

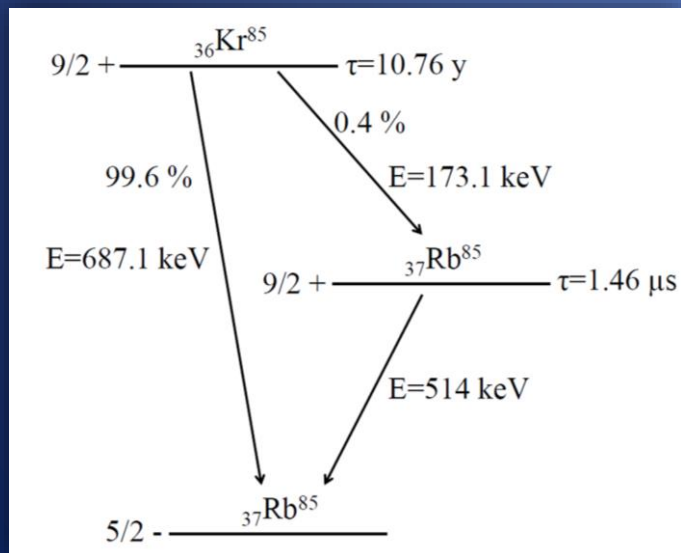
Krypton in Xenon

Determination of krypton concentration in cryogenic distilled xenon gas with a quadrupole mass spectrometer following a cold-trap at a temporarily reduced pumping speed

by Alexander Fieguth (WWU Münster)

Krypton is a radioactive background

- Krypton-85 decays via β -decay with an endpoint energy of 687 keV
- Trace amounts of ^{85}Kr are abundant in the atmosphere due to nuclear bomb tests and nuclear reprocessing

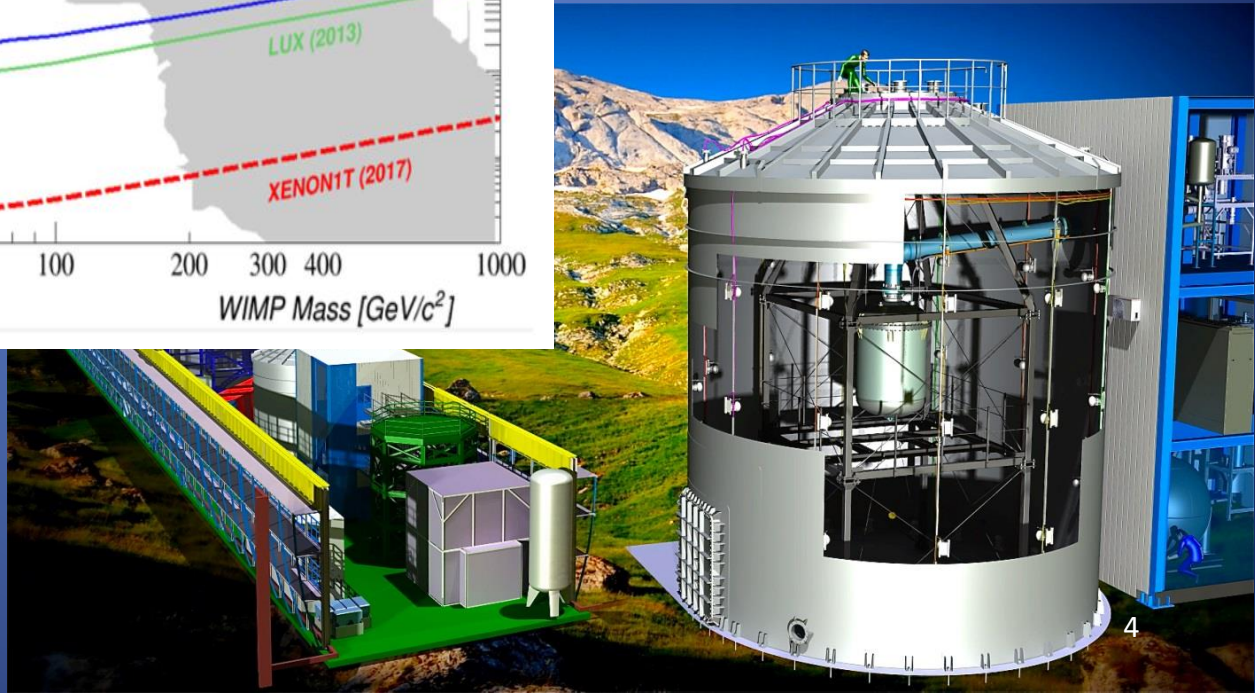
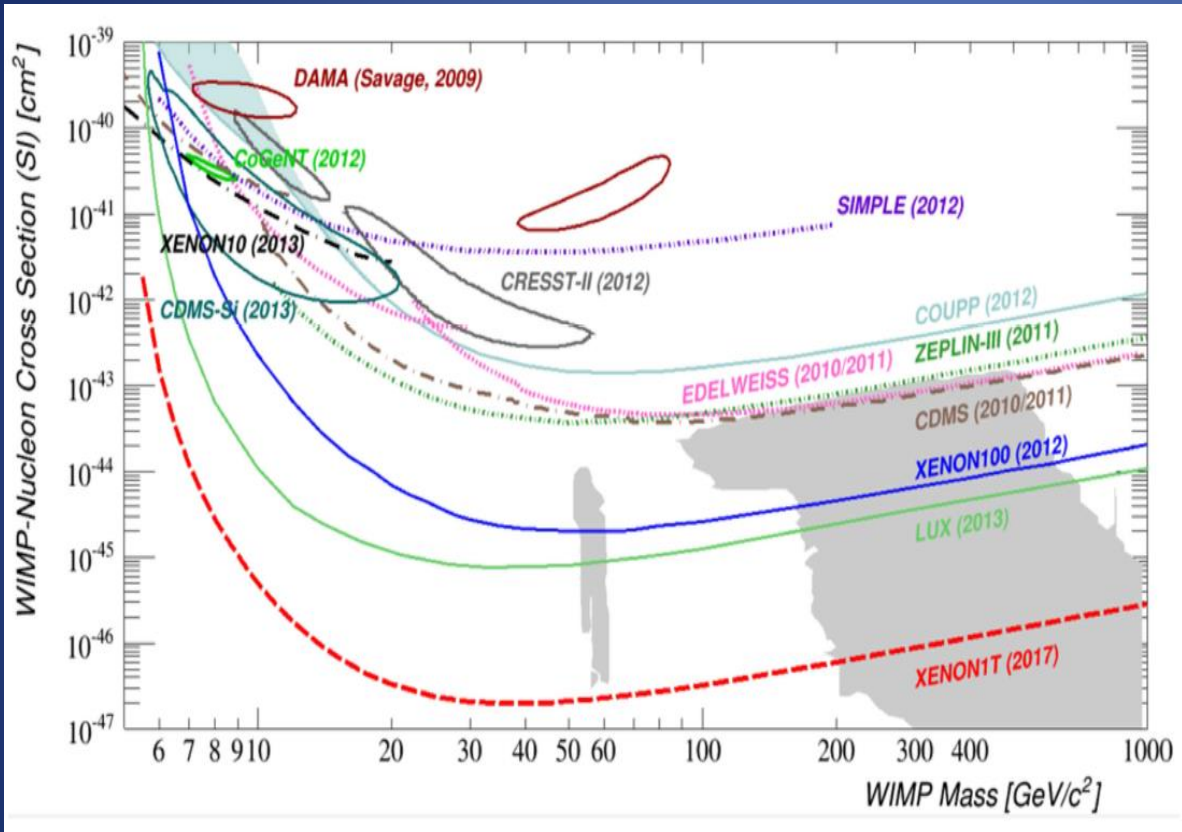


Xenon extraction
from atmosphere
leads to natural
krypton
contamination!

$$\frac{{}^{85}\text{Kr}}{\text{nat Kr}} \sim 10^{-11}$$

XENON Dark Matter Project

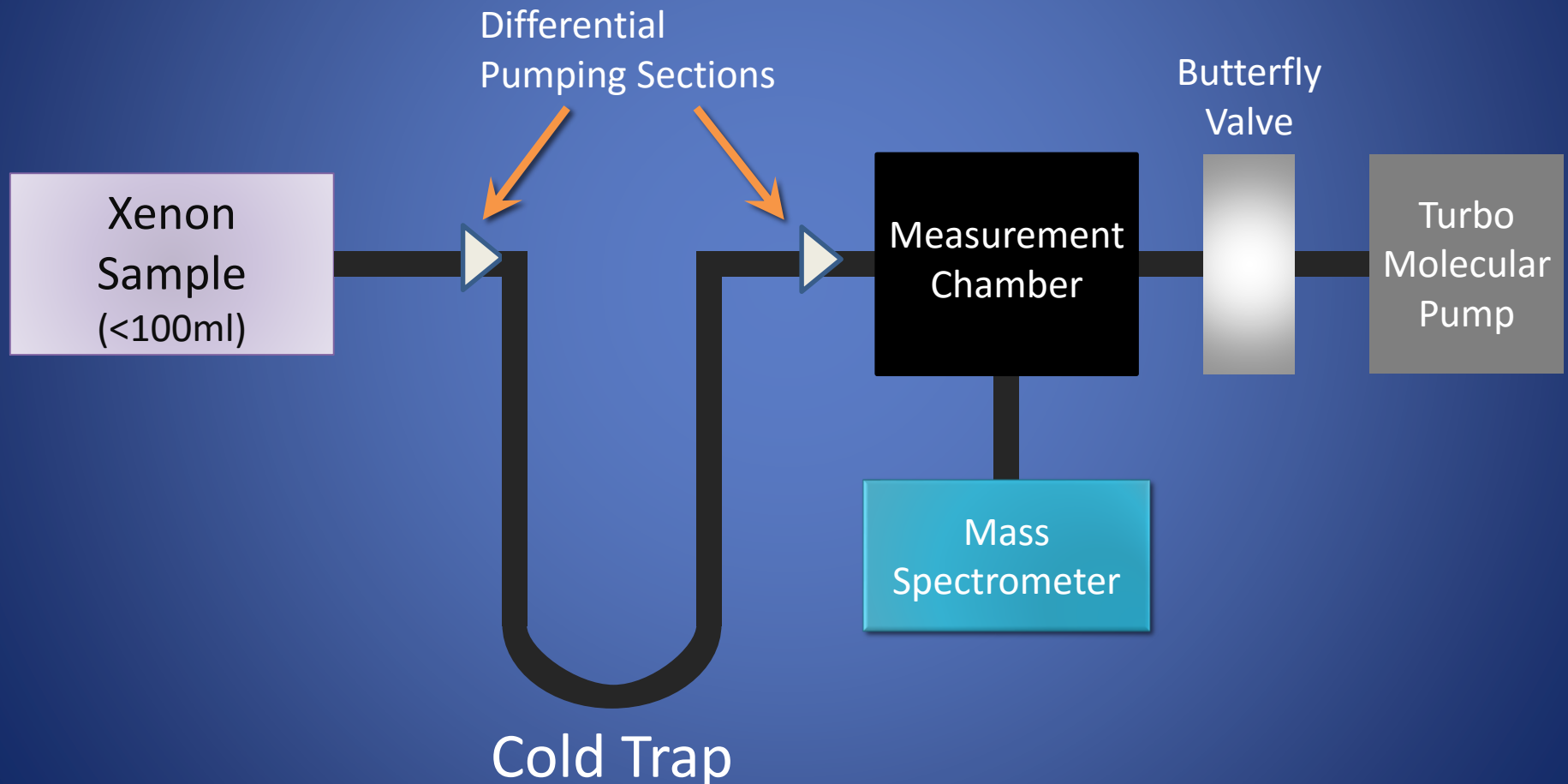
Xe
XENON
Dark Matter Project



XENON1T / XENONnT requirements

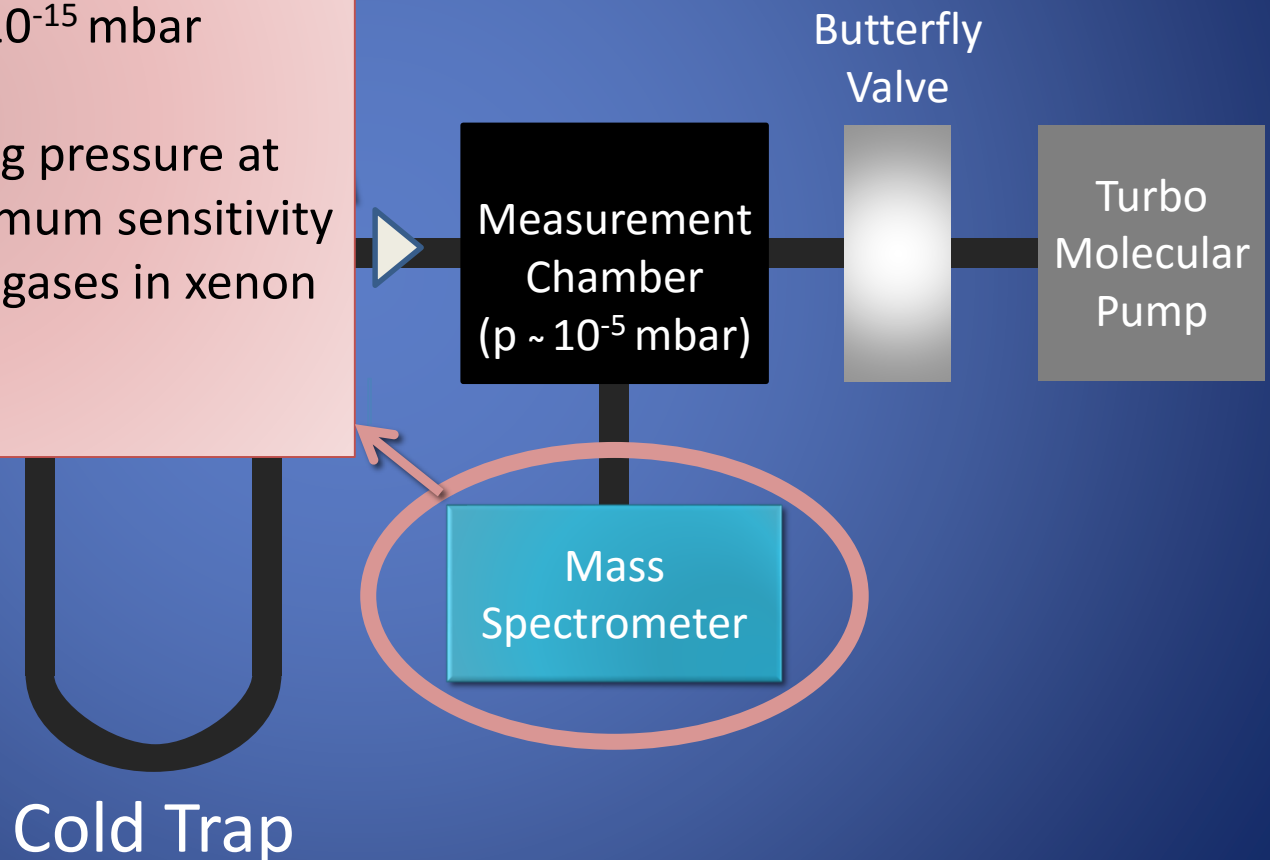
- Commercial xenon has a krypton content at the ppm to ppb level
- Removal of krypton is done by cryogenic distillation (see talk of M. Murra)
- Desired concentration is in the sub-ppt range
- Knowledge of the content is necessary for background event expectation due to krypton
- Measurement of the krypton concentration in this regime is not trivial

Measurement setup



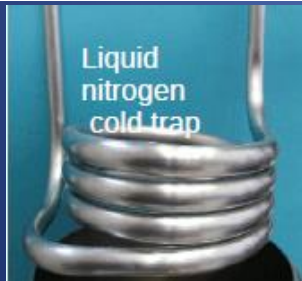
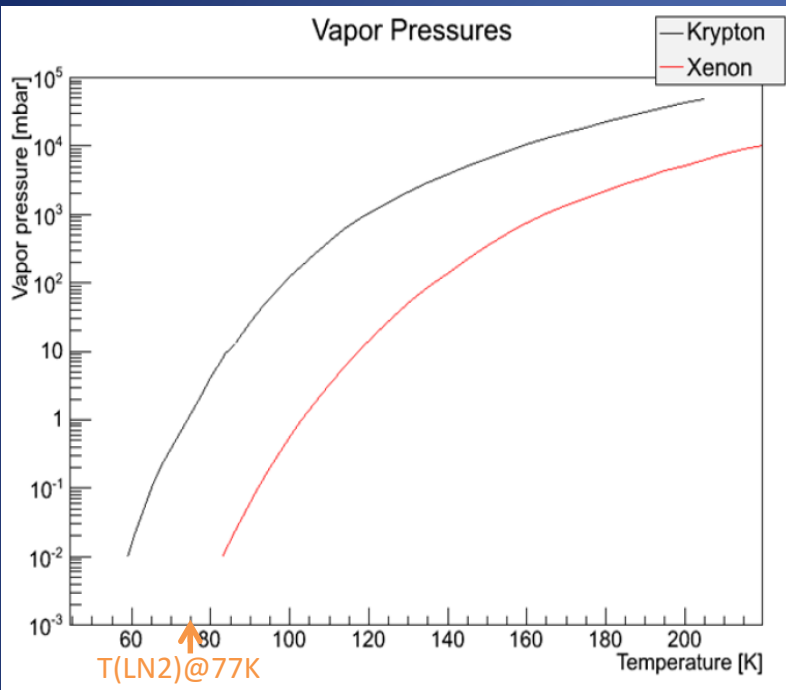
Mass spectrometer

- Using a commercial residual gas analyzer with a partial pressure sensitivity down to $\sim 10^{-15}$ mbar
- Limitation of operating pressure at 10^{-5} mbar sets a maximum sensitivity when detecting trace gases in xenon at ppb level



Cold trap

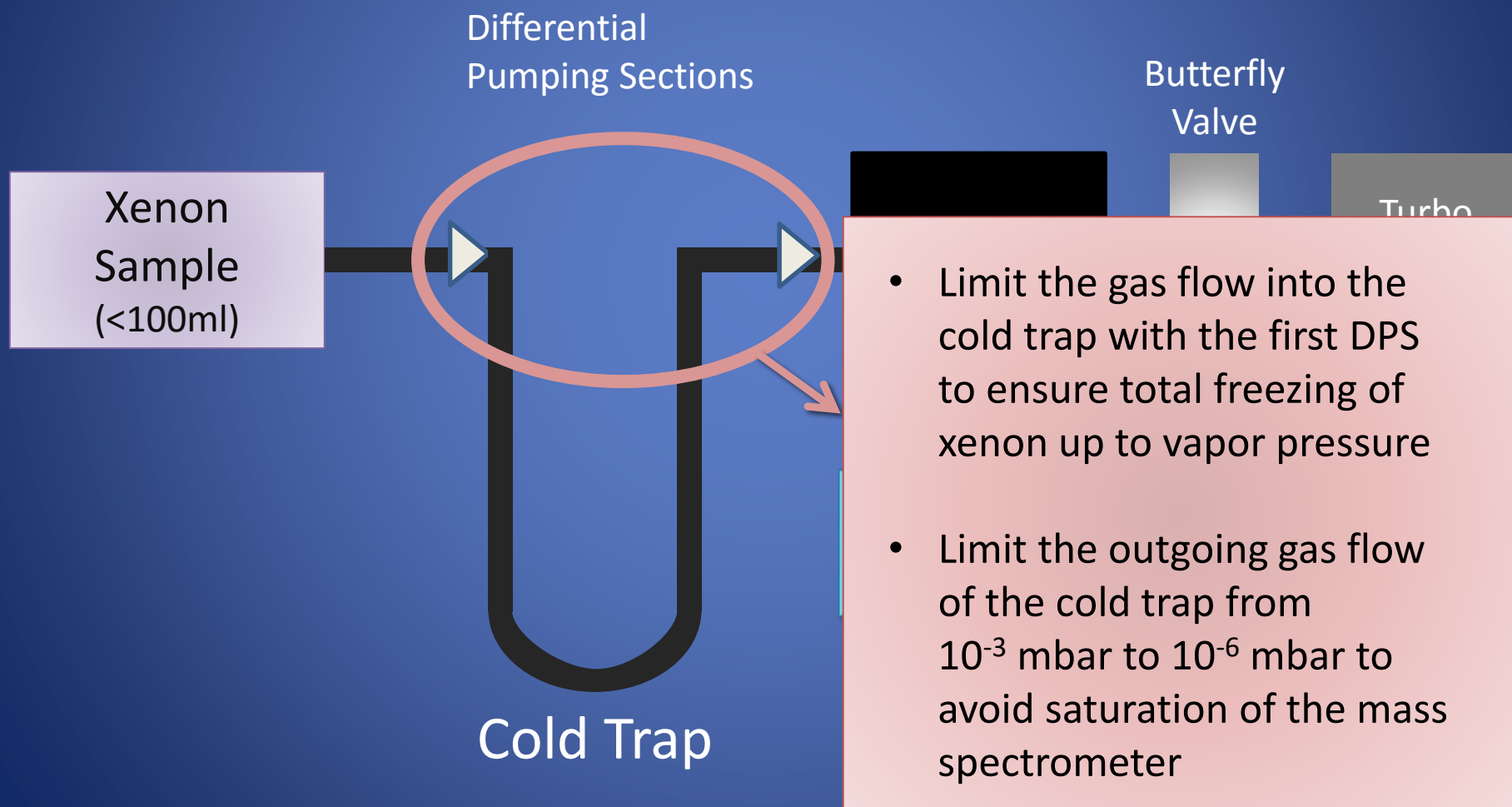
- Cool the stainless steel coil in a liquid nitrogen dewar down to 77K
- Xenon should freeze to the walls until the pressure reaches the vapor pressure of about 3×10^{-3} mbar at 77K
- Due to its low concentration krypton should pass unaffected and therefor the krypton concentration is enhanced



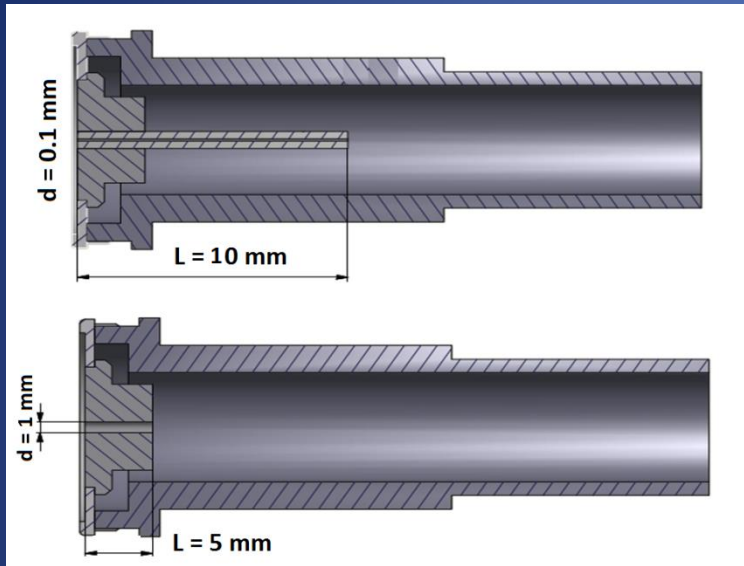
Mass
Spectrometer

Cold Trap

Differential pumping sections (DPS)



Differential pumping sections (DPS)

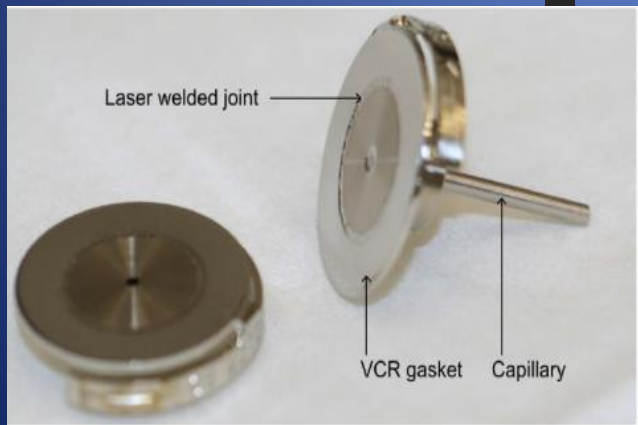


Differential
Sections

Butterfly
Valve

Turbo

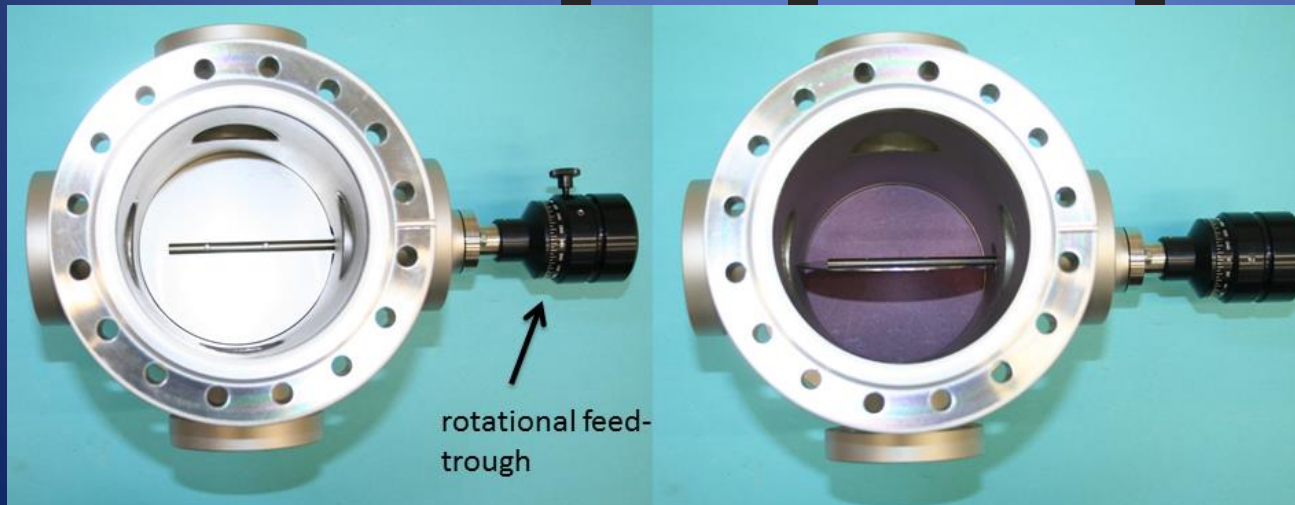
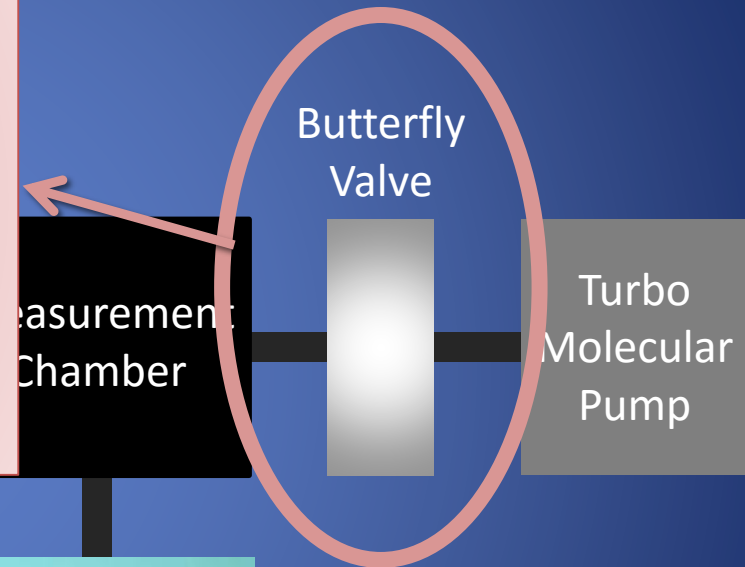
- Limit the gas flow into the cold trap with the first DPS to ensure total freezing of xenon up to vapor pressure
- Limit the outgoing gas flow of the cold trap from 10^{-3} mbar to 10^{-6} mbar to avoid saturation of the mass spectrometer



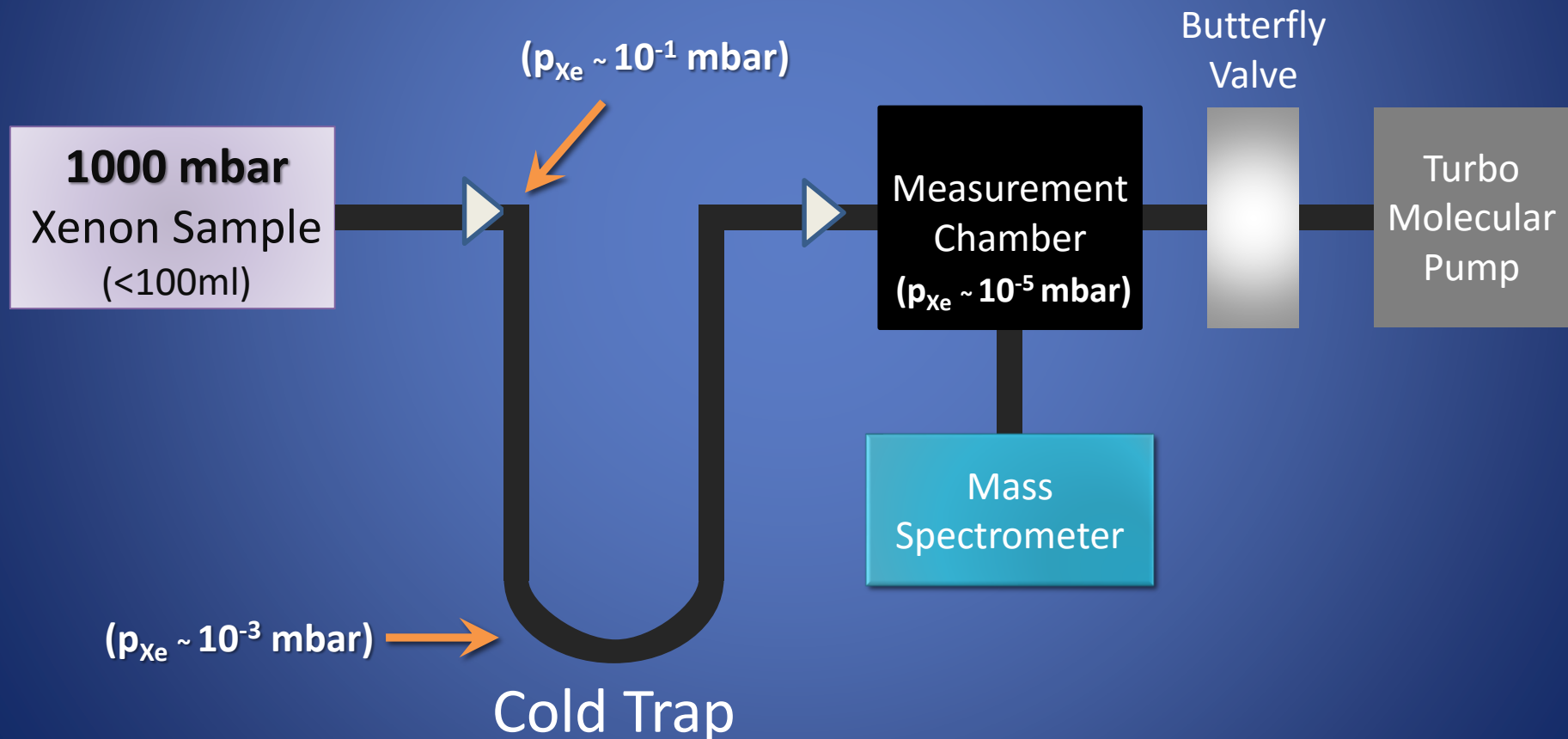
Cold Trap

Custom made butterfly valve

- Using a custom made butterfly valve allows to have a dynamic range of effective pumping speed (from 300 l/s down to 6 l/s) and therefore a regulation of gas load going to the mass spectrometer



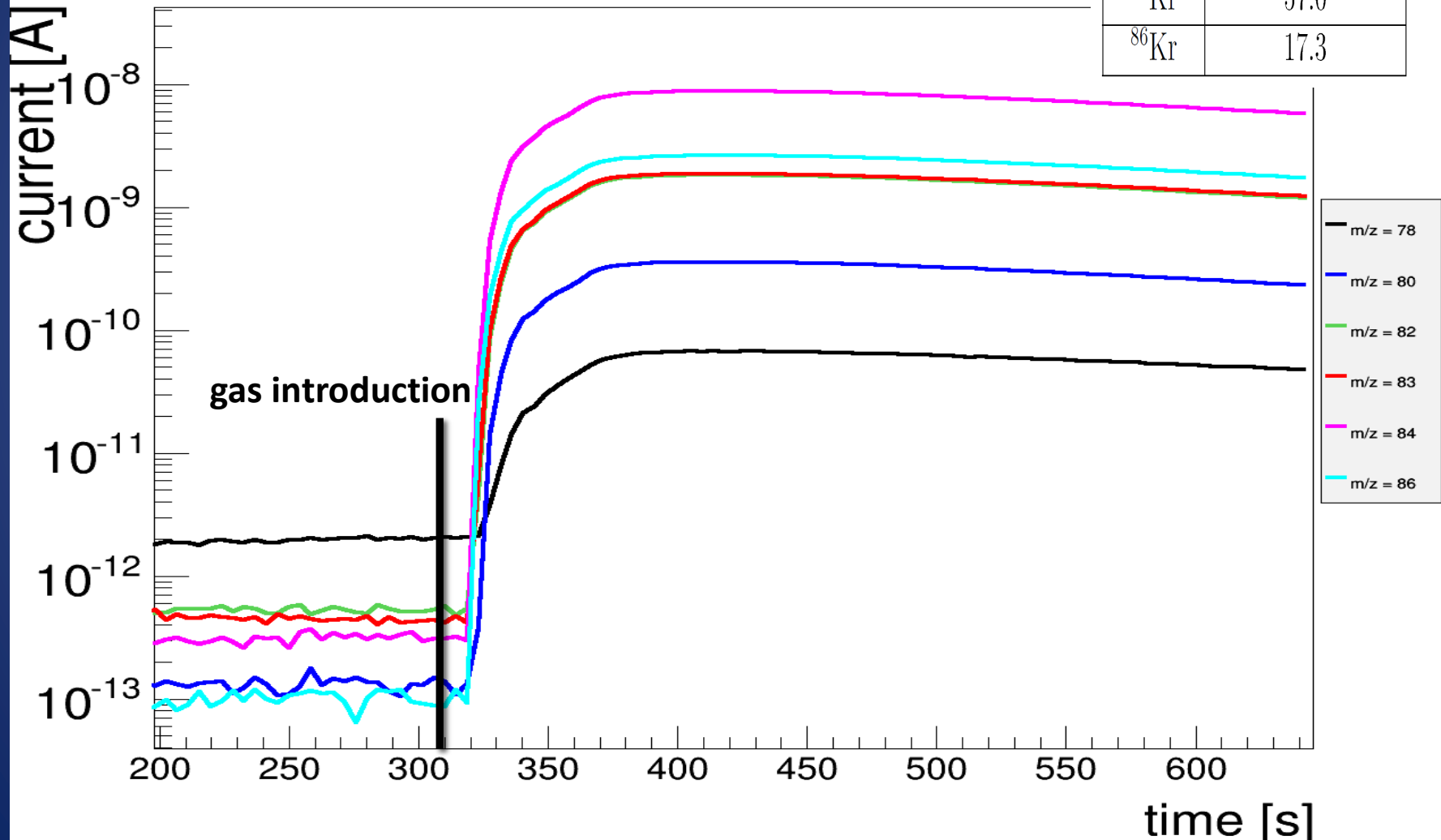
Measurement setup



Example signal

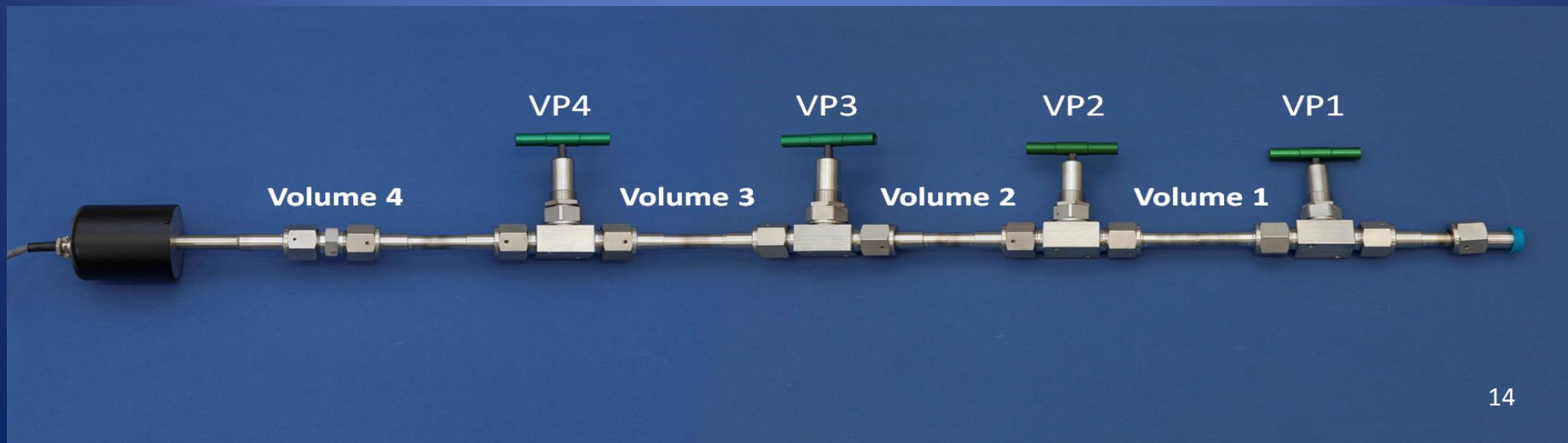
Isotope	Abundance in %
^{78}Kr	0.35
^{80}Kr	2.25
^{82}Kr	11.6
^{83}Kr	11.5
^{84}Kr	57.0
^{86}Kr	17.3

Kr isotopes RGA



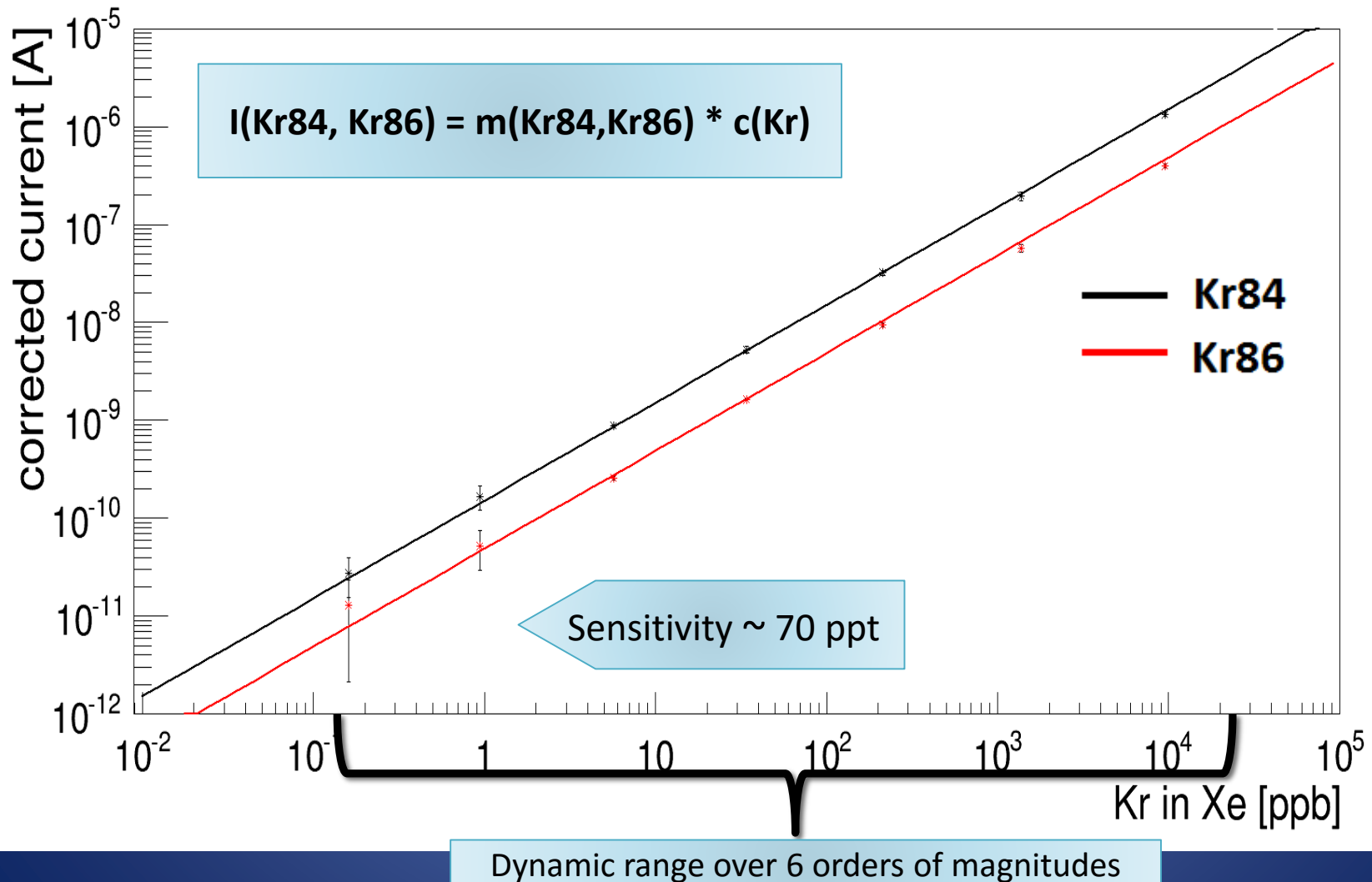
Calibration

- Artificially enhance the krypton concentration in xenon gas samples
- Using volume expansion for mixtures with known concentrations (“doping”)
- Advantage of a running distillation column allows for gas mixtures down to ppt



Calibration

Final calibration RGA2 via doping



Measuring krypton at the sub-ppt level?

- For measurements on the sub-ppt level other methods are available (ATTA, GC-RGMS, Low-level counting (LLC))
- These methods are offline-methods, as they post-analyze a taken sample off-site



Purpose of this system is
fast characterization and
online analysis

Applications

- Fast screening of gas bottles designated for the XENON1T experiment down to sub-ppb on-site (installed in 2015)
- Characterization and controlling of the distillation column online at processing



**Measured a separation factor
> 125 000 with artificially doped
gas (see talk M. Murra)**

Thank you for your attention

This work is supported by DFG.



Beyond the limit?!

- Increasing the amount of krypton relative to xenon in a reproducible way (change input flow -> increase DPS conductance)
- Decrease of the background or an better understanding of the background behavior
- Investigating the influence of other impurities on the sensitivity limit

Background ^{85}Kr

- Beta decay of ^{85}Kr into ^{85}Rb is a significant intrinsic background for XENON1T
- Determination of the ^{85}Kr concentration in xenon is of crucial importance for knowledge of the signal background
- Ratio of ^{85}Kr to natural krypton is at 10^{-11} , while natural krypton is aimed to be below <0.5 ppt in the used xenon

Background ^{85}Kr

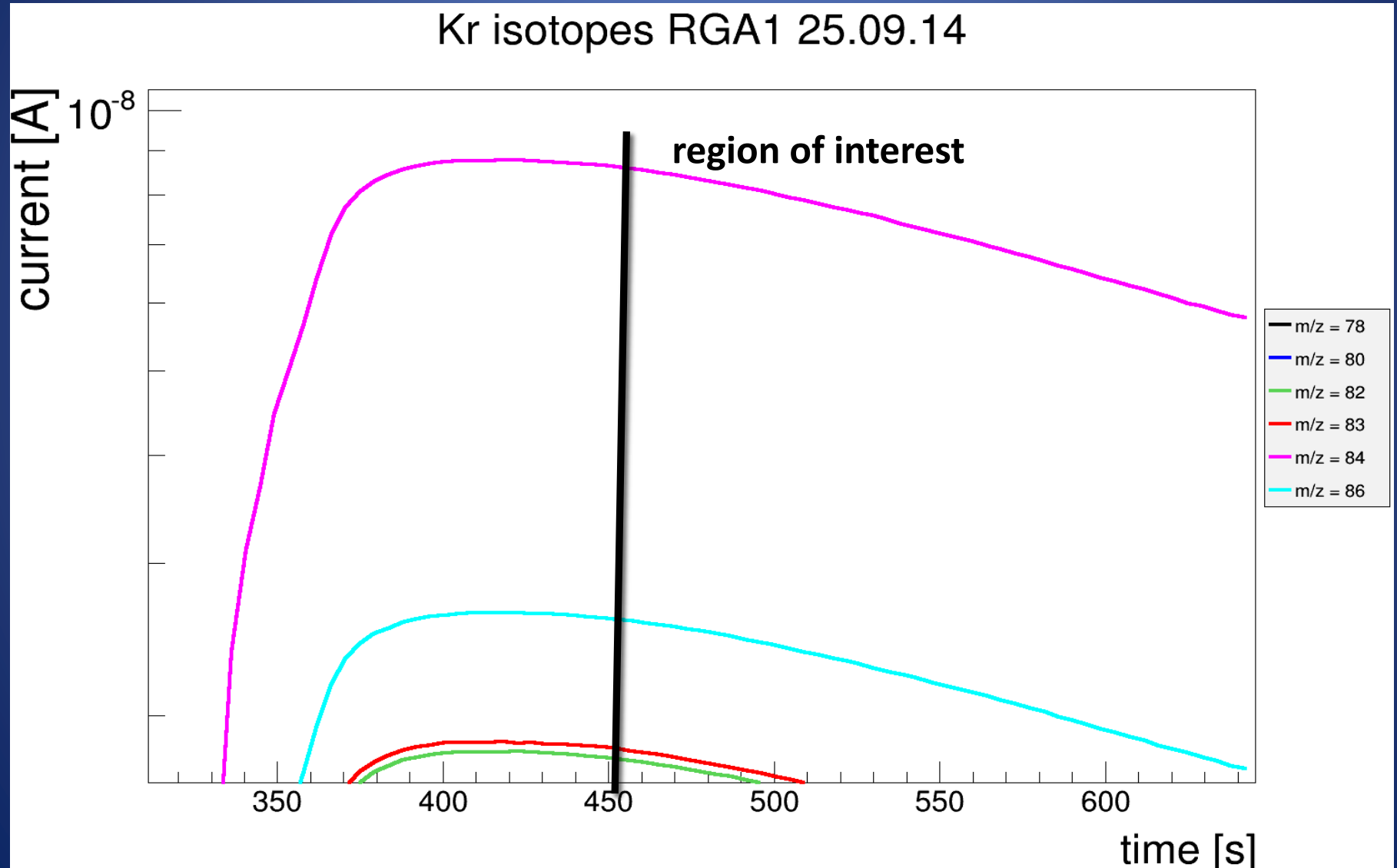
- Beta decay of ^{85}Kr into ^{85}Rb is a significant intrinsic background for Xenon-1T

Measuring the Krypton concentration at the sub-ppb level is not trivial!

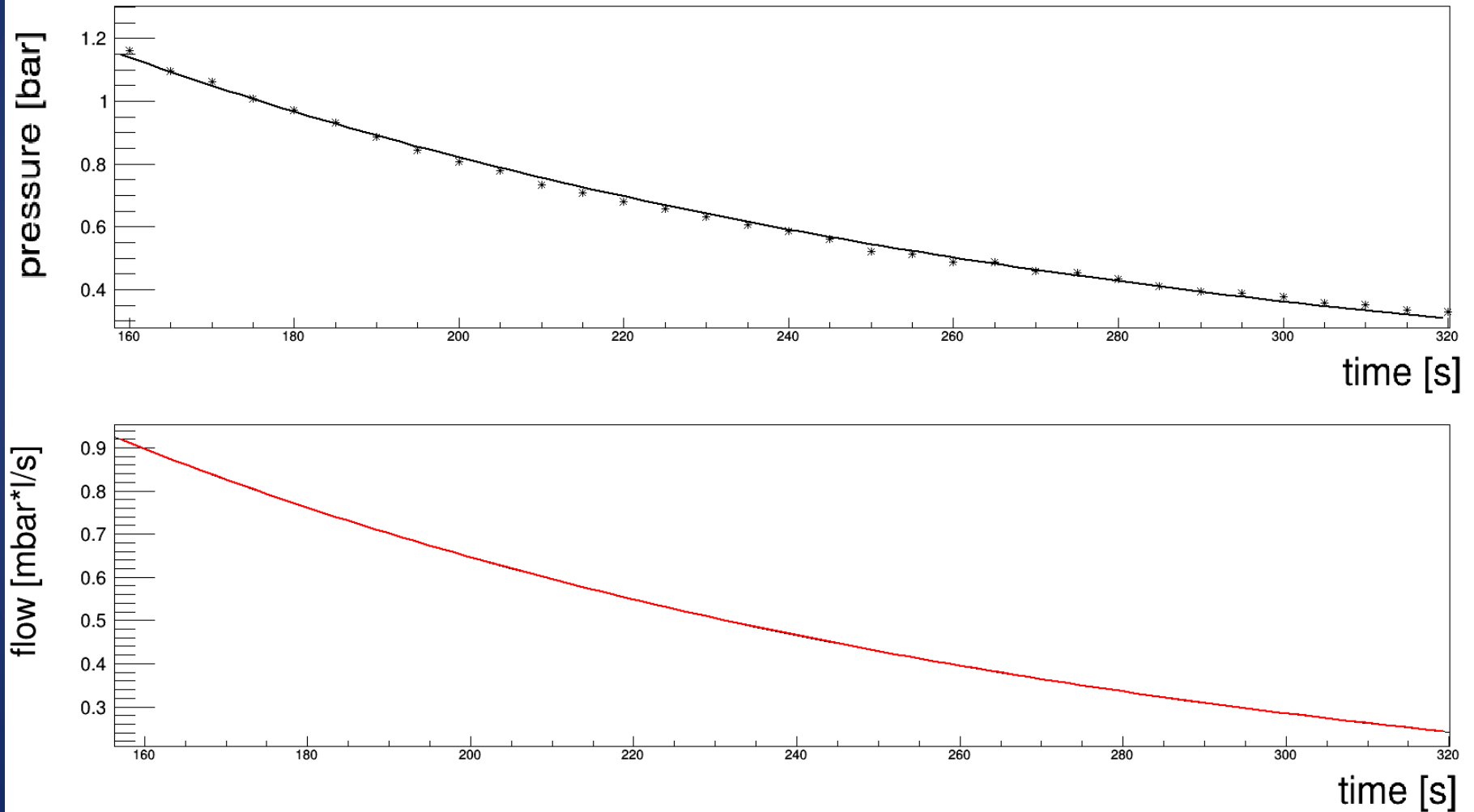
Possible offline methods used for Krypton detection are e.g. LLC, ATTA, GC-RGMS

while natural Krypton is aimed to be below <0.5 ppt in the used xenon

Example signal (~ 6ppm)



Flow correction

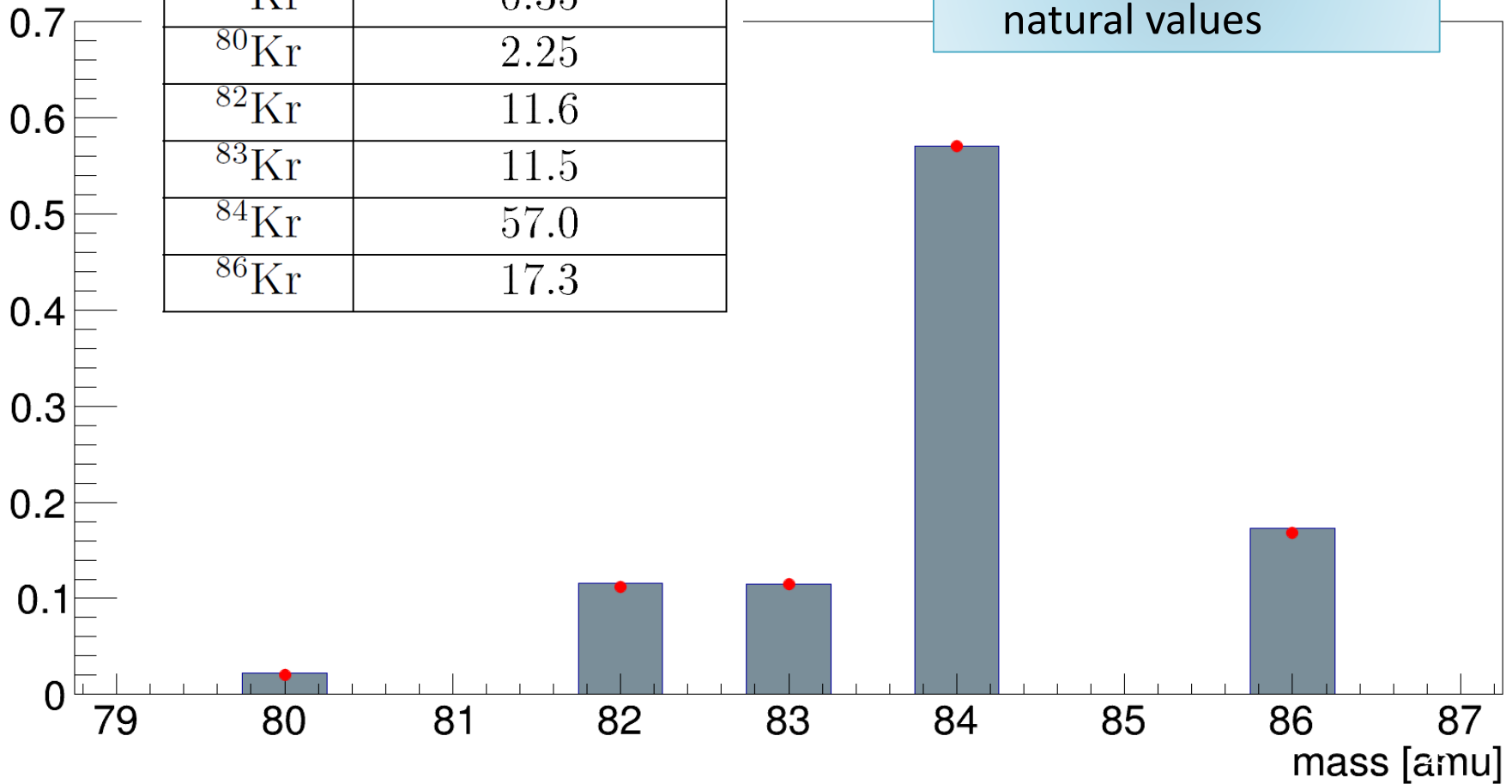


Isotopic fraction ($\sim 6\text{ppm}$)

Isotope	Abundance in %
^{78}Kr	0.35
^{80}Kr	2.25
^{82}Kr	11.6
^{83}Kr	11.5
^{84}Kr	57.0
^{86}Kr	17.3

Isotopic abundance is matching with natural values

relative abundance



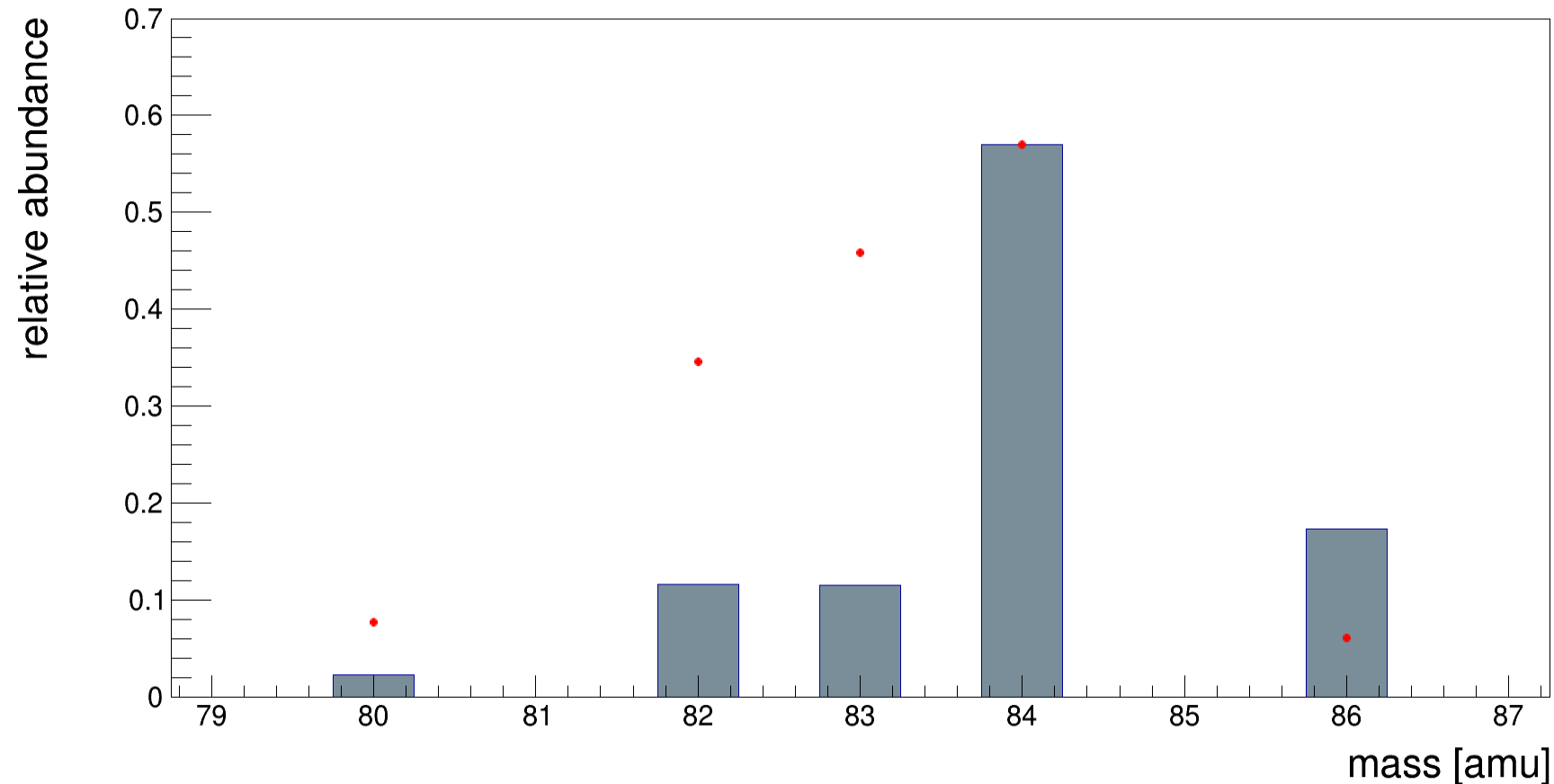
Isotopic fraction low doping (~ 0.3 ppb)

NO MATCH!



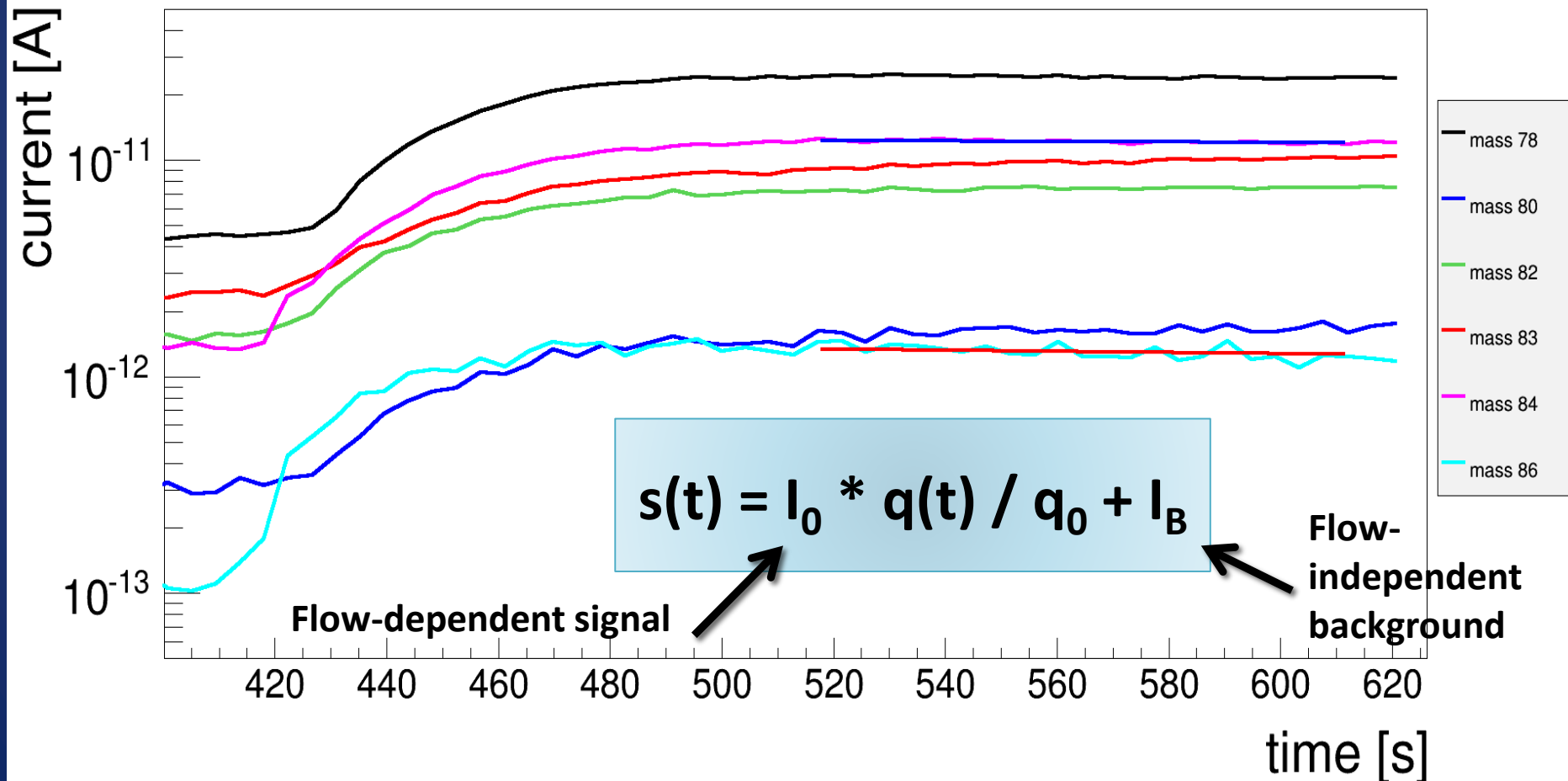
Background?

Kr isotopes doping (0.37 ppb) 07.07.14



Analysis with background fit

Kr isotopes doping (0.37 pbb) 07.07.14



Volume 1 – Volume 3

2300 mbar
xenon

Volume 4

200 mbar
krypton

Volume 1 – Volume 3

Volume 4

2300 mbar
xenon

200 mbar
krypton

75 mbar krypton + 1250 mbar xenon

→ 1500 mbar Kr + Xe

Volume 1 – Volume 3

Volume 4

2300 mbar
xenon

200 mbar
krypton

75 mbar krypton + 1250 mbar xenon

→ 1500 mbar Kr + Xe

evacuated down to 10^{-5} mbar

1325 mbar
Xe with
5% Kr

Volume 1 – Volume 3

Volume 4

2300 mbar
xenon

200 mbar
krypton

75 mbar krypton + 1250 mbar xenon

→ 1500 mbar Kr + Xe

evacuated down to 10^{-5} mbar

1325 mbar
Xe with
5% Kr

570 mbar xenon with 5% krypton

Volume 1 – Volume 3

Volume 4

2300 mbar
xenon

200 mbar
krypton

75 mbar krypton + 1250 mbar xenon
→ 1325 mbar Kr + Xe

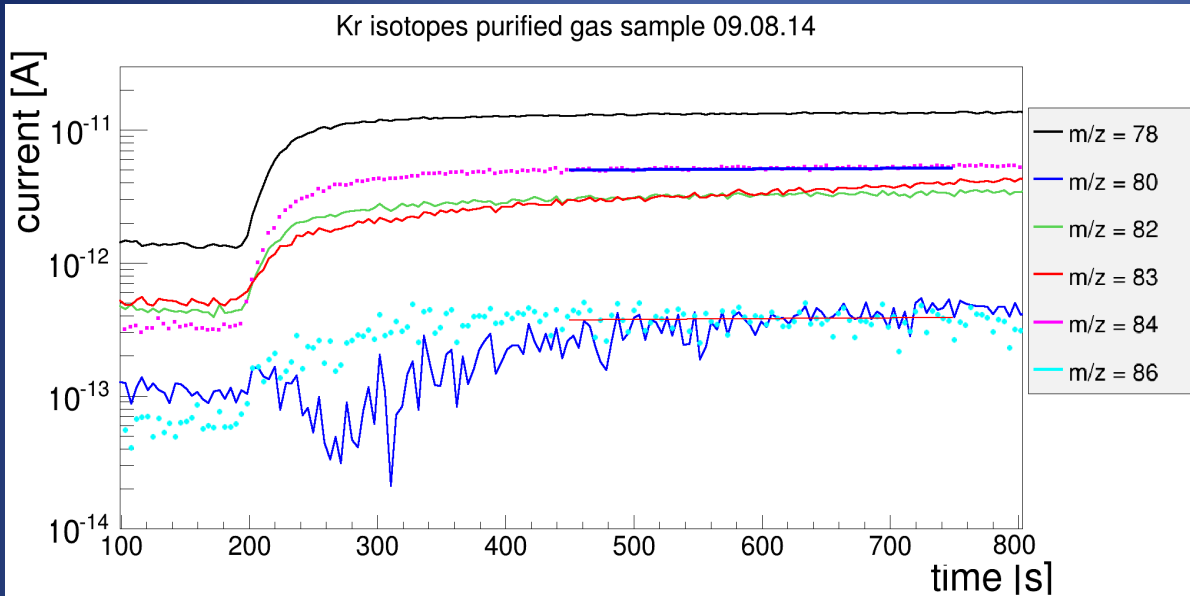
**Mixing time is
in order of
hours!**

evacuated down to 5 mbar

1325 mbar
Xe with
5% Kr

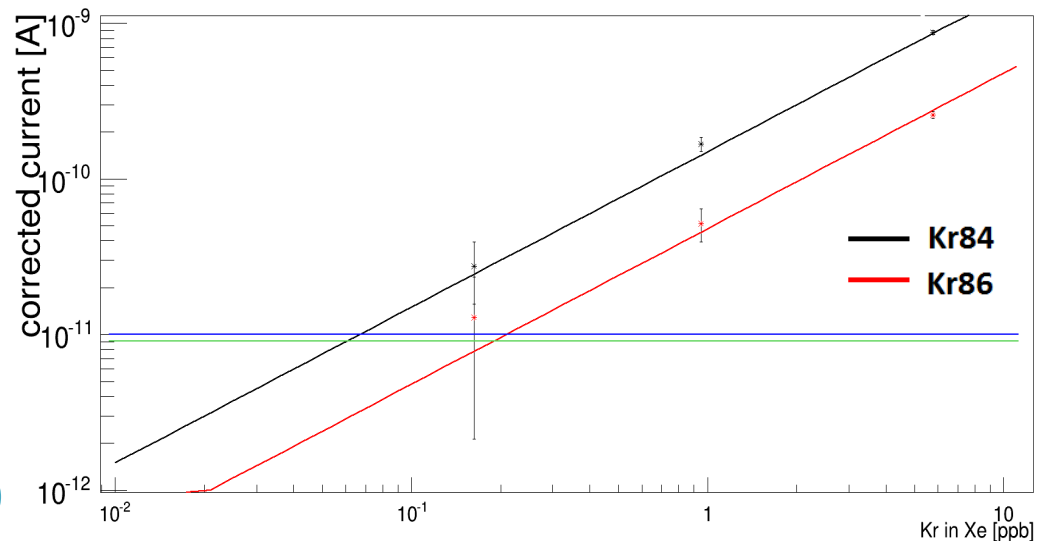
570 mbar xenon with 5% krypton

Sensitivity limit



Idea: Use ultrapure gas (sub-ppt) for an estimation of a 1σ -sensitivity limit -> Only background signals!

Final calibration RGA2 via doping July 14

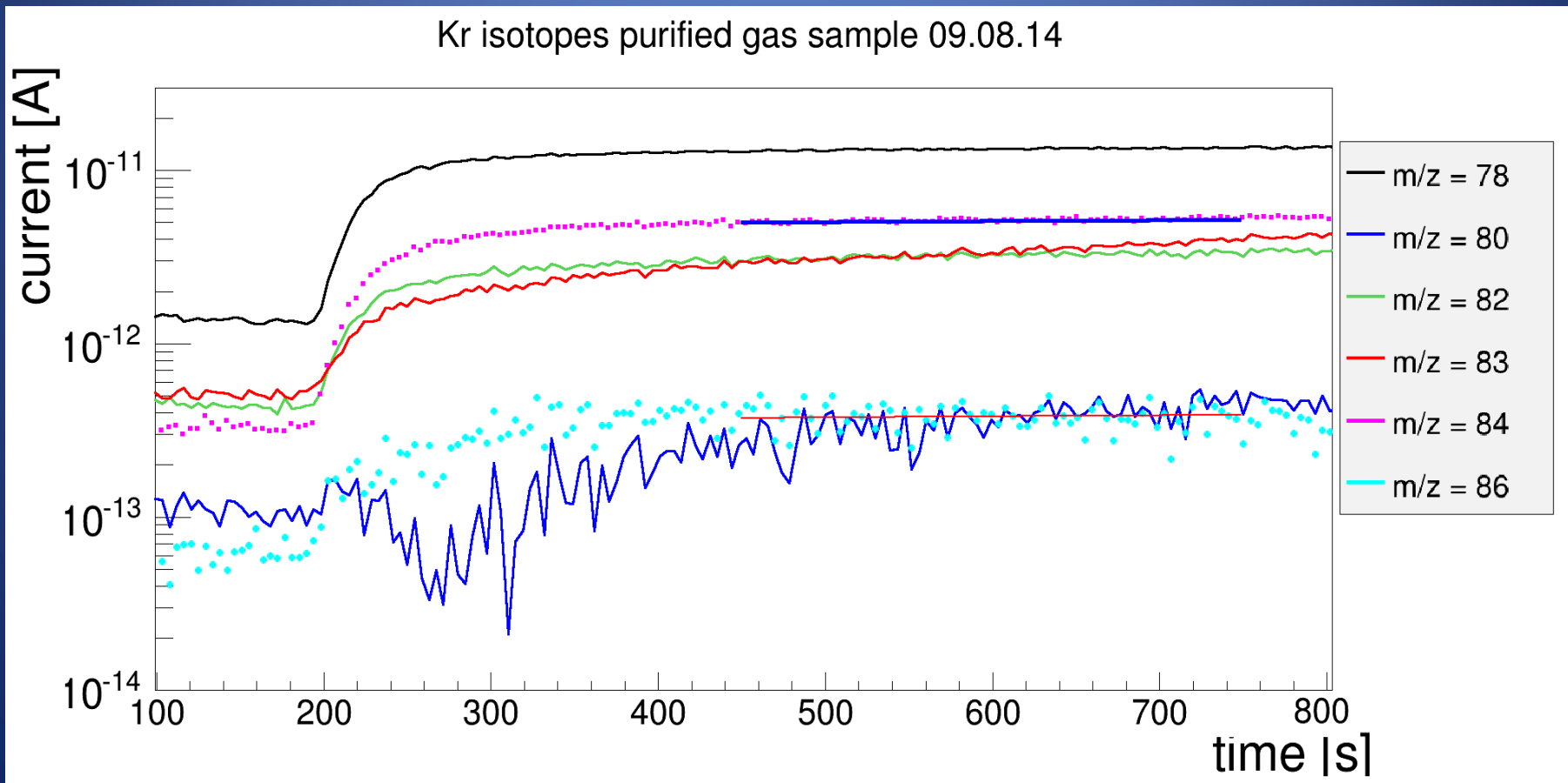


- Deviation of the background signal limits conservatively the detection sensitivity

67 ppt

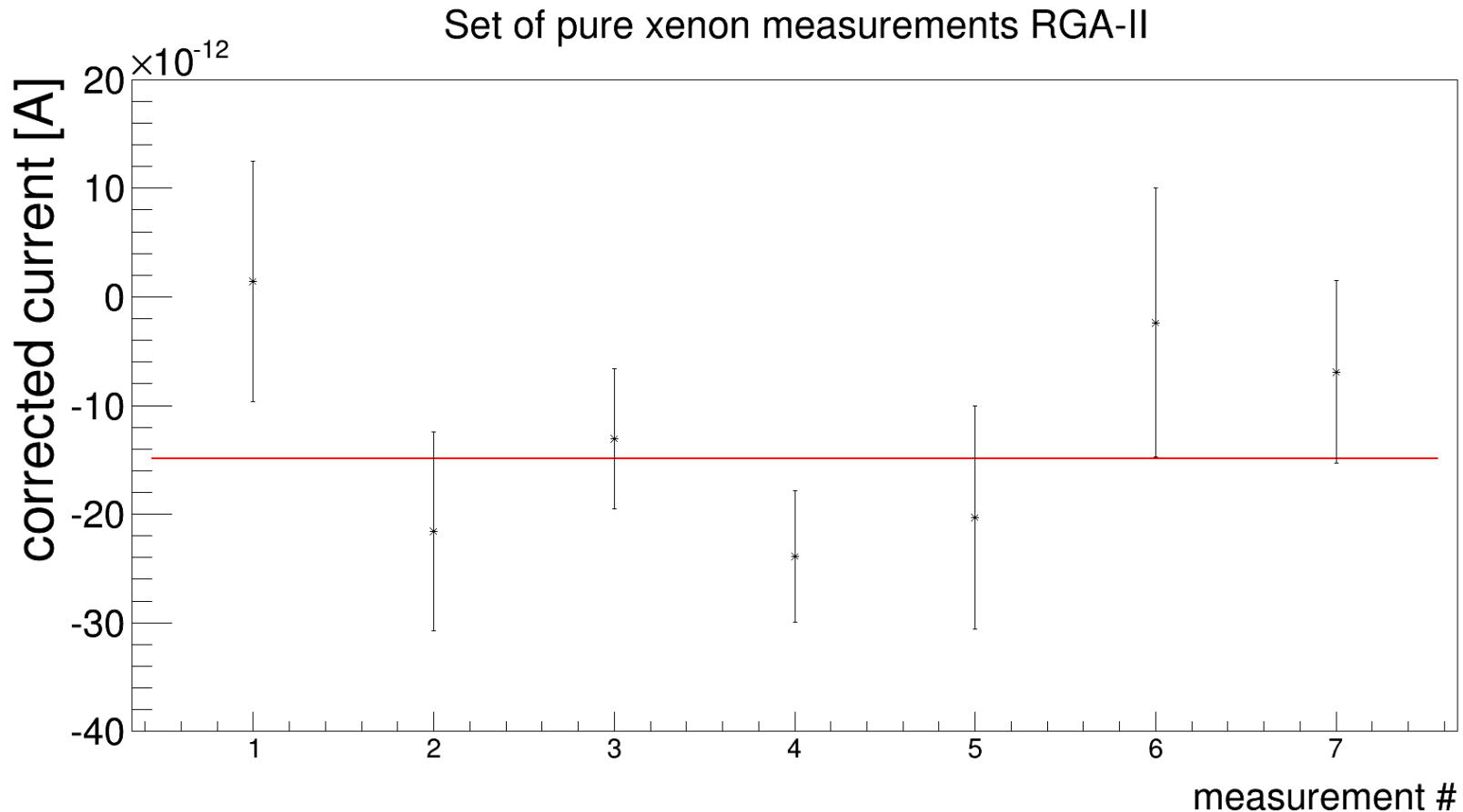
Sensitivity limit

- Idea: Use ultrapure gas (sub-ppt) for an estimation of a 1σ -sensitivity limit -> Only background signals!



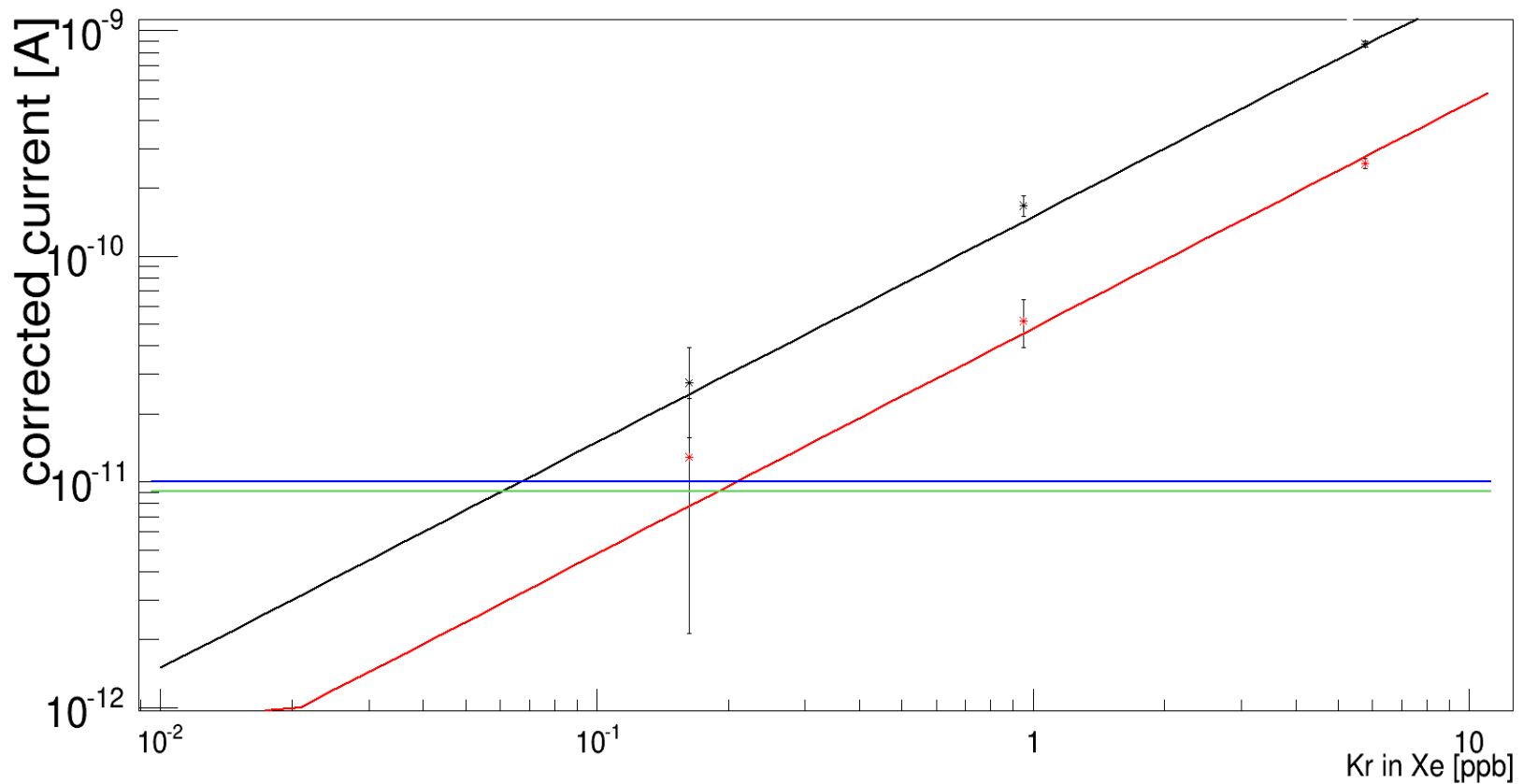
Sensitivity limit

- The uncertainty of the pure measurements determine the minimum detectable signal



Sensitivity limit

Final calibration RGA2 via doping July 14



Background ^{85}Kr

- Beta decay of ^{85}Kr into ^{85}Rb is a significant intrinsic background for XENON1T
- Determination of the ^{85}Kr concentration in xenon is of crucial importance for the experiment
Sensitivity is limited above the desired value of 0.5 ppt
**Make use of fast results and minimal consumption advantages for other applications!**
- Natural ^{85}Kr concentration is below <0.5 ppt in the used xenon
- Natural krypton abundance is measured and the ^{85}Kr concentration is derived from this value
- Can be removed along with cryogenic distillation