XENON GC/MS Analysis Neutrino Platform

R. Guida, B. Mandelli, M. Corbetta

CERN

EP-DT
Detector Technologies

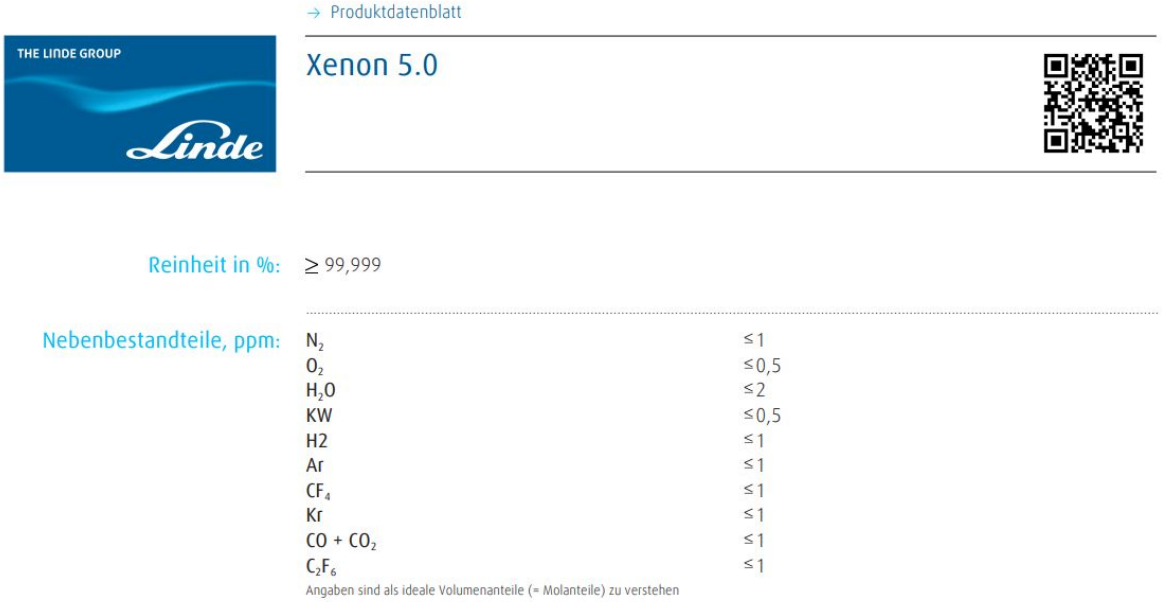
STATUS

- detector > 700’000 kg **liquid Argon**
- 1kg Xenon injected in liquid Argon >> 0.00015 % of Xe = **1.5 ppm of Xe**
- Xenon bottle from Linde, purity 5.0 >> **max impurities 10 ppm**
- possible impurity in liquid Argon 1.5 ppb

- *Linde* declares a set of impurities, each lower than 1-2 ppm
- Ar, Kr should not affect the detector; CO2, O2, H2O should be adsorbed by dedicated filters (on liquid phase, 700’000 kg recirculated every week)

BOTTLE IMPURITIES

- Impurities declared by Linde for Xenon 5.0



→ Produktdatenblatt

THE LINDE GROUP

Linde

Xenon 5.0

Reinheit in %:	≥ 99,999	
Nebenbestandteile, ppm:	N_2	≤ 1
	O_2	≤ 0,5
	H_2O	≤ 2
	KW	≤ 0,5
	H2	≤ 1
	Ar	≤ 1
	CF_4	≤ 1
	Kr	≤ 1
	$CO + CO_2$	≤ 1
	C_2F_6	≤ 1

Angaben sind als ideale Volumenanteile (= Molanteile) zu verstehen

GC/MS ANALYSIS

- Xenon is sent to Gas Chromatograph,
  three columns are available: PPU, OV, MolSiev
- OV1 and PPU column can be coupled with Mass Spectrometer
- Different temperatures were tried for OV1 and PPU but with similar results, best method is standard RPCMethod (95°C)
- First analysis are done with plastic pipe as connection, then moved to inox pipe to limit possible Air/O2/N2/H2O adsorption

Method:	C:\Soprane\Method\LAB256_Xenon		
	Module A	Module B	Module C
Module	OV1	PPU	MS5A
Inlet temp. (°C)			
Inject temp. (°C)	70.00	80.00	85.00
Column temp. (°C)	95.00	95.00	105.00
Pump (sampling time) (s)	Pump1:	60.00	
Sampling time (s)	60.00	60.00	60.00
Inject time (ms)	25.00	25.00	100.00
Backflush time (s)			
Run time (s)	240.00		
Column pressure (psi)	28.00	28.00	33.00
Detector	ON	ON	ON

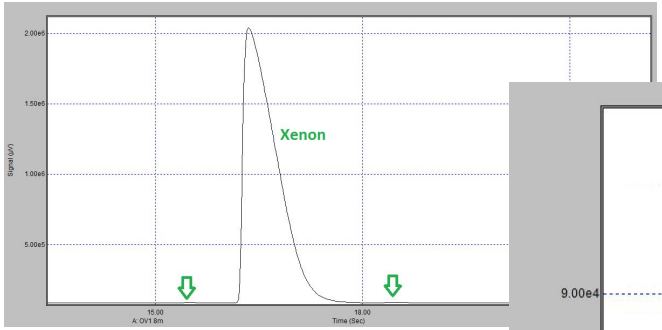
GC/MS ANALYSIS

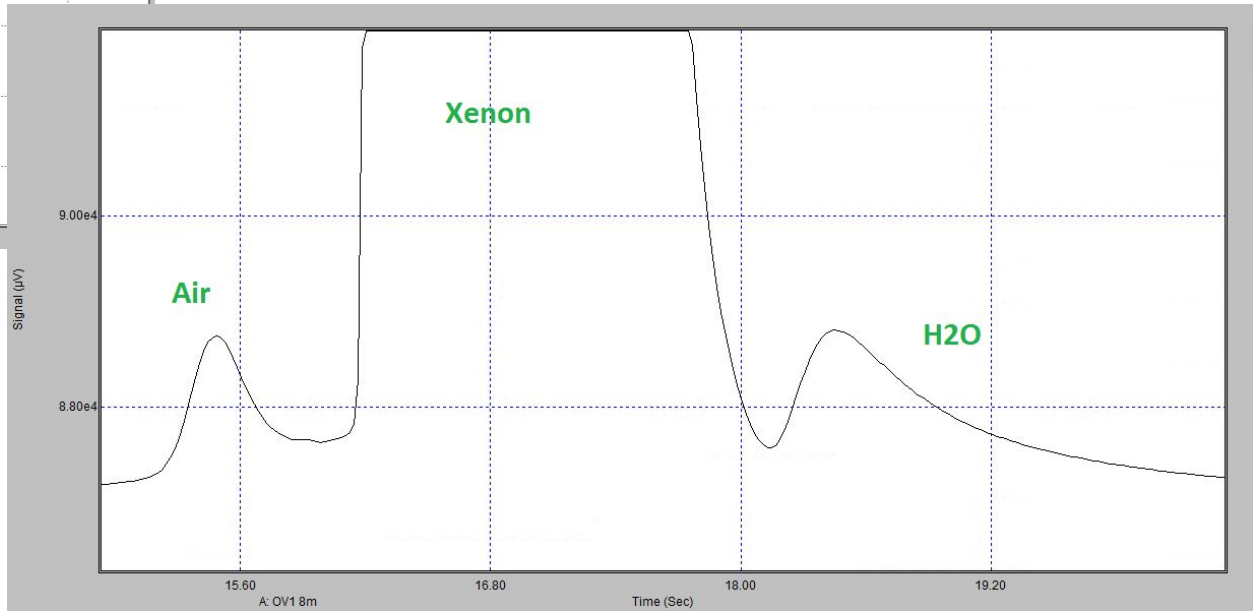
- Chromatograms can be used to quantify elements with peak area,
  but a calibration is needed > rough estimation with “fraction method”
- Proportionality between area and concentration is linear up to 20%
- Retention time of a peak in the GC is the same as in the MS
  > we can check peak correspondence

GC/MS ANALYSIS

- Both coupling are tested, still the most relevant is with PPU as it can separate possible impurities we are looking for > **CF4, C2F6**
- Mass spectrometer analysis are repeated for different mass ranges, as reducing the mass range can help improving instrument sensitivity to low concentrations (expected ~ fraction of PPM)
- NIST library available in MS software for identification
  > only possible if all mass lines are identified, i.e. not in small range

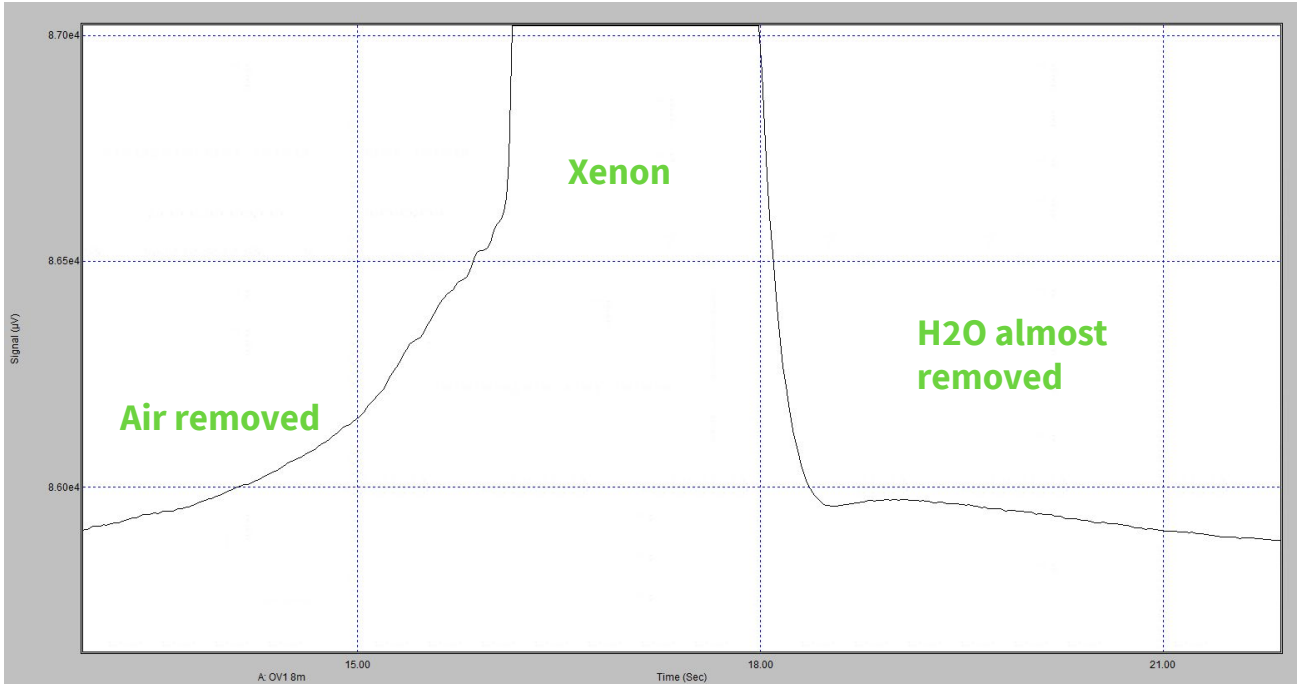
GC ANALYSIS : OV1 COLUMN plastic





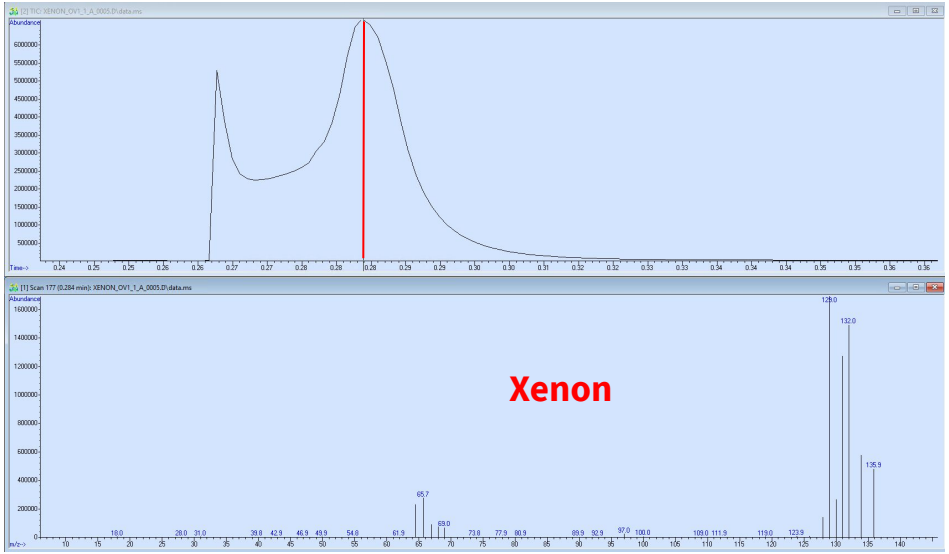
RT 15.5s > Air
RT 16.5s > Xe
RT 18.5s > H2O

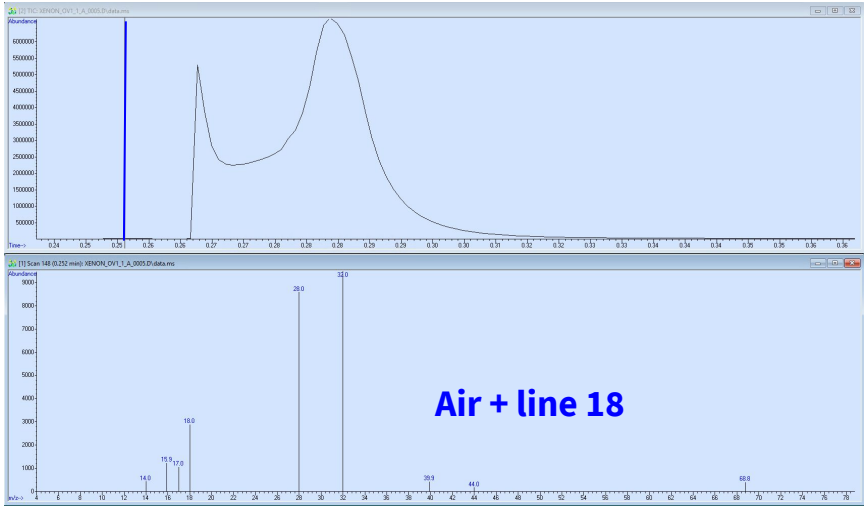
GC ANALYSIS : OV1 COLUMN inox



MS ANALYSIS : OV1 COUPLING

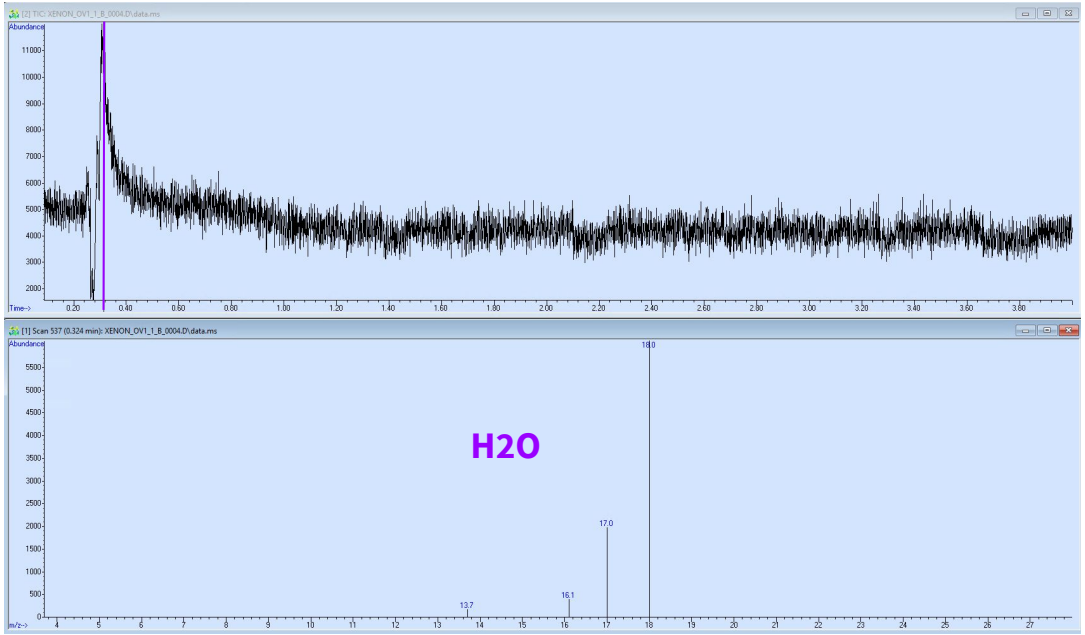
full mass range > identified Xenon, found lines 28/32 (Air) and 18(H2O)



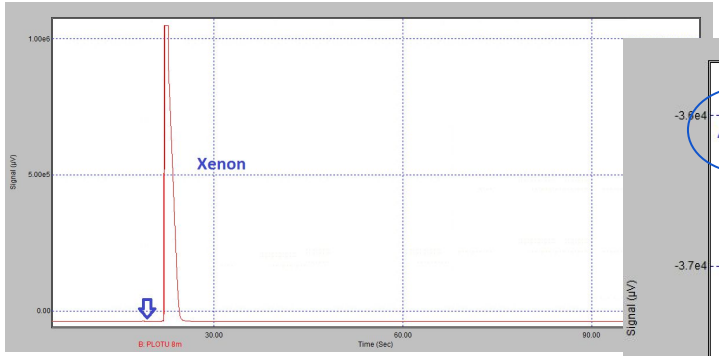


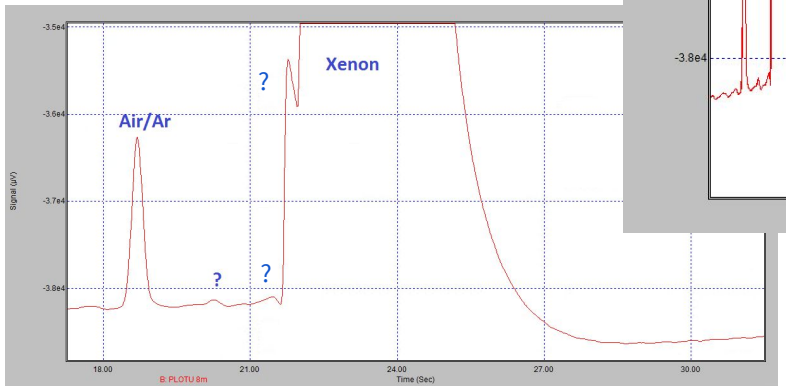
MS ANALYSIS : OV1 COUPLING

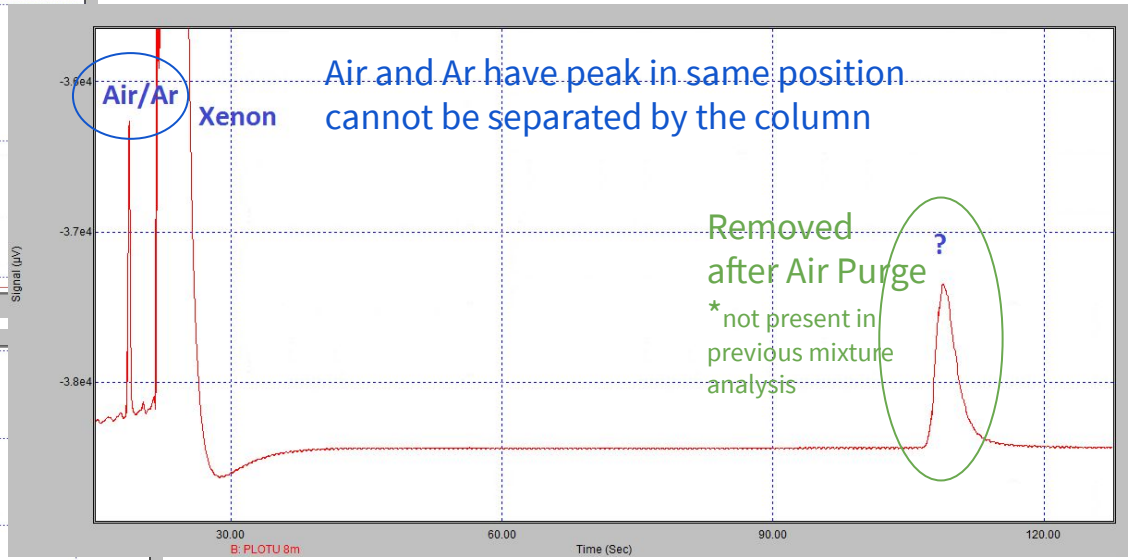
mass range 10-15 > identified H2O (RT same as GC)



GC ANALYSIS : PPU COLUMN plastic



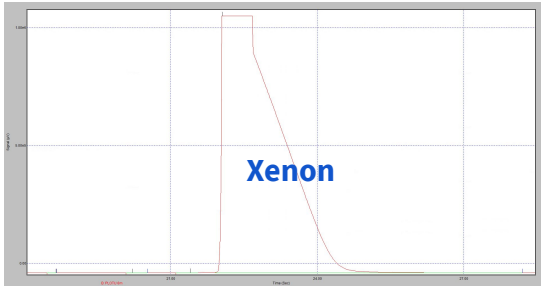


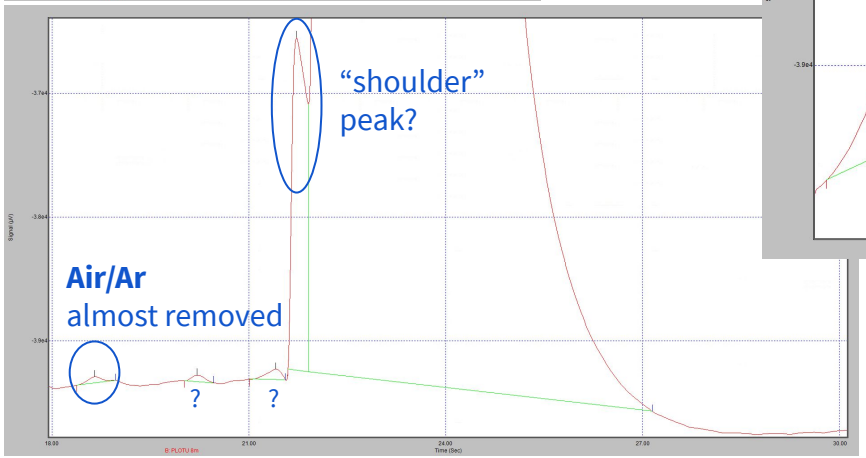


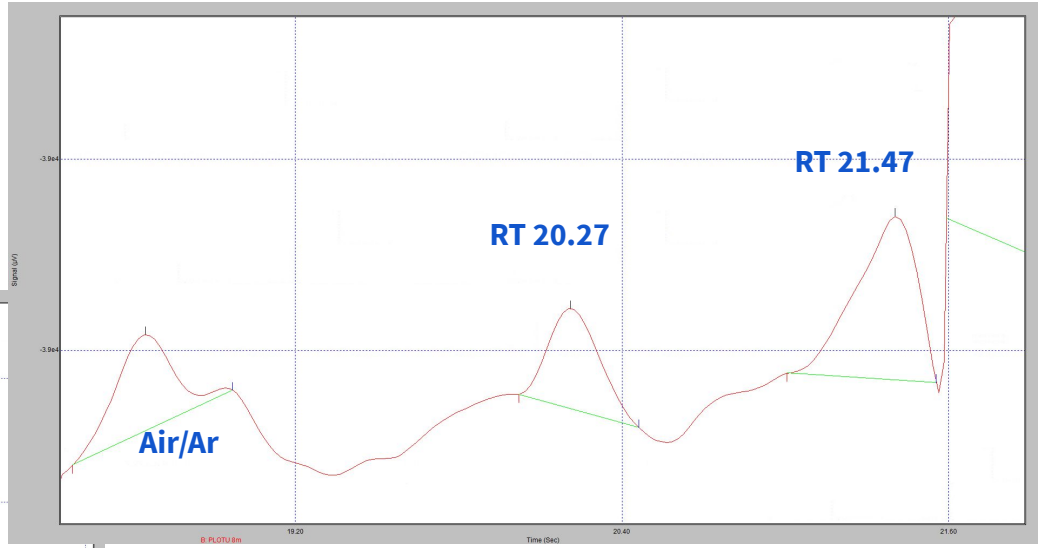
RT 18.7s > Air
RT 22.4s > Xe
RT 108.9s > Ion69?

RT 20.27s > unidentified
RT 21.47s > unidentified
RT 21.78s > unidentified

GC ANALYSIS : PPU COLUMN inox







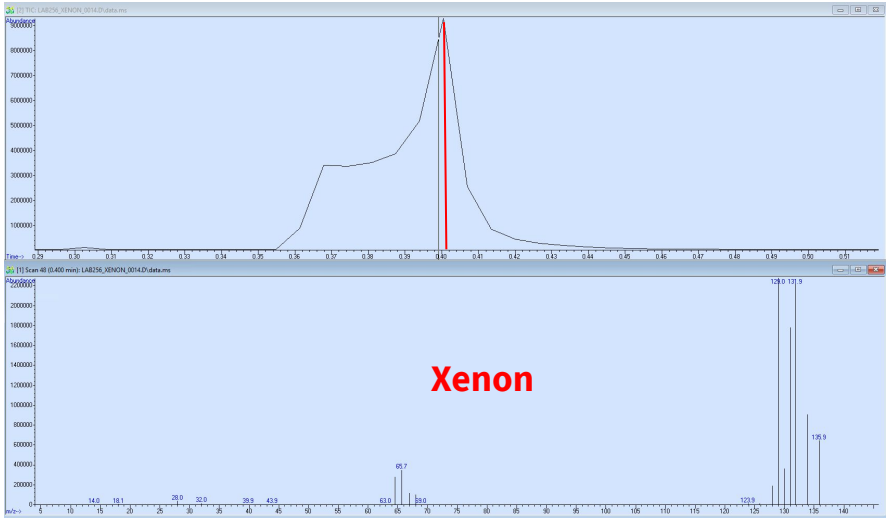
RT 18.7s > Air
RT 22.4s > Xe

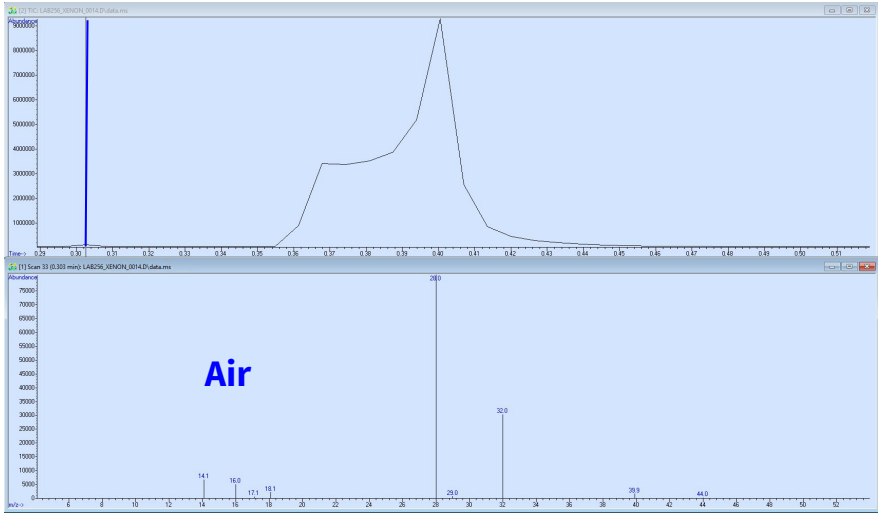
look at same RT in MS

RT 20.27s > unidentified
RT 21.47s > unidentified
RT 21.78s > unidentified

MS ANALYSIS : PPU COUPLING

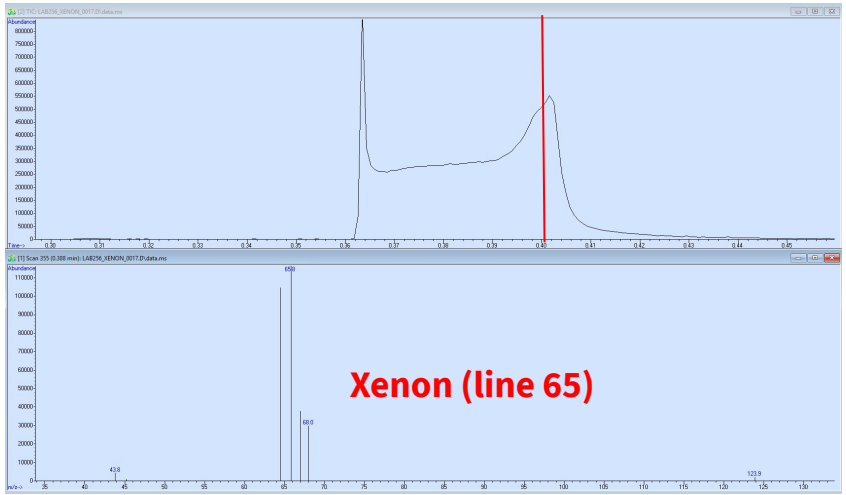
full mass range > identified Air and Xenon

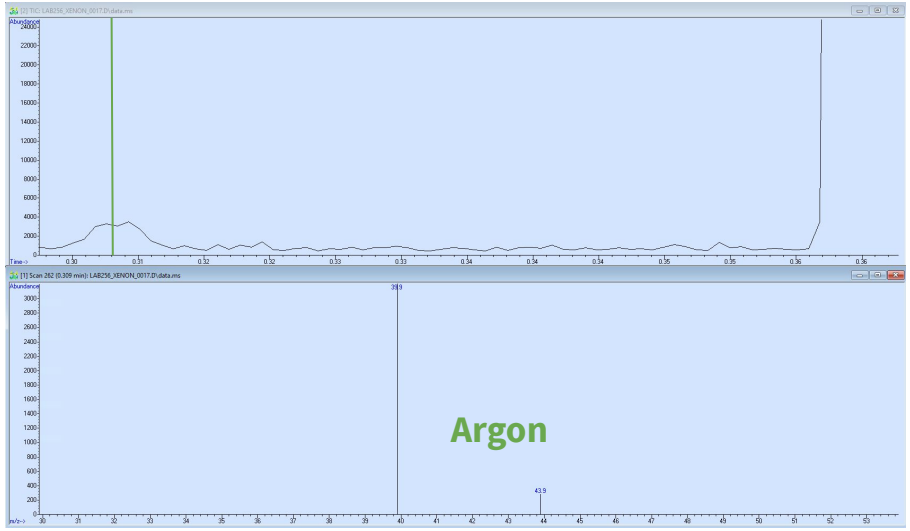




MS ANALYSIS : PPU COUPLING

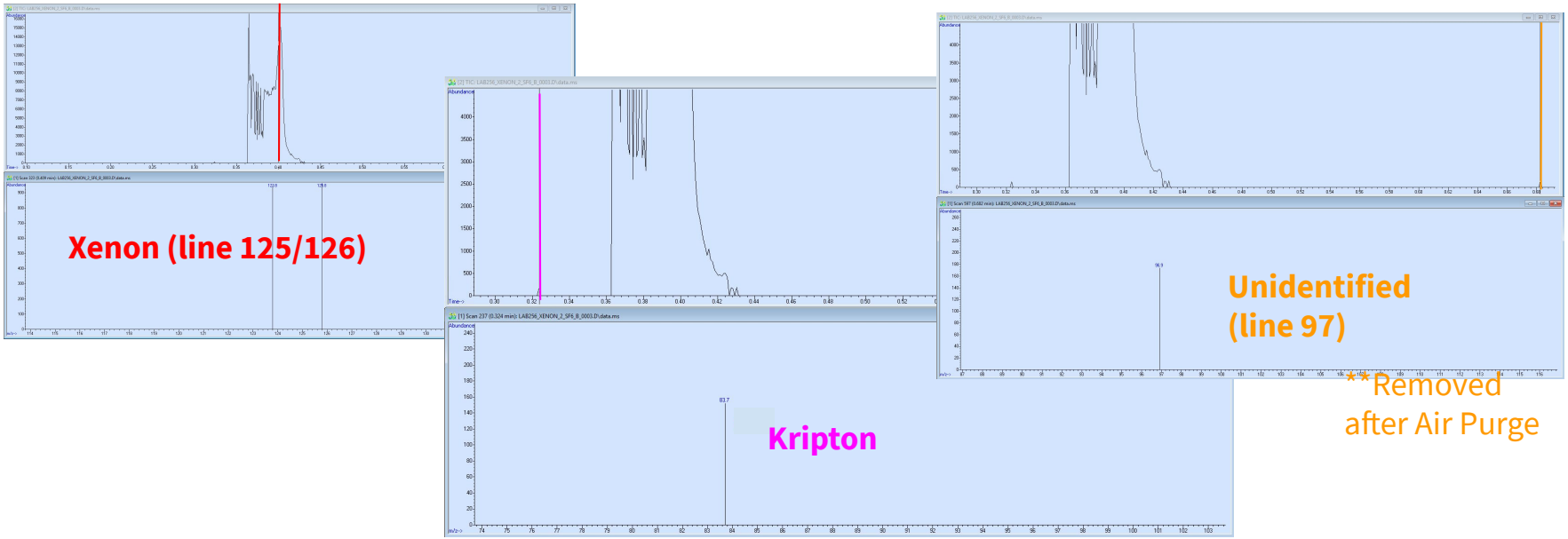
mass range 33-125 (out Air and Xe)
> identified lower line of Xe and Argon





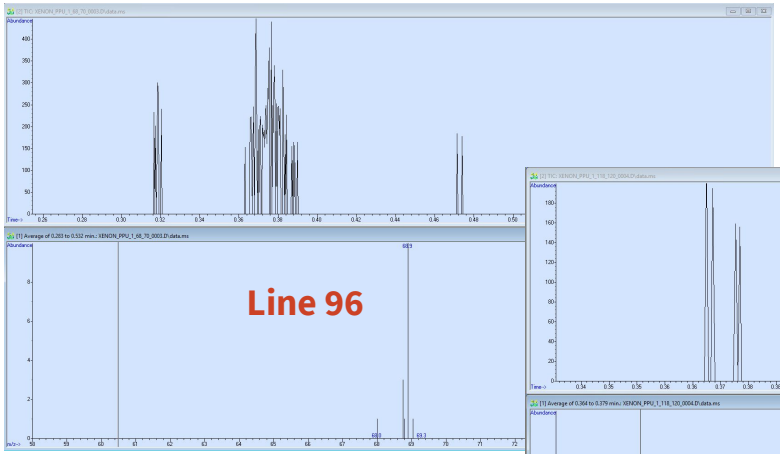
MS ANALYSIS : PPU COUPLING

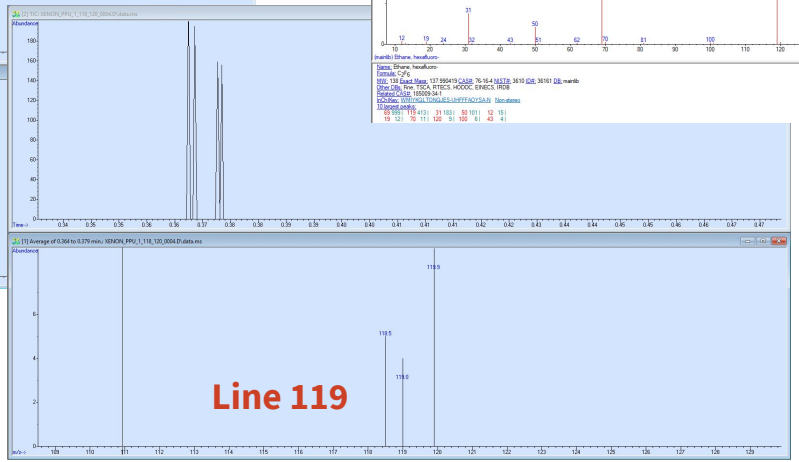
mass range 70-128 (out Air, Ar and Xe, possibly in SF6 (127))
> identified lower line of Xe, Kripton

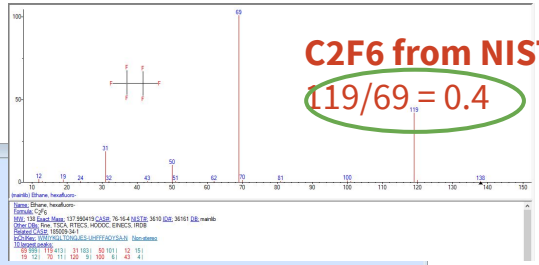


MS ANALYSIS : PPU COUPLING

specific search for CF4/C2F6 > range around mass 69 (CF4, C2F6) and 119 (C2F6)







C2F6 from NIST

119/69 = 0.4

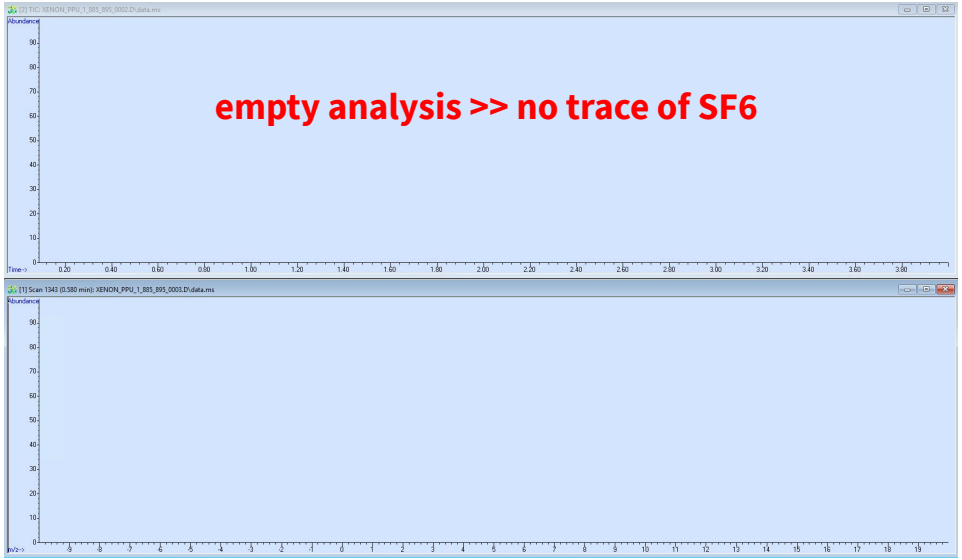
from MS relative abundance

119/69 = 0.41

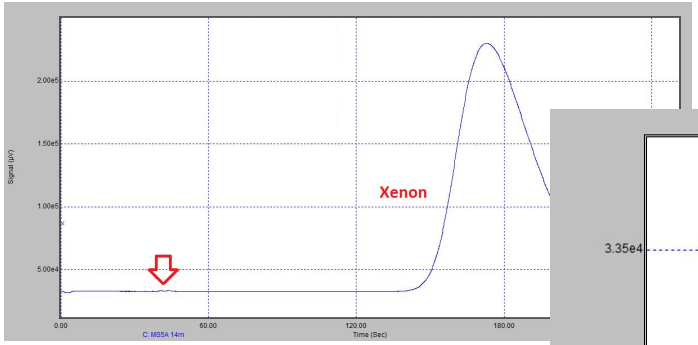
>> presence of C2F6

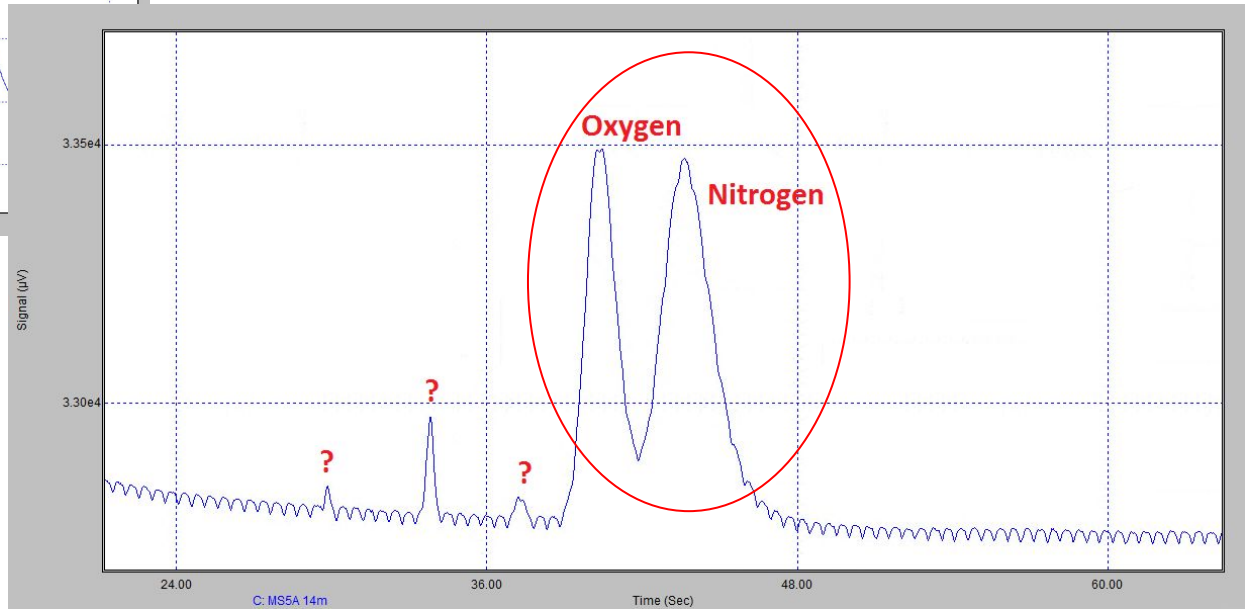
MS ANALYSIS : PPU COUPLING

specific search for SF6 > range around mass 89, 108



GC ANALYSIS : MS COLUMN plastic

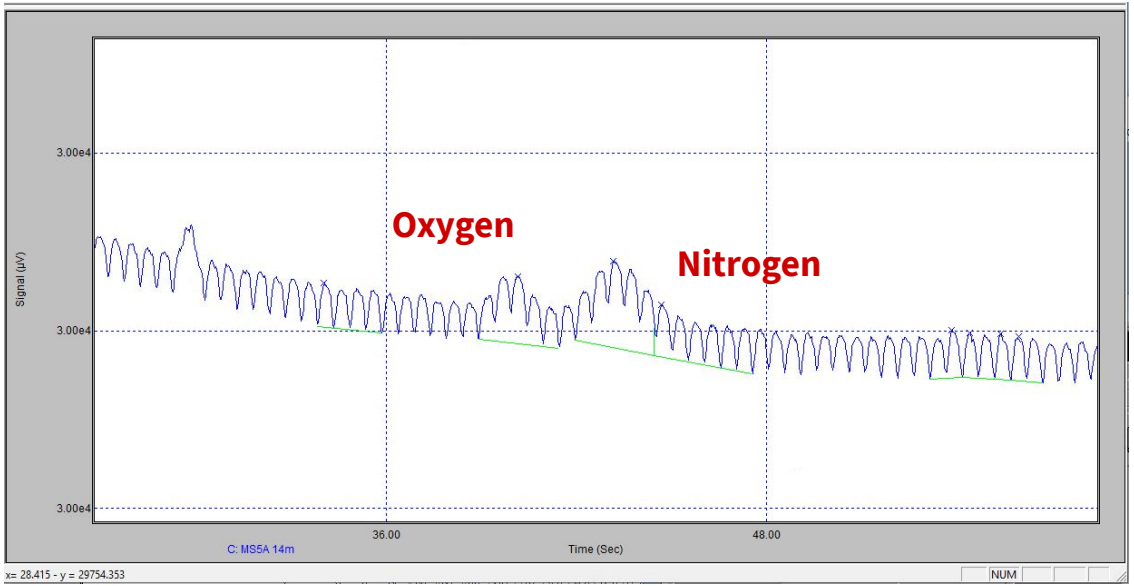




O2 and N2 peaks identified
not Air ratio (1:4)
> surely extra O2
> could be also extra N2
calibration needed to quantify

> 1k ppm > plastic pipe!

GC ANALYSIS : MS COLUMN inox



inox = almost no Air intake
O2 and N2 peaks identified
still not Air ratio (1:4)
>> extra O2 is present

quantification done from
(very) old calibration
O2 ~ 100 ppm

no coupling available with MS column…

CONCLUSION

- **Impurities found in the Xe 5.0 bottle:**
  - Argon, Kripton : expected, not relevant
  - H2O : only with plastic pipe = analysis pollution
  - **O2 : expected but in low conc. (<1ppm), quantified from GC around 100ppm**
  - **C2F6 : expected in low conc. (< 1ppm)**

    also identified with IR spettroscopy!
- **Ongoing:**
  - proper calibration for O2 quantification with GC
  - quantification of C2F6 with MS, not straightforward + calibration needed
  - analysis of previously used Xe bottle > no problems for detector prototype