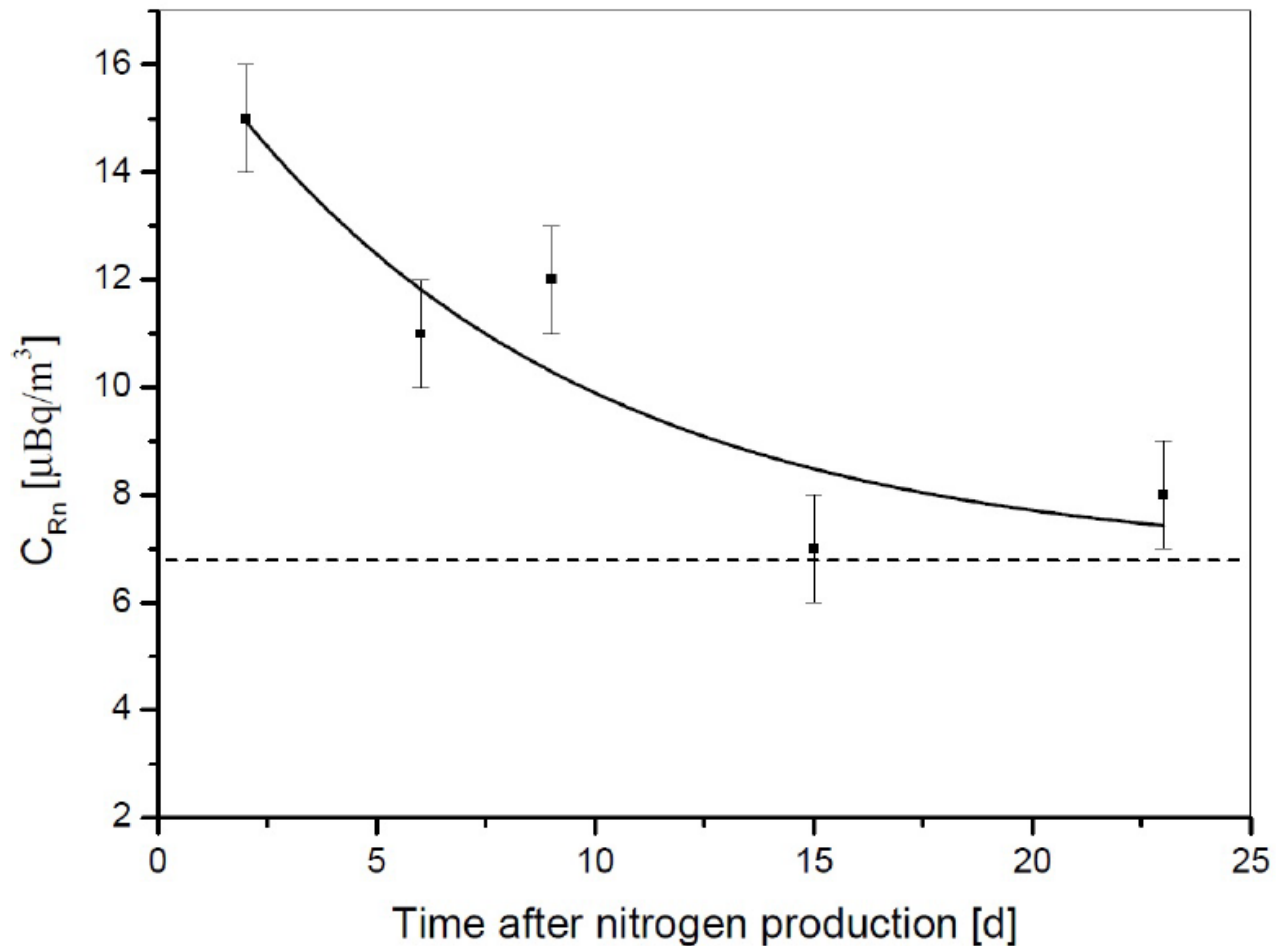
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Development of LAKN delivery chain

^{222}Rn specific activity in the SOL LAK nitrogen as a function of time elapsed since the gas production. The curve fitted to the experimental data reproduces the half-life of ^{222}Rn .



BX nitrogen

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Development of LAKN delivery chain

Ar, Kr and ^{222}Rn content in the SOL LAK nitrogen as a function of the filling level of the storage tank

LAr amount [m^3]	C_{Ar} [ppb]	C_{Kr} [ppt]	C_{Rn} [$\mu\text{Bq}/\text{m}^3$]
14.5	12.3 ± 1.7	0.030 ± 0.006	7.0 ± 1.0
14.2	11.1 ± 1.5	0.016 ± 0.004	5.5 ± 0.6
13.5	11.7 ± 1.6	0.011 ± 0.004	7.6 ± 0.7
6.0	7.4 ± 1.1	0.006 ± 0.004	10.0 ± 1.0
3.0	12.3 ± 1.8	0.024 ± 0.006	11.0 ± 1.0
0.8	16.0 ± 2.0	0.031 ± 0.008	38.0 ± 5.0
0.0 (Tank empty)	19.0 ± 3.0	0.030 ± 0.010	237 ± 15
Goal	≤ 400	≤ 0.14	≤ 7

For $V_{LN} > 0.8 \text{ m}^3$ in the tank practically all the specifications were met. For lower amounts the ^{222}Rn content increased sharply. Therefore, taking into account some safety margins it was decided that **during the normal operations the tank should always be at least 20% full** (min. 3 m^3 of LN_2 inside).



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Development of LAKN delivery chain

LAK tank installed at LNGS (next to entrance to Hall C)

BX nitrogen

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Development of LAKN delivery chain

Ar and Kr content in the SOL LAK nitrogen sampled at LNGS

Sample No	Conditions	Sampling port	C_{Ar} [ppb]	C_{Kr} [ppt]
1	Tank half-full	Storage tank	13 ± 2	0.22 ± 0.03
2	Tank full	Storage tank	30 ± 5	0.03 ± 0.01
3a	Tank full	Storage tank	8.6 ± 0.9	0.04 ± 0.01
3b	Tank full	End of distrib. line	8.7 ± 0.9	0.05 ± 0.01
4a	Lowest allowed filling level	Storage tank	12.8 ± 1.4	0.15 ± 0.02
4b	Lowest allowed filling level	End of distrib. line	12.5 ± 1.4	0.14 ± 0.02

- Various sources (suppliers) of the nitrogen gas fulfilling very strong purity requirements of BOREXINO were identified
- Procedure to deliver the LAK nitrogen to the experimental site was established
- Appropriate monitoring technique was developed

The LAK nitrogen is the radio-purest gas ever delivered to an experimental site and it was crucial to the success of BOREXINO



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Synthetic air

The BOREXINO inner vessel had to be inflated in the stainless steel sphere with a Rn-free gas. HPN could not be used for safety reasons → synthetic air has been produced for this purpose.

BX nitrogen

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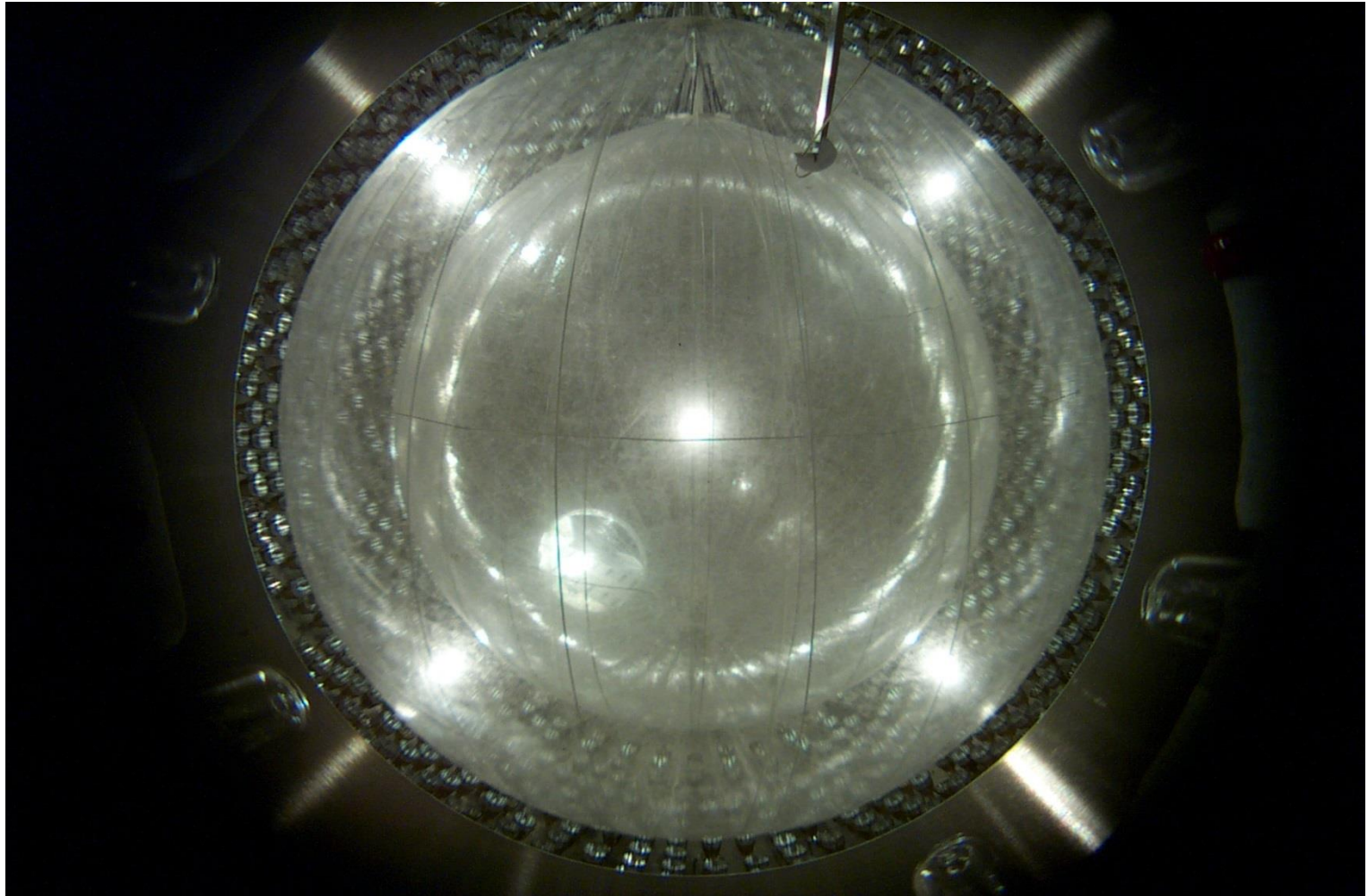
^{222}Rn removal

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Synthetic air

The synthetic air was produced by mixing HPN with oxygen extracted from standard gas cylinders, selected with respect to ^{222}Rn emanation. A commercial mixer, also tested for ^{222}Rn emanation, was used.

Sample	Description	^{222}Rn activity
Single cylinder	50 l, carbon steel	(2.32 ± 0.43) mBq
Cylinder package No.1	16 cylinders	(35.1 ± 6.9) mBq
Cylinder package No.2	16 cylinders	(12.9 ± 0.7) mBq
Gas mixer	$\text{N}_2 - \text{O}_2$ mixer	(1.61 ± 0.68) mBq



From all the emanation measurements a ^{222}Rn concentration in the synthetic air of

$$< 0.1 \text{ mBq/m}^3$$

could be estimated. This was confirmed by a null measurement with the radon monitor. Produced synthetic air had the lowest ^{222}Rn level ever obtained in breathable air.

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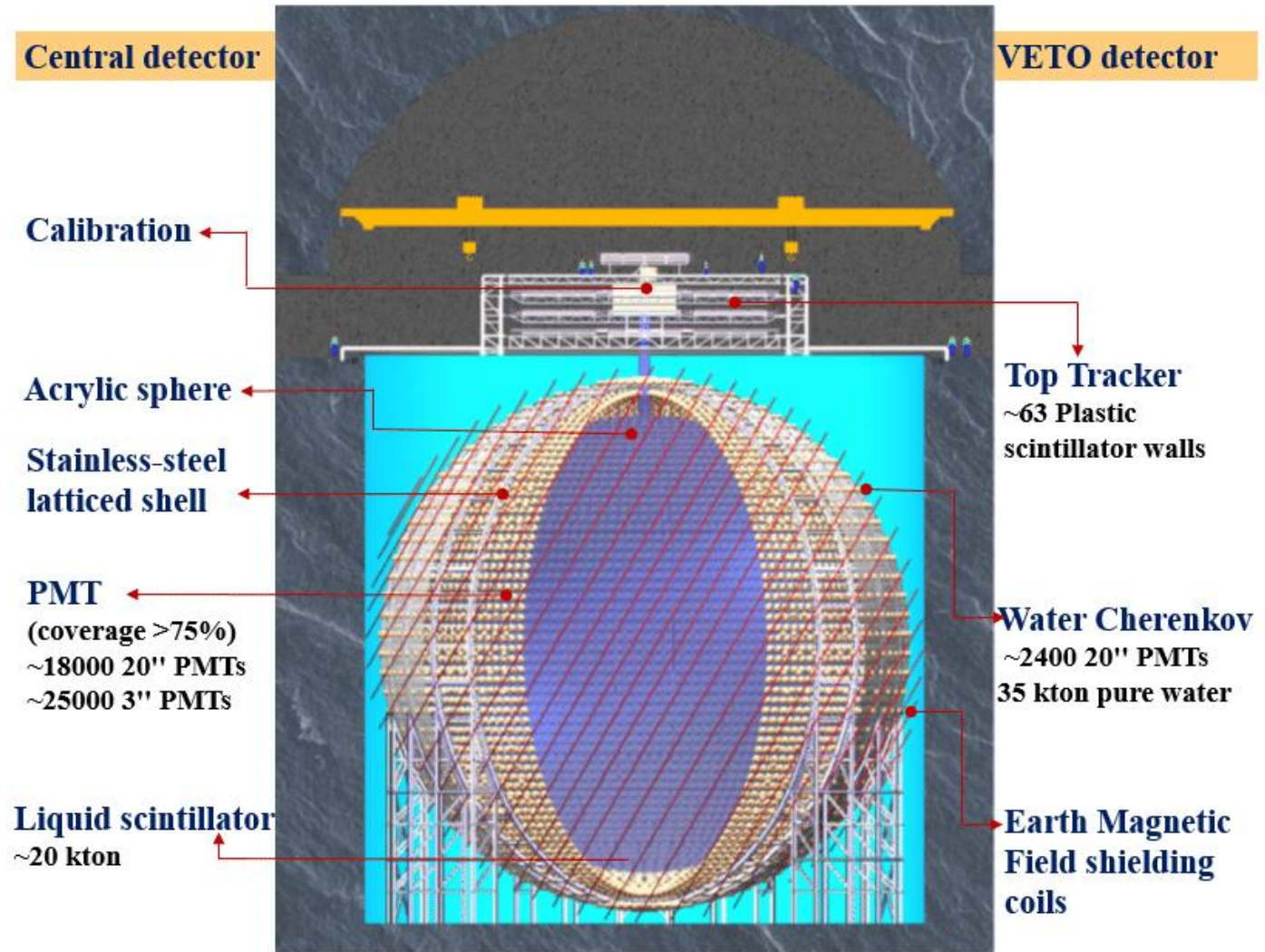
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JUNO detector

JUNO Detector



BX nitrogen

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Conclusions



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Purification of nitrogen for JUNO

High Purity Nitrogen (HPN) is used in JUNO mainly for detector purging, pipe cleaning and scintillator purification.

Requirements:

- Production rate up to 50 m³/h (STP)
- $C_{\text{Rn}} < 10 \mu\text{Bq/m}^3$
- $C_{\text{Kr-85}} < 50 \mu\text{Bq/m}^3 \rightarrow C_{\text{Kr}} < 35 \text{ ppt}$
- $C_{\text{Ar-39}} < 1 (50) \mu\text{Bq/m}^3 \rightarrow C_{\text{Ar}} < 0.8 (40) \text{ ppm}$

BX nitrogen

Requirements

Purification

LAKN development

Synthetic air

JUNO nitrogen

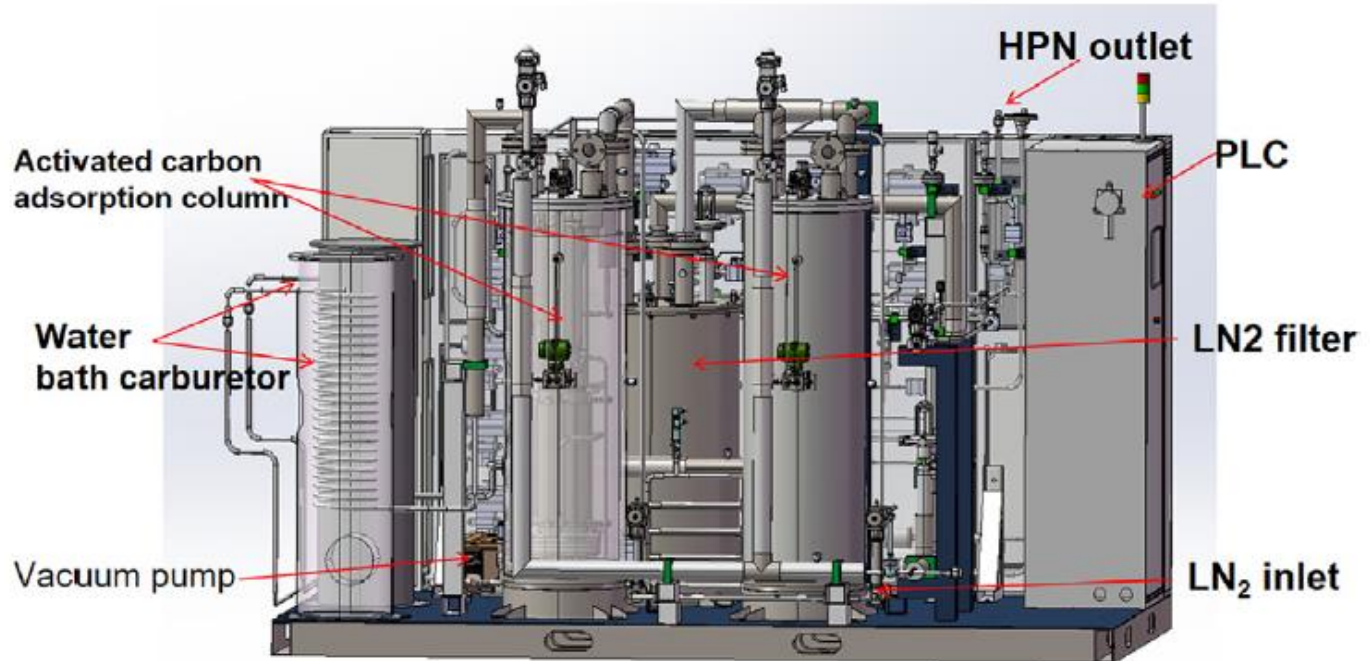
²²²Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

Conclusions



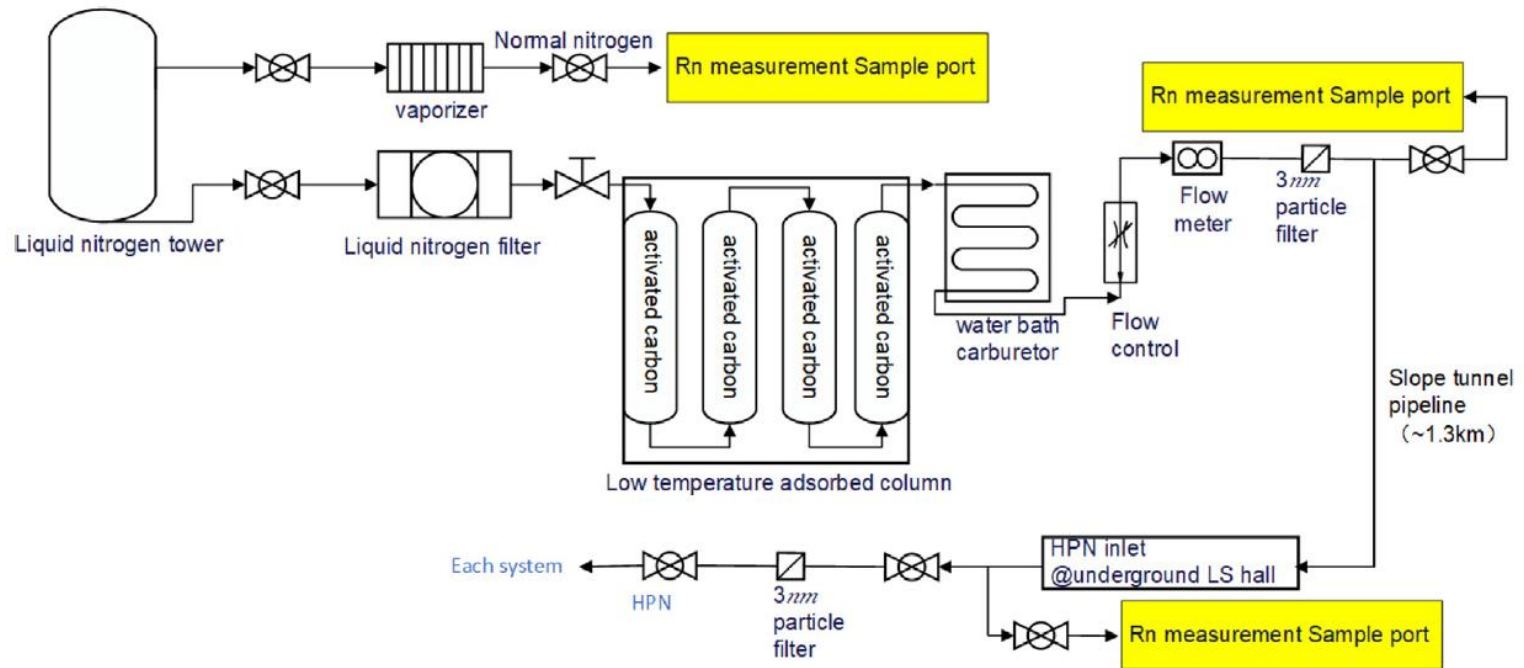


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Purification of nitrogen for JUNO

Nitrogen purification from radon:

- Liquid phase purification
- Cryo-adsorption of ^{222}Rn on high purity activated carbon



$$\text{HPN flow} = 50 \text{ m}^3/\text{h} \Rightarrow C_{\text{Rn}} = 1.3 / 5.6 \text{ } \mu\text{Bq}/\text{m}^3 \text{ (plant / underground)}$$

BX nitrogen

Requirements

Purification

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Synthetic air

JUNO nitrogen

^{222}Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

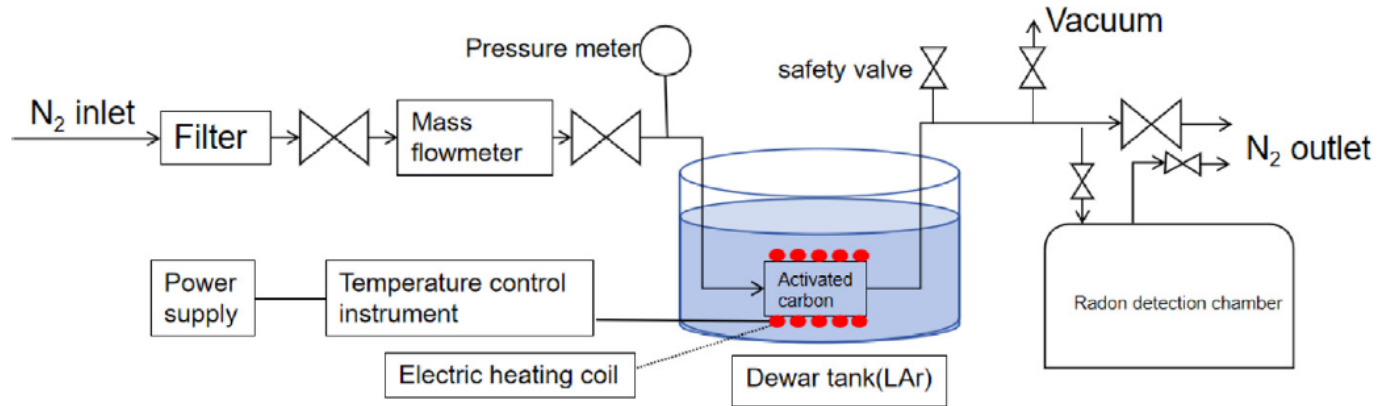
Conclusions



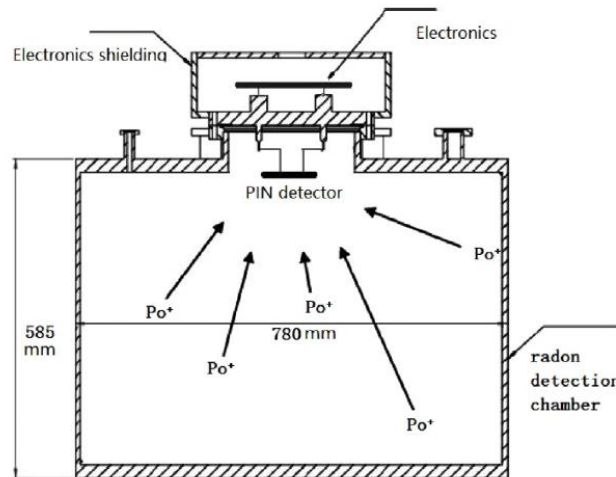
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Purification of nitrogen for JUNO

Radon measurement in HPN based on pre-concentration an AC trap (cooled with LAr) and counting of the Rn activity with an electrostatic detector.



Radon enrichment system



Electrostatic
radon detector

Appl. Rad. Isot. 208 (2024) 111305

- BX nitrogen
- Requirements
- Purification
- LAKN development
- Synthetic air
- JUNO nitrogen
- ²²²Rn removal
- Ar and Kr removal
- Rn-free clean room
- Rn diff/emanation
- Conclusions

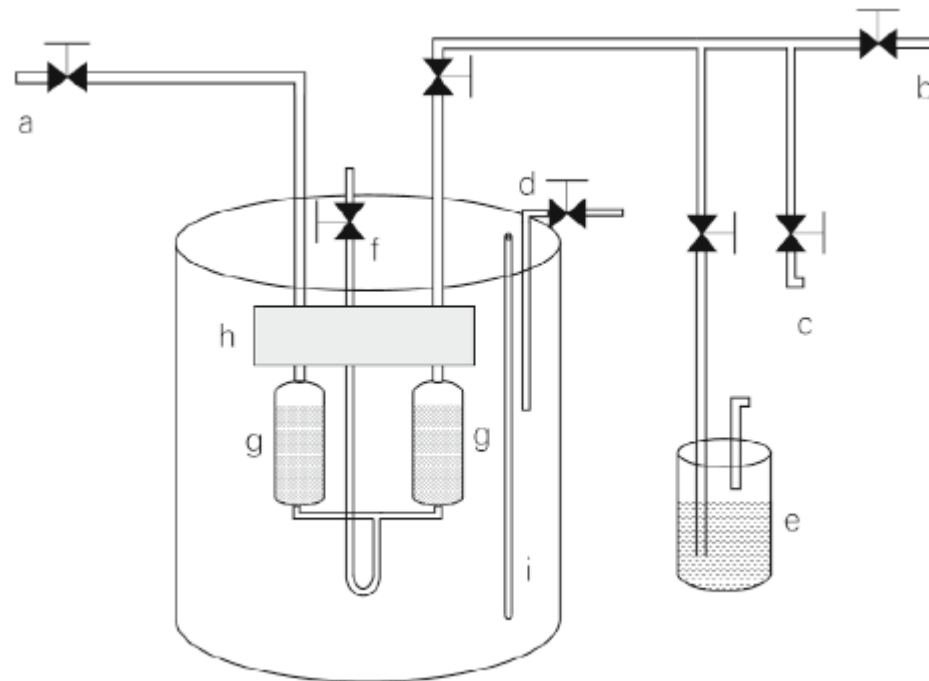


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Purification of nitrogen for JUNO

Nitrogen purification from Ar and Kr:

- Liquid phase purification
- Cryo-adsorption of Ar and Kr on coconut shell activated carbon (nitrogen purification pilot plant)
- Measurement of Ar and Kr performed with a mass spectrometer (Helix MC Plus)



BX nitrogen

Requirements

Purification

LAKN development

Synthetic air

JUNO nitrogen

^{222}Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

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Purification of nitrogen for JUNO

Nitrogen purification from Ar and Kr: results

Sample name	^{84}Kr concentration	^{85}Kr specific activity
Normal nitrogen	56.1 ± 5.61 ppt	98.42 ± 9.84 $\mu\text{Bq}/\text{m}^3$
Overground HPN (March)	3.9 ± 0.39 ppt	6.84 ± 0.68 $\mu\text{Bq}/\text{m}^3$
Overground HPN (July)	1.5 ± 0.15 ppt	2.63 ± 0.26 $\mu\text{Bq}/\text{m}^3$
Underground HPN	17.9 ± 1.79 ppt	31.4 ± 3.14 $\mu\text{Bq}/\text{m}^3$
Underground air	1.09 ± 0.1 ppm	1.91 ± 0.19 $\mu\text{Bq}/\text{m}^3$
Coconut shell activated carbon	0.26 ± 0.03 ppt	0.46 ± 0.46 $\mu\text{Bq}/\text{m}^3$

Sample name	^{40}Ar concentration	^{39}Ar specific activity
Normal nitrogen	4.28 ± 0.43 ppm	4.28 ± 0.43 $\mu\text{Bq}/\text{m}^3$
Overground HPN (March)	3.6 ± 0.36 ppm	3.6 ± 0.36 $\mu\text{Bq}/\text{m}^3$
Overground HPN (July)	15 ± 1.5 ppm	15 ± 1.5 $\mu\text{Bq}/\text{m}^3$

- Effect of Kr removal from HPN
- Underground HPN affected by the long pipe?
- Ar not removed from HPN

BX nitrogen

Requirements

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Rn diff/emanation

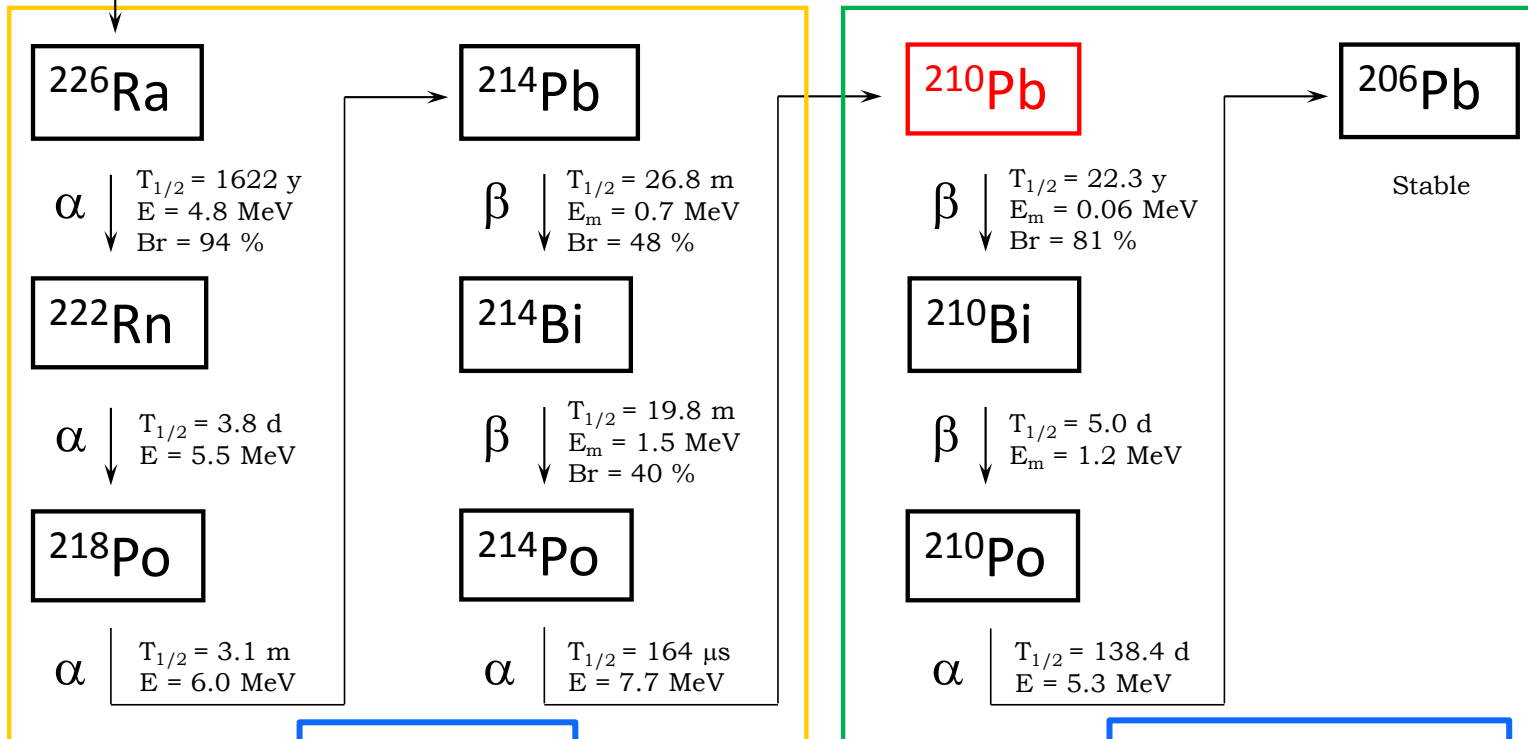
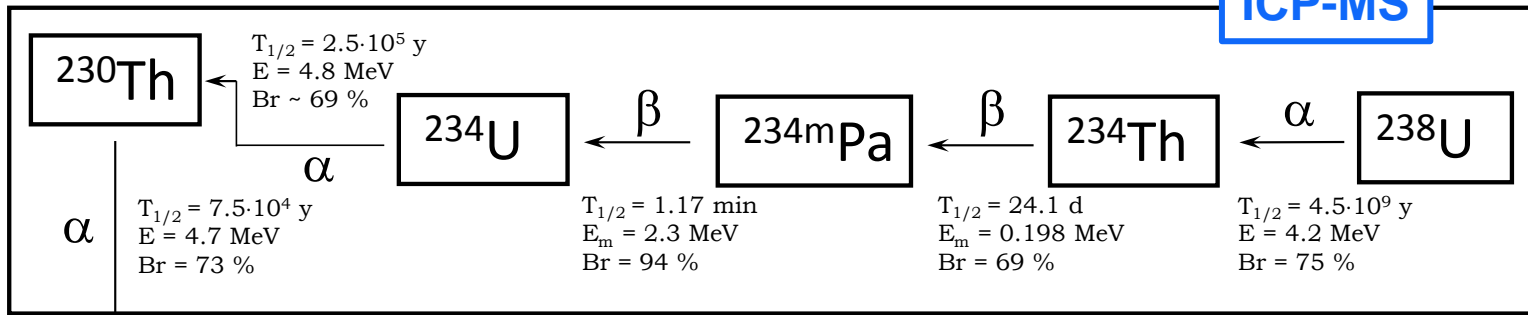
Conclusions



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^{222}Rn -free clean rooms

ICP-MS



HPGe / Rn

α spectrometry

BX nitrogen

Requirements

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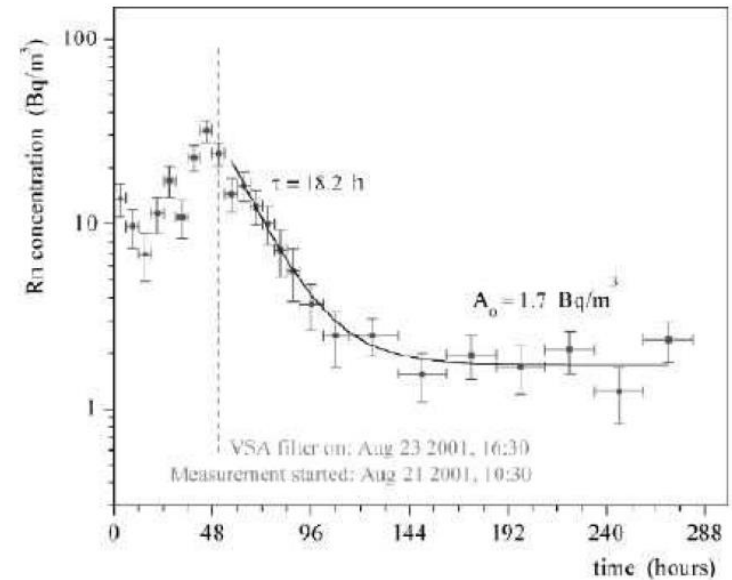
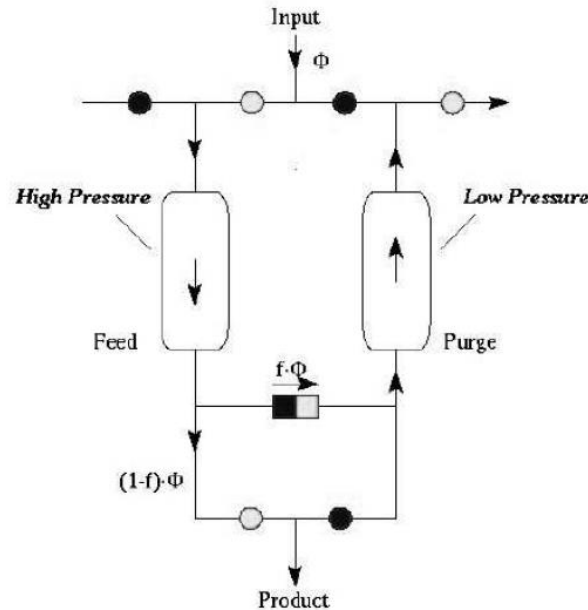


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^{222}Rn -free clean rooms

Long-lived radon daughters plating out the surface (^{210}Pb) of the BOREXINO Inner Vessel will produce ^{210}Bi and ^{210}Po . In DM detectors ^{210}Po may cause alpha-induced nuclear recoils or produce neutrons in the α -n reactions

Assembly of the IV in a first clean room with ^{222}Rn -reduced atmosphere



PSA system with two AC columns
used to purify the air ($\sim 100 \text{ m}^3/\text{h}$)

^{222}Rn content in the air $\sim 1.5 \text{ Bq/m}^3$

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BX nitrogen

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^{222}Rn -free clean rooms

Scheme of the DARKSIDE ^{222}Rn abatement system installed in Hall C

Provided by ATEKO

Air flow rate: 200 m³/h

$C_{\text{Rn}} \sim 1 \text{ mBq/m}^3$

Adsorption col. AC₁/AC₂:

- vol. 1.5 m³ each

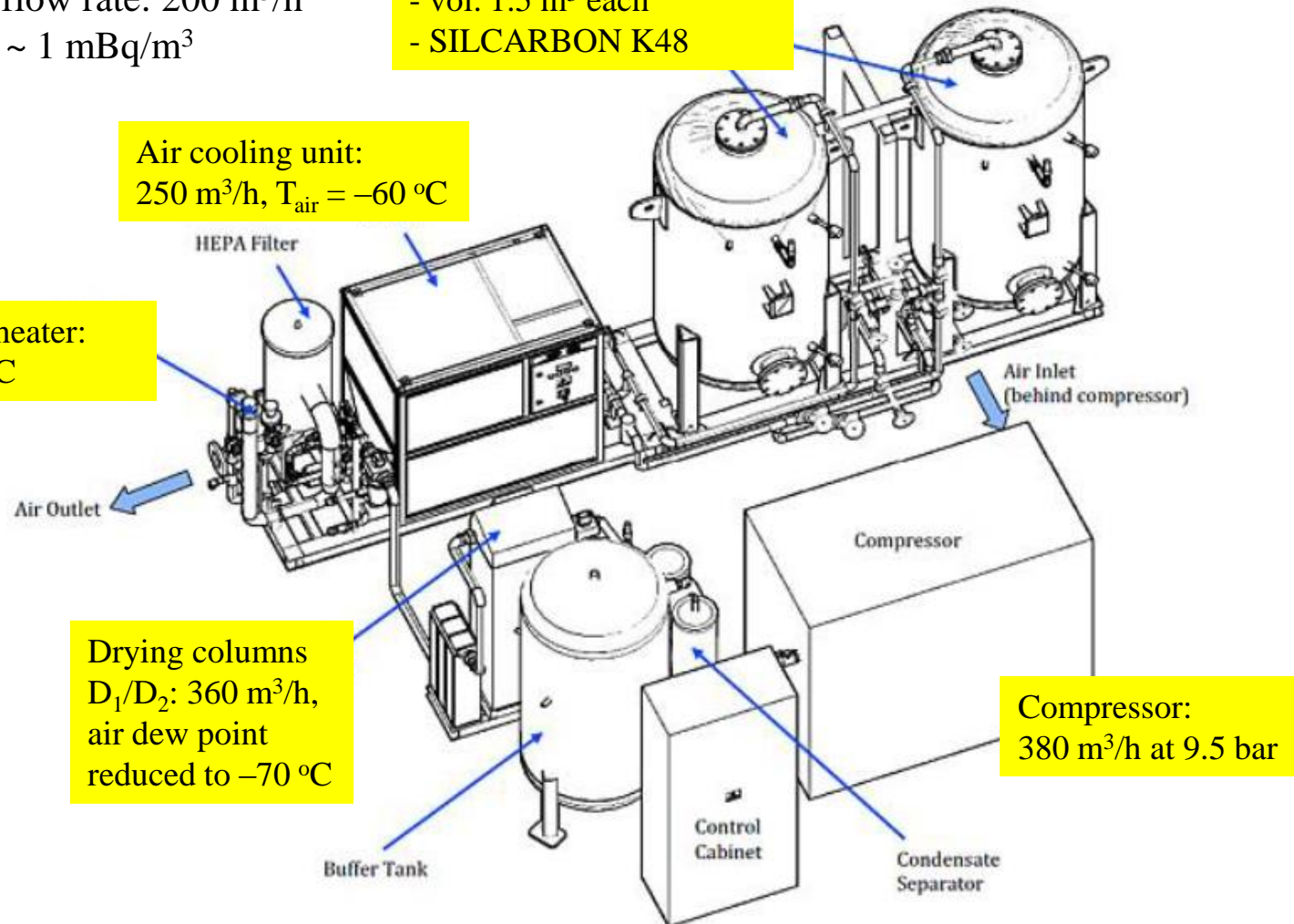
- SILCARBON K48

Air cooling unit:

250 m³/h, $T_{\text{air}} = -60 \text{ }^\circ\text{C}$

Air heater:

20 °C



Compressor:
380 m³/h at 9.5 bar

Drying columns
D₁/D₂: 360 m³/h,
air dew point
reduced to $-70 \text{ }^\circ\text{C}$

BX nitrogen

Requirements

Purification

LAKN development

Synthetic air

JUNO nitrogen

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^{222}Rn -free clean rooms

The system will be used to provide Rn-reduced air to the DARKSIDE-20k cryostat operated as a clean room during construction of the detector.

BX nitrogen

Requirements

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Rn diff/emanation

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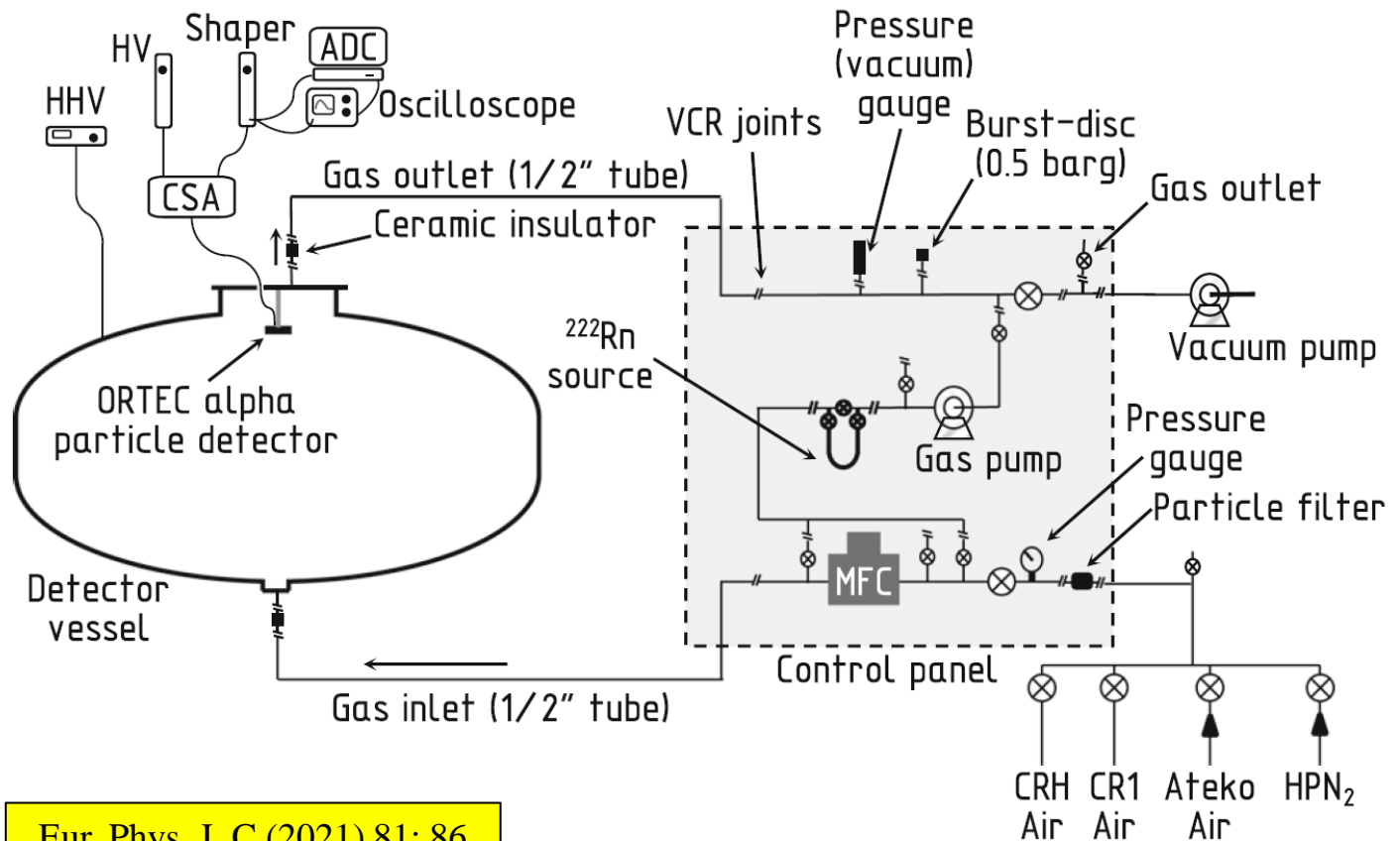




^{222}Rn -free clean rooms

Electrostatic radon detector

- For real-time monitoring of ^{222}Rn in DS cleanrooms
- Design based on electrostatic collection of the charged ^{222}Rn daughters ($^{218}\text{Po}/^{214}\text{Po}$)
- Low-background alpha detector (ORTEC) used to register decays of the ^{222}Rn daughters
- Sensitivity goal: $\sim 0.5 \text{ mBq/m}^3$
- **Sensitivity achieved: $\sim 0.05 \text{ mBq/m}^3$**



Eur. Phys. J. C (2021) 81: 86

BX nitrogen

Requirements

Purification

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Synthetic air

JUNO nitrogen

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Ar and Kr removal

Rn-free clean room

Rn diff/emanation

Conclusions



^{222}Rn -free clean rooms

Radon concentration in different air samples measured with the electrostatic detector. Measurements performed with high purity nitrogen reflect the background of the detector (self-emanation).

Sample	Description	^{222}Rn content [mBq/m^3]
Ateko air	Taken directly from the abatement system	15 ± 2
		1.4 ± 0.4
		11 ± 1
CRH	Various periods between 2013 and 2017	21 ± 3
		12 ± 2
		5.8 ± 0.9
		36 ± 6
		4.6 ± 0.6
CR1	Between 2013 and 2017	208 ± 21
		5000 ± 350
		2200 ± 180
HPN ₂	$^{222}\text{Rn} < 1 \mu\text{Bq}/\text{m}^3$	0.5 ± 0.1
		0.7 ± 0.2
		0.6 ± 0.2

Radon in hall C air: $\sim 50 \text{ Bq}/\text{m}^3$



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BX nitrogen

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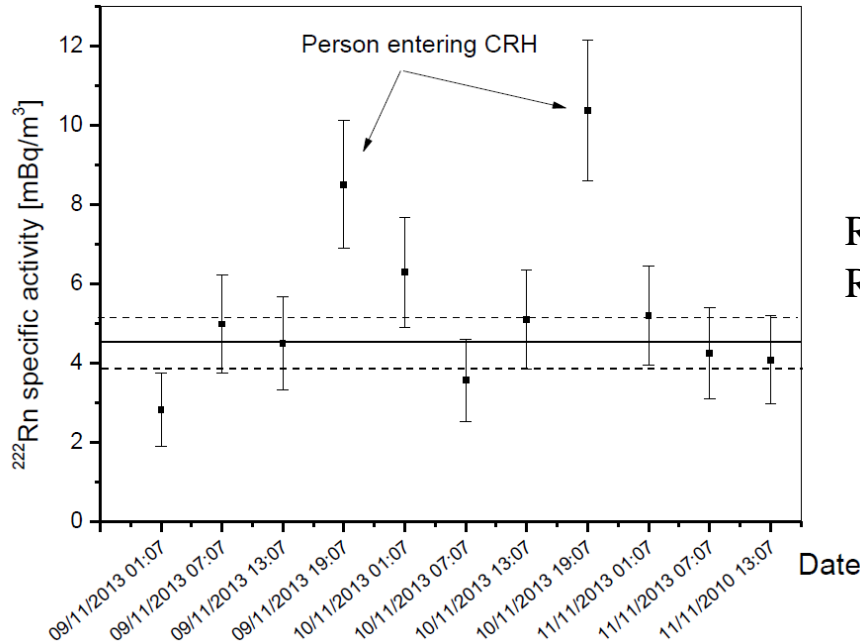
Rn diff/emanation

Conclusions

^{222}Rn -free clean rooms



Clean Room Hanoi (CRH)
built to minimize the ^{222}Rn
emanation: completely
stainless steel lined



Radon in hall C air: $\sim 50 \text{ Bq/m}^3$
Radon in CRH air: $5 - 50 \text{ mBq/m}^3$



^{222}Rn diffusion and emanation

- Information about the middle part of the ^{238}U chain
- Non-destructive, time consuming
- High sensitivity: $\sim 20 \mu\text{Bq}$ ($^{222}\text{Rn}/^{226}\text{Ra}$)
- Limited number of high sensitivity systems: proportional counters, cryogenic detectors, electrostatic detectors, Lucas cells

Relevant if one wants to know:

- Amount of emanated ^{222}Rn (detector active part, infrastructure, piping, storage vessels, ...)
- Surface cleanliness
- For some materials bulk ^{226}Ra may be determined (diffusion constant needs to be known)

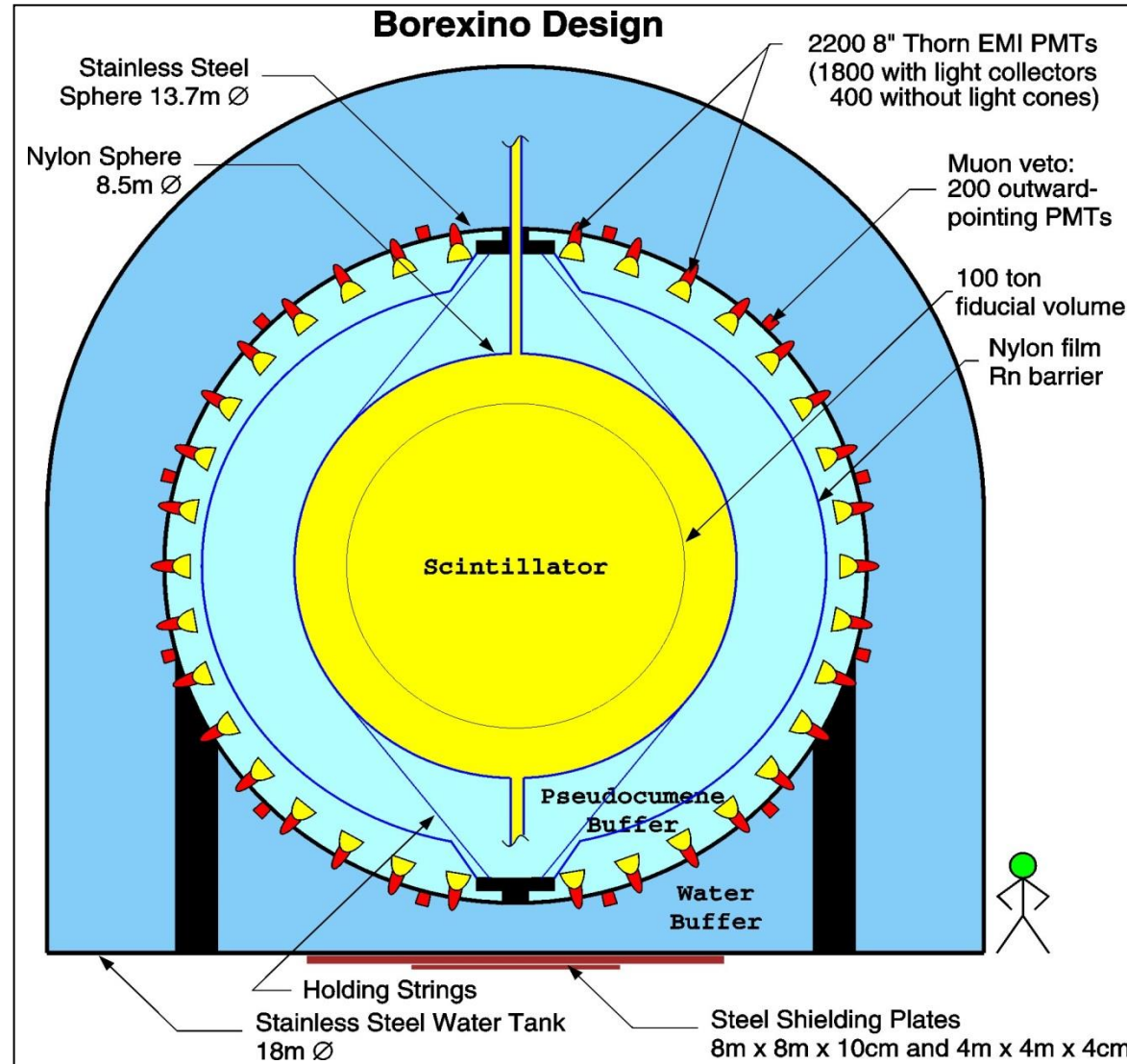
See review talks from LRT 2024



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^{222}Rn diffusion and emanation

Good ^{222}Rn barriers are required in many experiments (BOREXINO)



BX nitrogen

Requirements

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LAKN development

Synthetic air

JUNO nitrogen

^{222}Rn removal

Ar and Kr removal

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^{222}Rn diffusion and emanation

BX nitrogen

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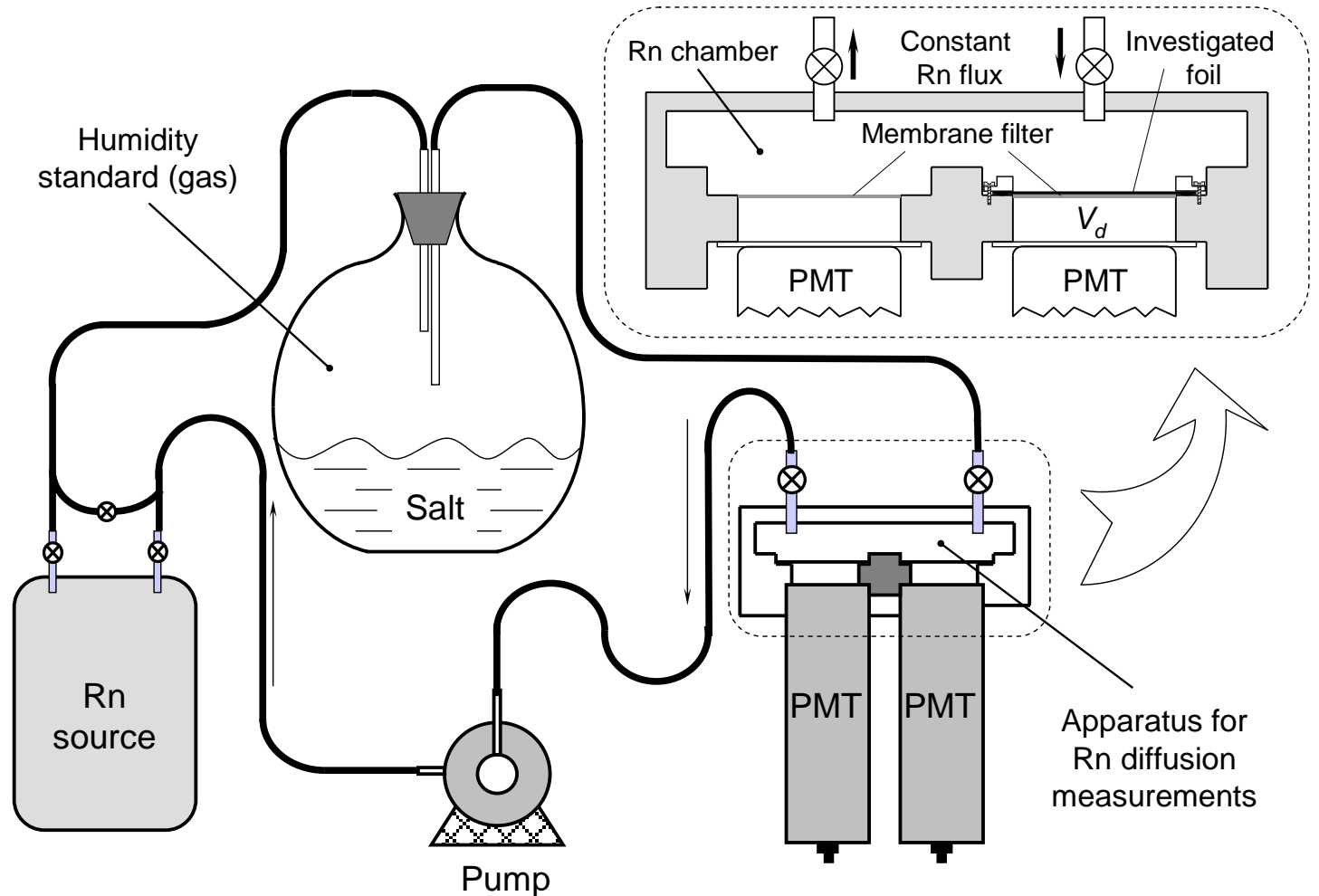
^{222}Rn removal

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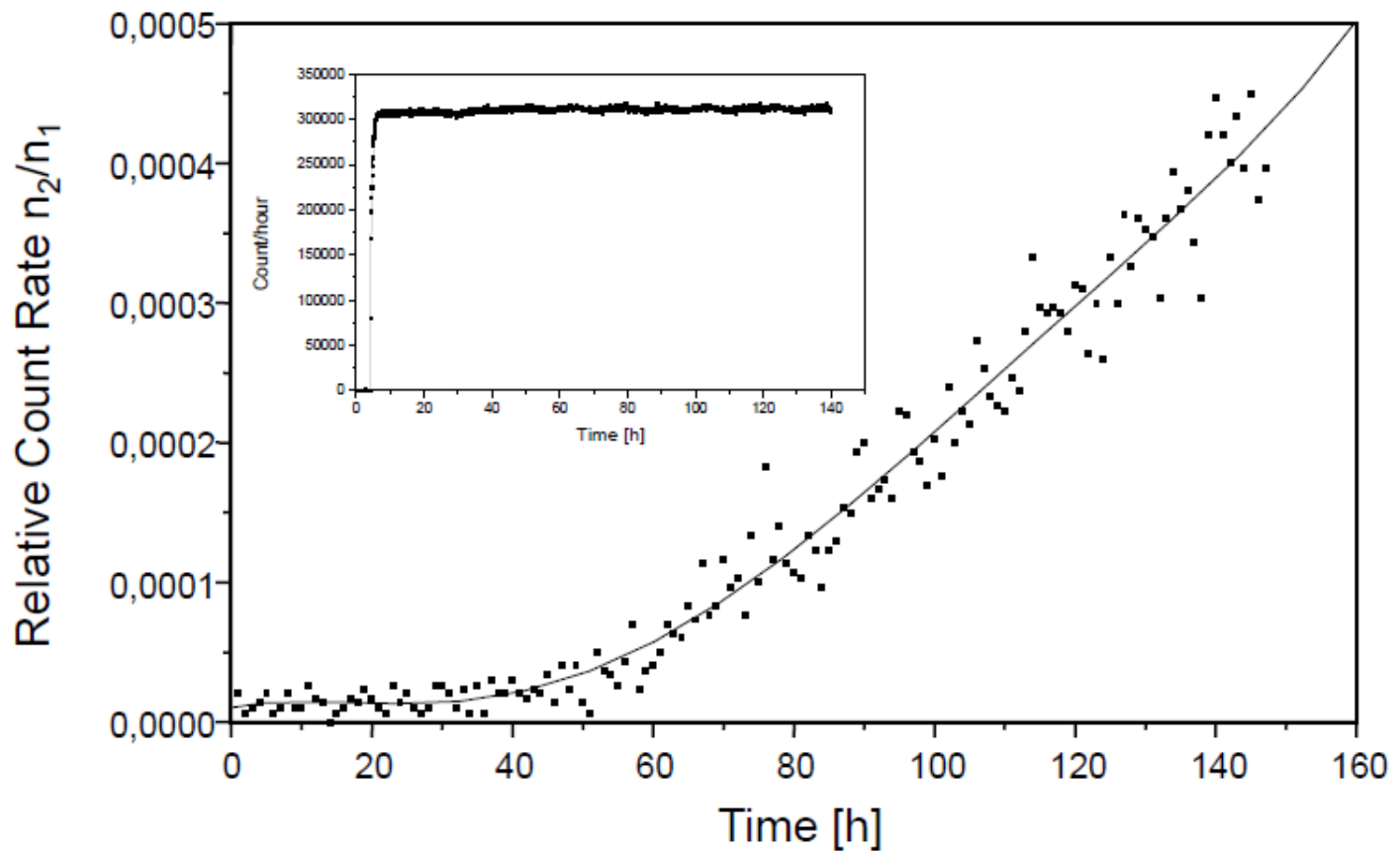
NIM A 498 (2003) 240



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^{222}Rn diffusion and emanation

Time dependence of the relative count rate obtained for a dry nylon membrane. Solid line represents the least-square fit to the experimental data. Constant Rn activity in the chamber is shown in as an inset the upper-left corner.



BX nitrogen

Requirements

Purification

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^{222}Rn diffusion and emanation

Results obtained for the 0.018 mm thick C38F film (BOREXINO)

RH standard salt	RH in gas phase (%)	Water amount in nylon, M (%)	Diffusion coefficient, D (cm^2/s)	Solubility, S
$\text{Mg}(\text{ClO}_4)_2$	~ 0	~ 0	$(2.1 \pm 0.4) \times 10^{-12}$	4.5 ± 0.7
$\text{H}_3\text{PO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$	9 ± 1	0.72 ± 0.04	$(2.3 \pm 0.3) \times 10^{-12}$	2.5 ± 0.3
$\text{LiCl}_2 \cdot \text{H}_2\text{O}$	12 ± 1	0.87 ± 0.04	$(2.2 \pm 0.3) \times 10^{-12}$	2.2 ± 0.3
$\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$	32 ± 2	2.09 ± 0.04	$(4.3 \pm 0.5) \times 10^{-12}$	1.8 ± 0.2
$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$	52 ± 2	3.74 ± 0.05	$(1.9 \pm 0.3) \times 10^{-11}$	1.4 ± 0.2
$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$	76 ± 2	6.35 ± 0.05	$(6.5 \pm 0.9) \times 10^{-11}$	1.5 ± 0.2
K_2CrO_4	88 ± 3	7.60 ± 0.05	$(1.3 \pm 0.2) \times 10^{-10}$	1.5 ± 0.2
$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$	93 ± 3	9.12 ± 0.07	$(3.3 \pm 0.4) \times 10^{-10}$	1.0 ± 0.1
H_2O vapors	100 ± 3	10.14 ± 0.09	$(1.3 \pm 0.2) \times 10^{-9}$	0.7 ± 0.1

BX nitrogen

Requirements

Purification

LAKN development

Synthetic air

JUNO nitrogen

^{222}Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

Conclusions

There is 3 orders of magnitude difference between the diffusion in the dry and in the foil saturated with water!

The BOREXINO barriers reduce ^{222}Rn concentrations by several orders of magnitude

$$d_e = \sqrt{\frac{D}{\lambda}} \quad \begin{array}{l} d_e^d = 7 \mu\text{m} \\ d_e^w = 270 \mu\text{m} \end{array}$$

NIM A 524 (2004) 355



^{222}Rn diffusion and emanation

- ^{226}Ra content in the foil used for the construction of the BOREXINO Inner Vessel should not exceed 1 ppt-U equivalent ($12 \mu\text{Bq/kg}$).
- Because of the low density of the nylon (1.135 g/cm^3) Ge spectrometers are not able to reach that sensitivity.
- Other techniques, like ICP-MS, determine only long-living parent isotope (^{238}U), which is not necessarily in equilibrium with ^{226}Ra .
- New measurement idea was based on the radon emanation under different conditions (humidity). It allowed in addition to distinguish between the surface and bulk ^{226}Ra contaminations.

BX nitrogen

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^{222}Rn diffusion and emanation

BX nitrogen

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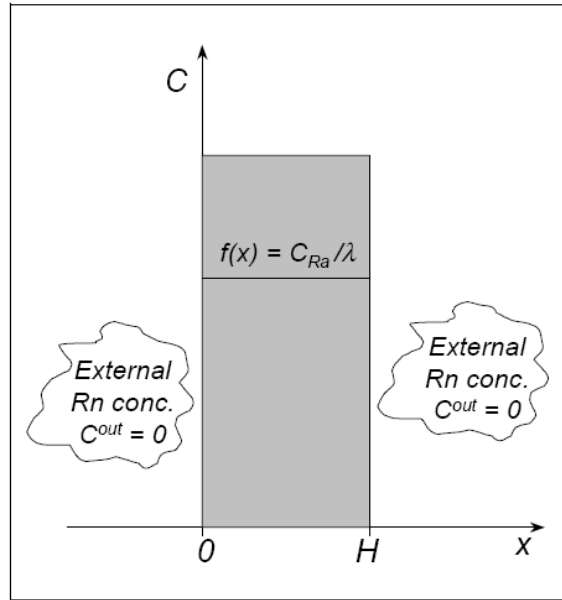
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Conclusions



$$\frac{\partial C(x,t)}{\partial t} = D \frac{\partial^2 C(x,t)}{\partial x^2} - \lambda C(x,t) + P$$

$$\begin{cases} C(x,t) = 0 & x = 0 & t \geq 0 \\ C(x,t) = 0 & x = H & t \geq 0 \\ C(x,t) = f(x) & 0 < x < H & t = 0 \end{cases}$$

$$c_1(x,t) = \frac{C_{Ra}}{\lambda} \left[1 - \frac{\sinh[dx] + \sinh[d(H-x)]}{\sinh[dH]} \right] + \frac{4C_{Ra}}{\lambda\pi} \sum_{n=0}^{\infty} \frac{\alpha e^{-\beta t}}{(2n+1)\beta} \sin \frac{(2n+1)n\pi}{H}$$

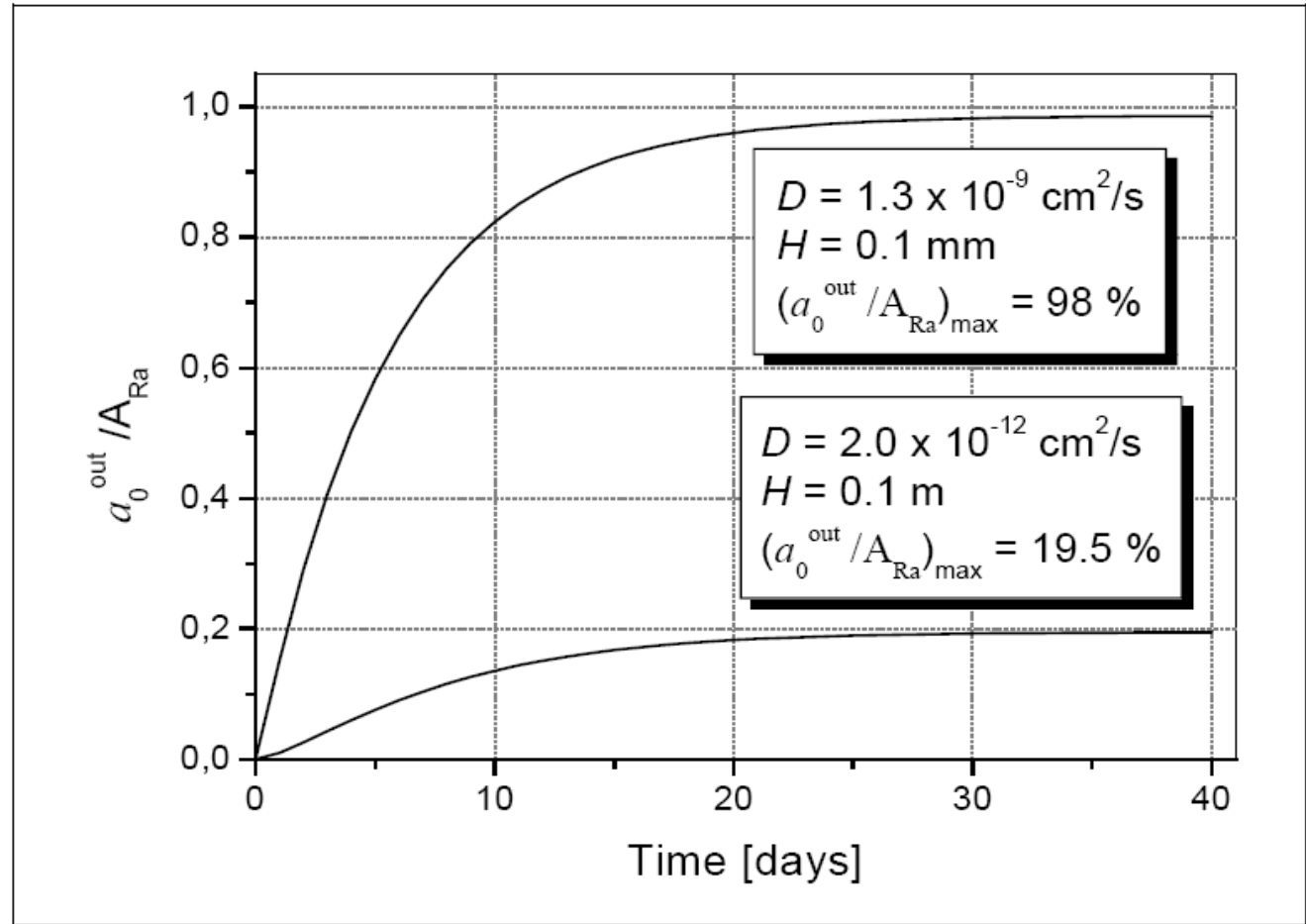
$$a_1^{\text{in}}(t) = \lambda F \int_0^H c_1(x,t) dx = FHC_{Ra} \left[1 - \frac{2(\cosh[dH] - 1)}{dH \cdot \sinh[dH]} \right] + \frac{8FHC_{Ra}}{\pi^2} \sum_{n=0}^{\infty} \frac{\alpha e^{-\beta t}}{(2n+1)^2 \beta}$$

$$\beta = \alpha + \lambda = \frac{D(2n+1)^2 \pi^2}{H^2} + \lambda$$

$$d = \sqrt{\lambda/D}$$



^{222}Rn diffusion and emanation



$$\begin{cases} \mathcal{A}^{\text{dry}}(t_{\text{dry}}) = a_{\text{Rn}}^{\text{sf}}(t_{\text{dry}}) + a^{\text{out}}(D_{\text{dry}}, t_{\text{dry}}) \\ \mathcal{A}^{\text{wet}}(t_{\text{wet}}) = a_{\text{Rn}}^{\text{sf}}(t_{\text{wet}}) + a^{\text{out}}(D_{\text{wet}}, t_{\text{wet}}) \end{cases} \quad a_{\text{Rn}}^{\text{sf}}(t) = a_{\text{Ra}}(1 - e^{-\lambda t})$$

BX nitrogen

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$M \approx 10 \text{ kg}$
 $S_f \approx 150 \text{ m}^2$

$C_B \sim 10 \mu\text{Bq/kg}$

$C_F \sim 0.5 \mu\text{Bq/m}^2$



^{222}Rn diffusion and emanation

Direct ^{226}Ra measurements

No	Nylon type, thickness, mass, surface area	Bulk ^{226}Ra [mBq/kg] [ppt]	Surface ^{226}Ra [μBq/m ²]	Total ^{226}Ra [mBq/kg] [ppt]
1	Capron CF400 0.1 mm, 9.8 kg, 171 m ²	0.58 ± 0.08 47 ± 6	—	0.58 ± 0.08 47 ± 6
2	Capron/Sniamid* 0.1 mm, 9.5 kg, 167.5 m ²	0.10 ± 0.02 8 ± 2	3.0 ± 1.0	0.14 ± 0.03 11 ± 2
3	Capron B73ZP (1st batch) 0.1 mm, 11.8 kg, 208 m ²	0.21 ± 0.04 17 ± 3	1.1 ± 0.7	0.22 ± 0.05 18 ± 4
4	Capron B73ZP (2nd batch) 0.125 mm, 9.5 kg, 134 m ²	0.46 ± 0.07 37 ± 6	6.4 ± 2.2	0.55 ± 0.08 45 ± 7
5	Sniamid (blend) 0.125 mm, 9.2 kg, 130 m ²	< 0.02 < 1.6	< 0.8	0.016 ± 0.004 1.3 ± 0.4

ICP-MS on U/Th

Material brand name	sample form	^{238}U (ppt)	^{232}Th (ppt)
Sniamid ADS40T	pellets	1.1 ± 0.0	1.6 ± 0.1
Sniamid ADS40T	film (Leistriz)	2.8 ± 0.1	3.8 ± 0.2
Capron B73ZP	pellets	0.46 ± 0.04	1.1 ± 0.1
Selar PA3426	pellets	0.22 ± 0.02	0.65 ± 0.17
C90 (90% Capron, 10% Selar)	film (Leistriz)	1.6 ± 0.2	2.9 ± 0.1
C38F	film (Mobay)	1.7 ± 0.2	3.9 ± 0.5

BX nitrogen

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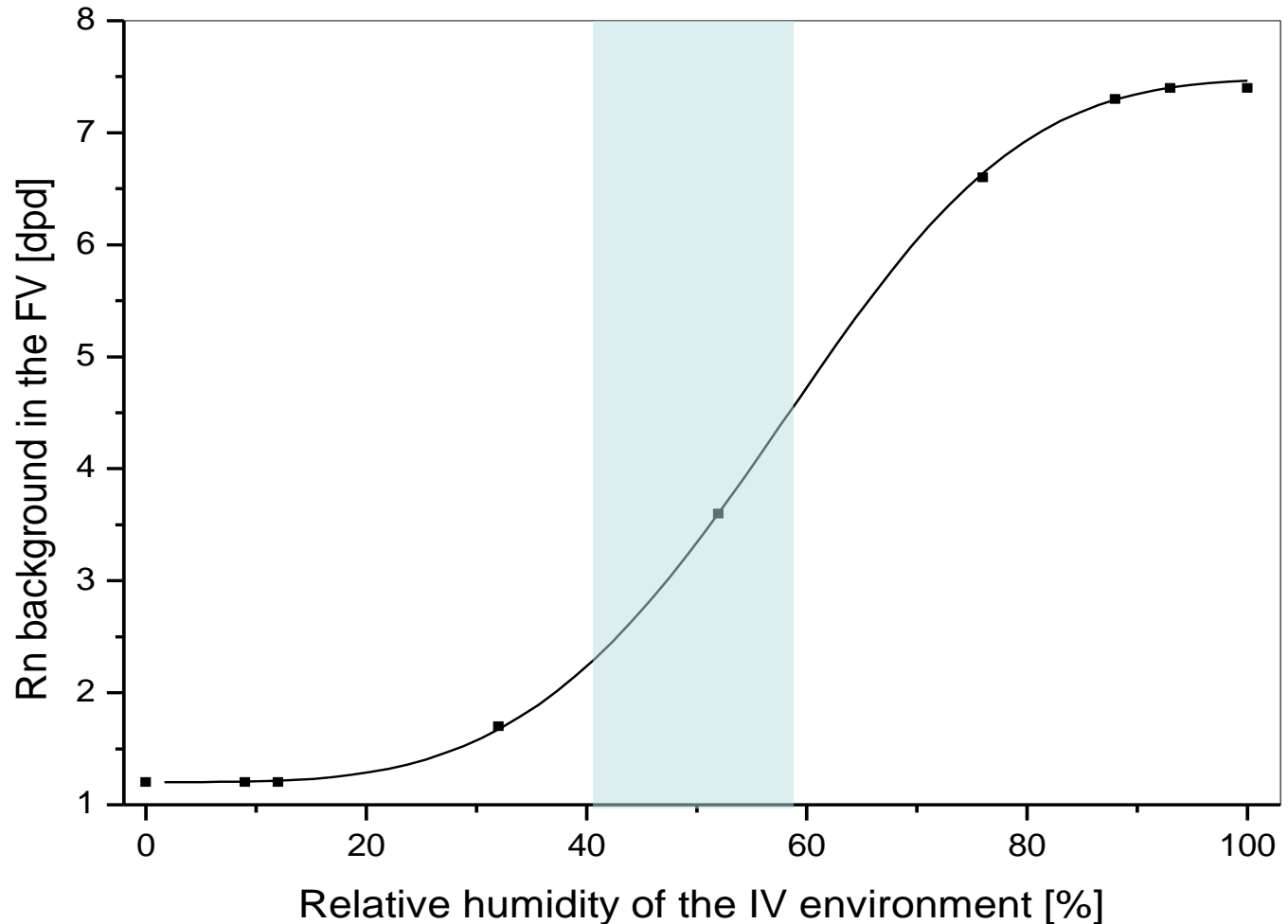
Conclusions



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^{222}Rn diffusion and emanation

Expected ^{222}Rn signal in the FV as a function of the water content in the scintillator / buffer.



BX nitrogen

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^{222}Rn diffusion and emanation

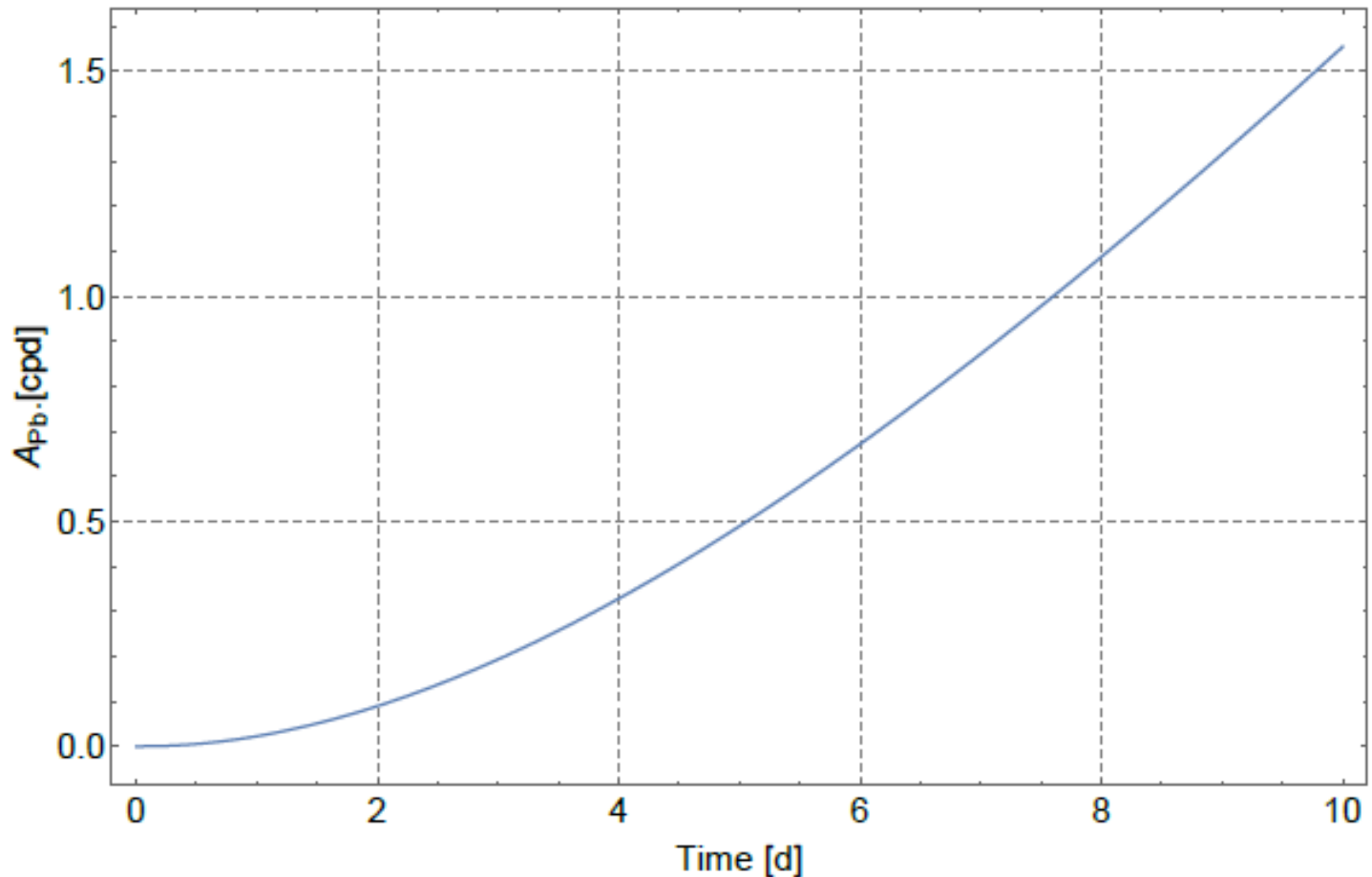
Subsystem	Sample	Description	A_{Rn}
BOREXINO PC storage area	SS vessel TK1	$\sim 114 \text{ m}^3, \sim 140 \text{ m}^2$	$< 60 \text{ mBq}$
	SS vessel TK2	$\sim 114 \text{ m}^3, \sim 140 \text{ m}^2$	$(45 \pm 8) \text{ mBq}$
	SS vessel TK4	$\sim 114 \text{ m}^3, \sim 140 \text{ m}^2$	$(24 \pm 5) \text{ mBq}$
GERDA cryostat	SS vessel, $\sim 65 \text{ m}^3$	after construction/first cleaning	$(23.3 \pm 3.6) \text{ mBq}$
		after additional cleaning	$(13.7 \pm 1.9) \text{ mBq}$
		after copper mounting	$(34.4 \pm 6.0) \text{ mBq}$
		after wiping of inner surfaces in final configuration	$(30.6 \pm 2.4) \text{ mBq}$ $(54.7 \pm 3.5) \text{ mBq}$
LN ₂ /LAr storage and transport tanks	LAr transport tank, 0.67 m^3	for 6.0 quality only	$(42 \pm 2) \text{ mBq}$
	LAr storage tank, 3 m^3	for 5.0 quality only	$(177 \pm 6) \text{ mBq}$
	LAr storage tank, 3 m^3	for 5.0 quality only*	$\sim 38 \text{ mBq}$
	LN ₂ storage tank, 3 m^3	for 7.0 quality only	$(2.7 \pm 0.3) \text{ mBq}$
	LN ₂ storage tank, 16 m^3	for 6.0 quality only	$(65 \pm 6) \text{ mBq}$
BOREXINO N ₂ distribution line	Electrical heater		$(0.92 \pm 0.29) \text{ mBq}$
	Particle filter	Code: AB1F0023EH11	$(0.34 \pm 0.13) \text{ mBq}$
	1.5" distribution line	$\sim 100 \text{ m}$ long	$(0.47 \pm 0.13) \text{ mBq}$
BOREXINO Scintillator purification plant	SS package for H ₂ O extraction column	25 m^2 surface	$< 0.12 \text{ mBq}$
	H ₂ O extraction column with 24 SS packages	$0.6 \text{ m}^3, 608 \text{ m}^2$	$(4.83 \pm 0.70) \text{ mBq}$
	N ₂ sparging column with 26 SS packages	$0.2 \text{ m}^3, 280 \text{ m}^2$	$(1.78 \pm 0.21) \text{ mBq}$
	HT2 Tank	2.1 m^3	$(1.22 \pm 0.37) \text{ mBq}$
	D330 Tank	1.58 m^3	$(7.13 \pm 1.15) \text{ mBq}$
TIG welds	2.5 m	not treated	$(0.36 \pm 0.04) \text{ mBq/m}$
	0.8 m	etched and passivated	$< 0.1 \text{ mBq/m}$
	1.2 m	electropolished	$(0.10 \pm 0.04) \text{ mBq/m}$
	1.2 m	electropol./etched/passivated	$< 0.04 \text{ mBq/m}$
Inner Vessel	Bulk of sniamid nylon	$125 \mu\text{m}$, cleaned by <i>CleanFilm</i>	$< 21 \mu\text{Bq/kg}$
	Surface of sniamid nylon	$125 \mu\text{m}$, cleaned by <i>CleanFilm</i>	$< 0.8 \mu\text{Bq/m}^2$



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^{222}Rn diffusion and emanation

Expected ^{210}Pb activity in 100 ton of the BOREXINO scintillator as a function of the storage time in a tank with the ^{222}Rn emanation rate of 40 mBq. 1 cpd of ^{210}Pb is reached already after 7.5 d, thus this would be the maximal storage time



BX nitrogen

Requirements

Purification

LAKN development

Synthetic air

JUNO nitrogen

^{222}Rn removal

Ar and Kr removal

Rn-free clean room

Rn diff/emanation

Conclusions



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Conclusions

- Technologies to provide nitrogen (argon) of highest radiopurity have been established
- Procedure to deliver LAKN to BOREXINO has been established with close cooperation with the SOL company
- Comprehensive screening program carried out within BOREXINO, based on developed ultra sensitive counting techniques (e.g. γ -ray spectrometry, detection of single ^{222}Rn atoms, ...), allowed to reach unprecedented background level in the detector making the detection of CNO neutrinos possible

BX nitrogen

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