Sub-ppb Level Detection of BTEX Gaseous Mixtures with a Compact Prototype GC Equipped with a Preconcentration Unit

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Atmosfair – June 5 & 6 2019

Outline

1. General Background

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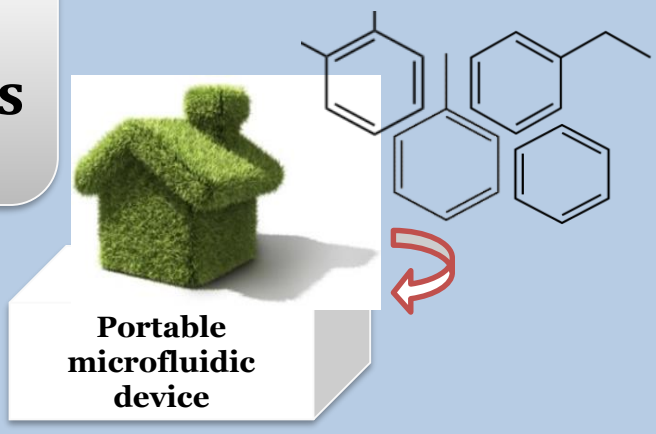
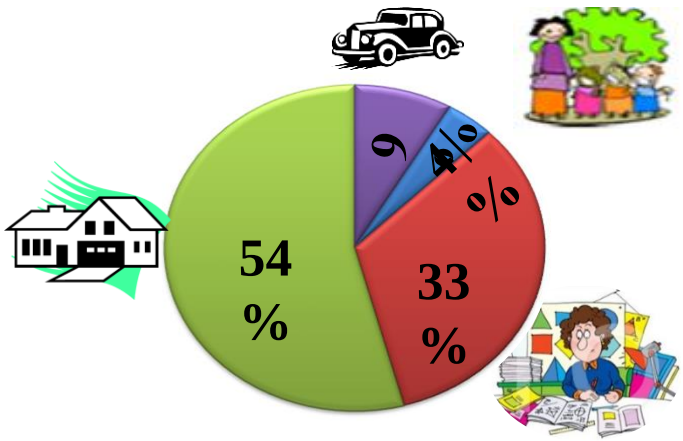
5. Conclusion and perspectives



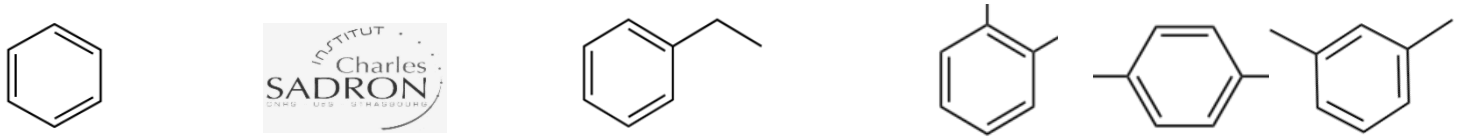
Time spent in enclosed environment can reach 90%



Indoor air quality (IAQ) is responsible of more than 4 millions of premature deaths per year*

Indoor Air is contaminated by a wide variety of Volatile organic compounds (VOCs)

BTEX: Benzene, Toluene, Ethyl Benzene and Xylenes



BTEX emission sources in indoor air

** World Health Organisation*

1. General background

BTEX effets on health and regulations

Compound	Effect*
Benzene	Human carcinogenic class A (leukaemia)
Toluene	Harmful to Nervous central system
Ethylbenzene	Pneumonitis
Xylenes	Liver and kidney disorder

European Union has fixed a threshold value of **1.6 ppb (5 µg/m³)** in public building since 2013**

In France this threshold value will decrease to 0.6 ppb (2 µg/m³) in 2018

This new regulation makes necessary the development of **portable and sensitive instruments** for BTEX monitoring in public buildings

** World Health Organisation ** Decret n°2011-1728 of December 2011 for Indoor air monitoring-French government*

1. General background | *BTEX detection methods*

		Methods	Detection limit	Analytical time	Portability	Gas consumption	References
OFFLINE	ATD-GC-FID 	(Laboratory GC) coupled to different detectors MS/FID/PID	0,22-85 ppt	1 h	No (bulky)	200 mL/min	Liaud et *al.* (2014) [1]
ONLINE		PTR-MS	12 ppt	10 min	No (bulky)	50 mL/min	Graus et *al.*(2010) [2]
		**Transportable Analyzer**	**100 ppt**	**10 min**	**Transportable (15 kg)**	**30 mL/min**	Liaud *et al.* (2014) [1]

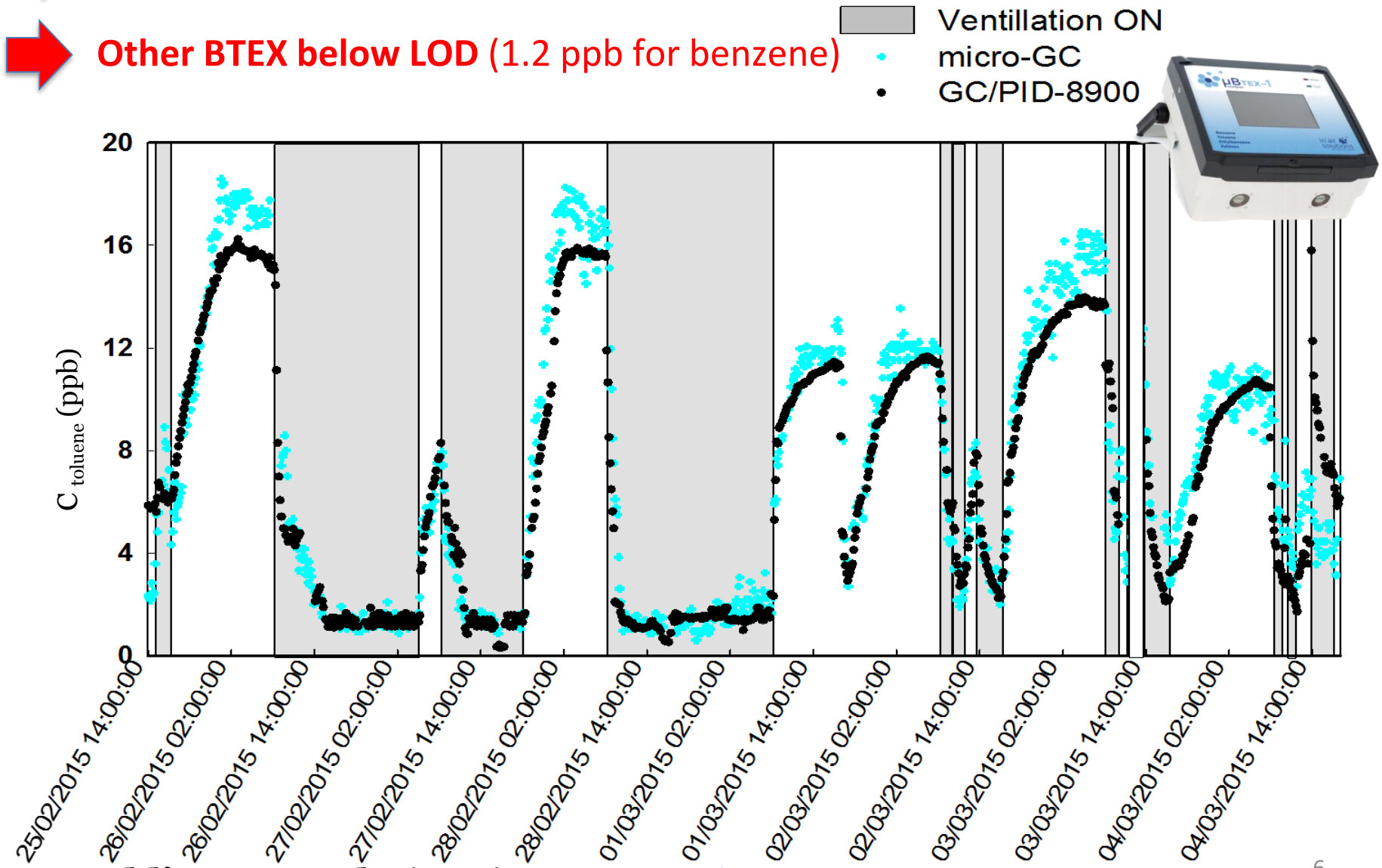
major default: the need of big gas cylinder due to their high gas consumption

Design of a new instrument based on microfluidic device

[1] C. Liaud, N.T. Nguyen, R. Nasreddine, S. Le Calvé, Talanta. 127 (2014),
[2] M. Graus, M. Müller, A. Hansel, J. Am. Soc. Mass Spectrom. 21 (2010)

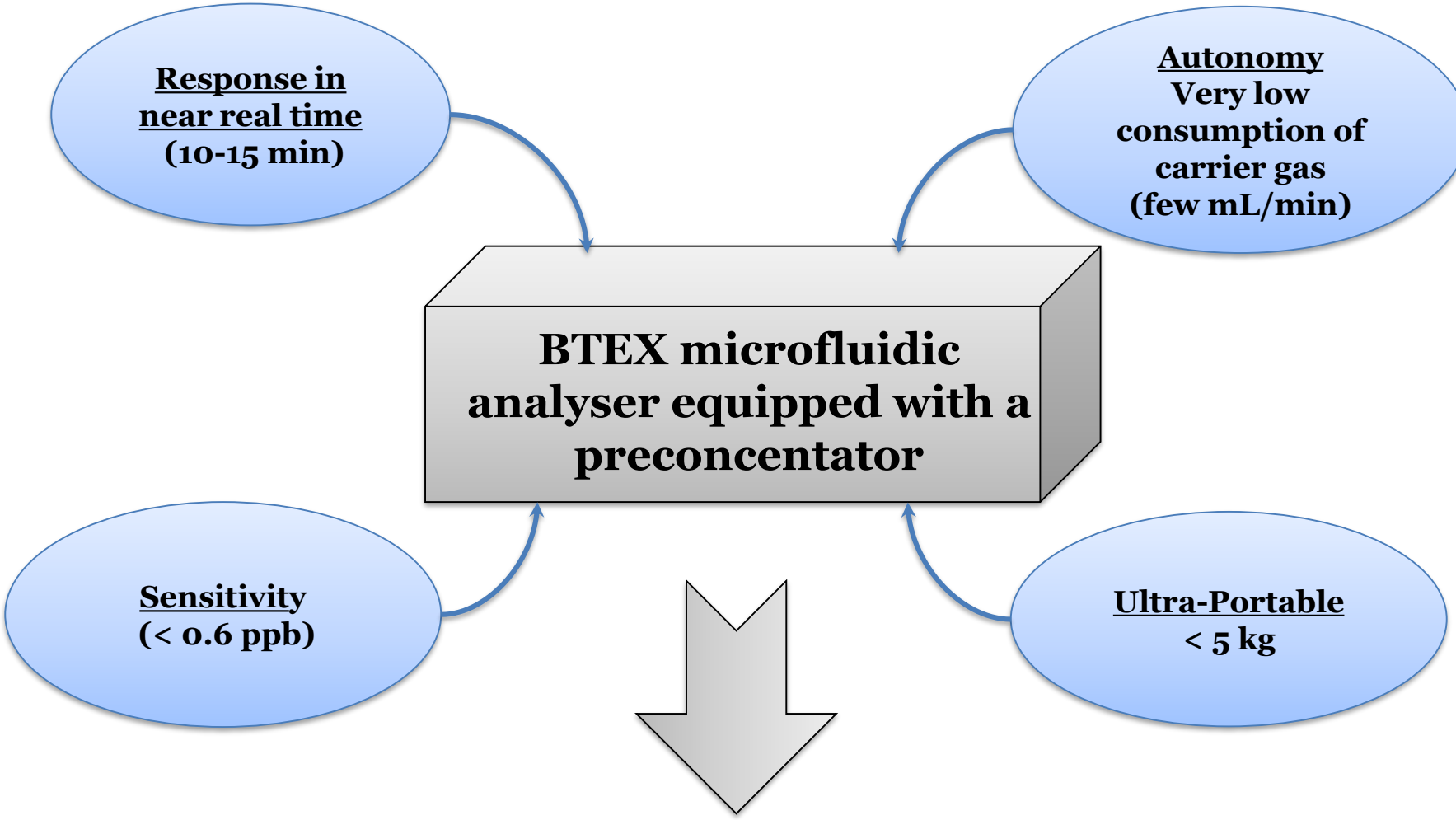
Toluene: major VOC in this classroom (floor)

Other BTEX below LOD (1.2 ppb for benzene)



Nasreddine, R. et al., (2016). *Atmos. Environ.*, **126**, 211–217.

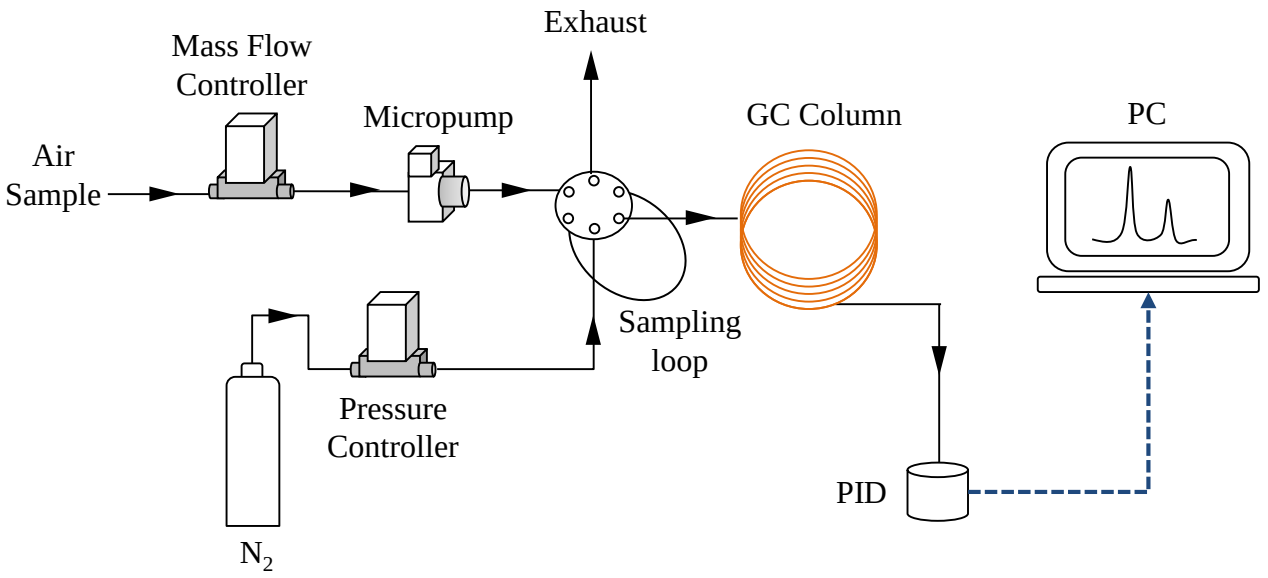
2. Objectives

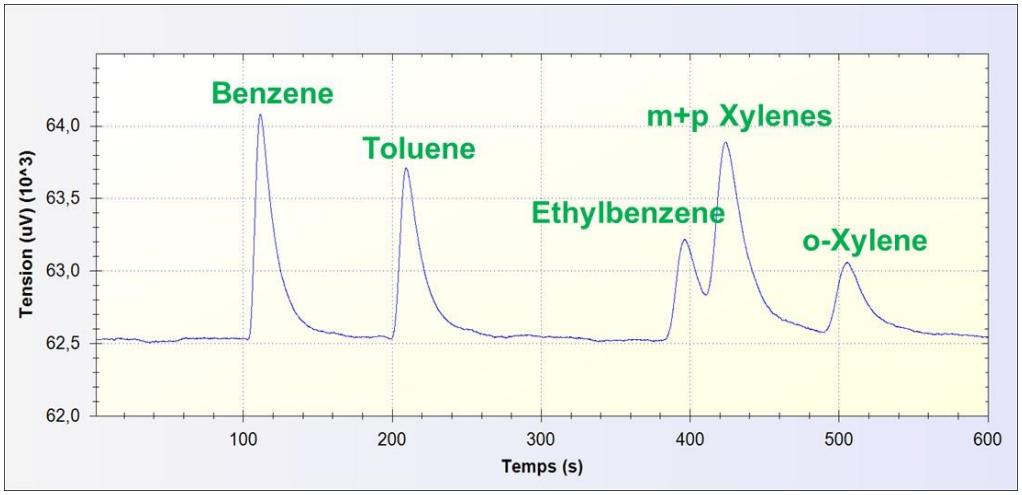


Continuous real-monitoring of BTEX in indoor air

Three steps analysis:

1. Air Sampling
2. Separation
3. Detection

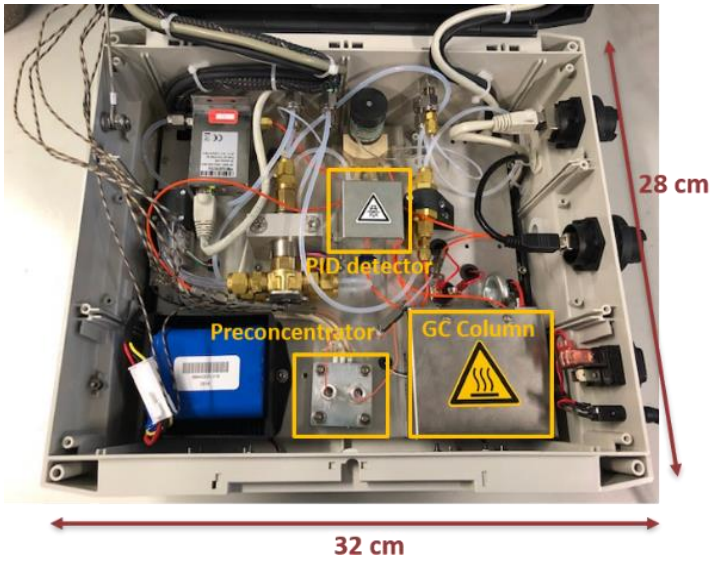




Temperature: 65°C
Carrier gas flow: N2 at 2.5 mL/min
Time of analysis: 10 min

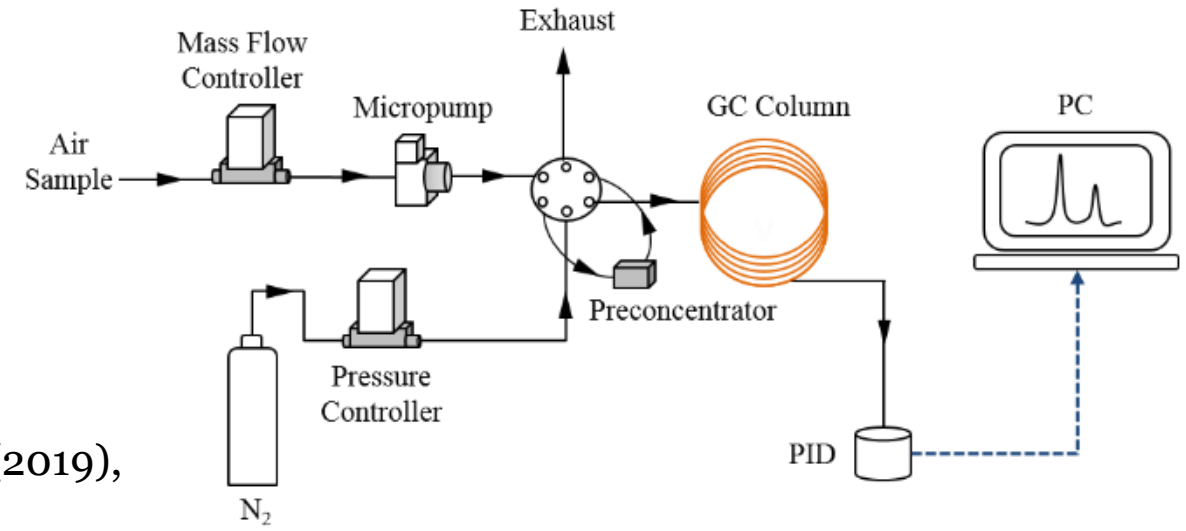
Nasreddine, R. et al. (2016), *Sensors and actuators B Chem.*, **224**, 159–169.

3. Experimental device | *Operating principle with preconcentration*



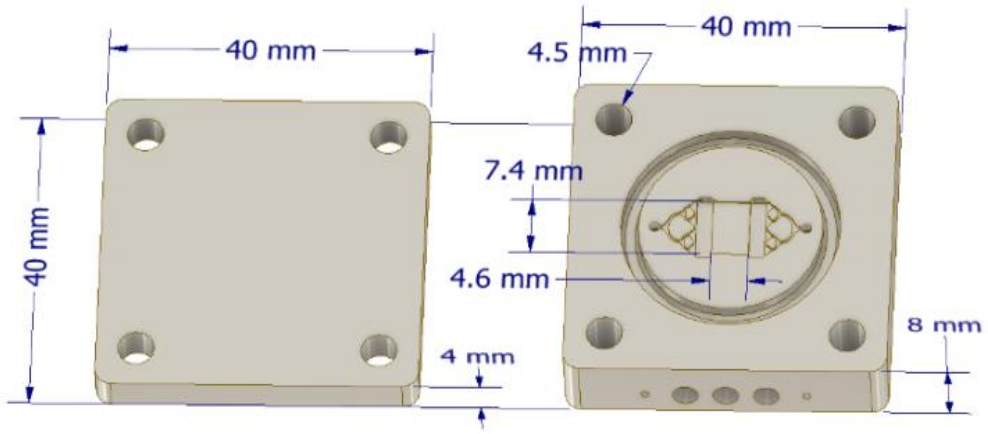
Four steps analysis:

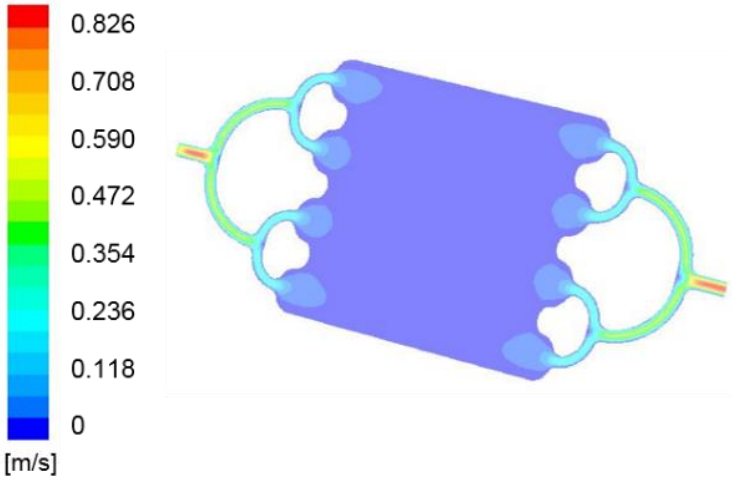
1. Air Sampling (adsorption in a preconcentrator)
2. Injection / desorption at high temperature
3. Separation
4. Detection

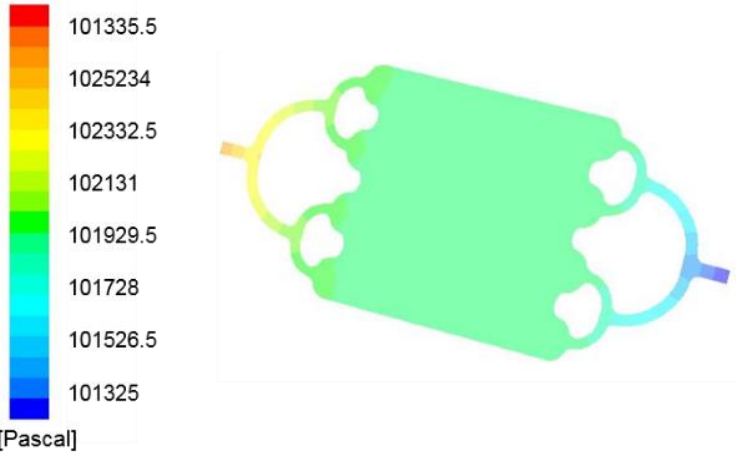


Lara-Ibeas et al. (2019), *Micromachines*, **10**, 187.

4. Results and discussion | *1st prototype of preconcentrator*







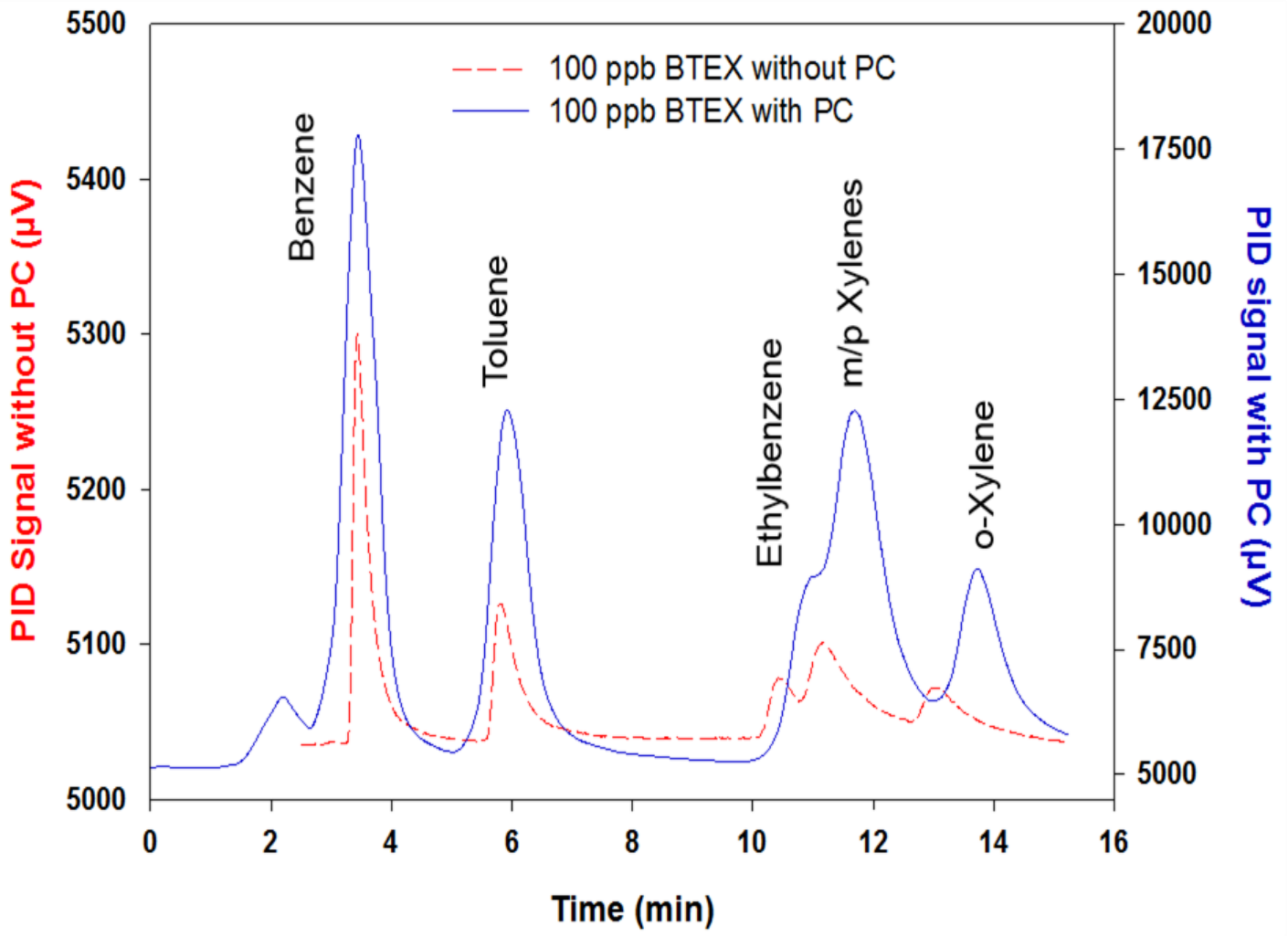
Uniform flow distribution inside the microfluidic cavity

Flow rate = 5 mL/min

Pressure drop = 9.3 Pa

Lara-Ibeas et al. (2019), *Micromachines*, **10**, 187.

4. Results and discussion | *1st prototype of preconcentrator*



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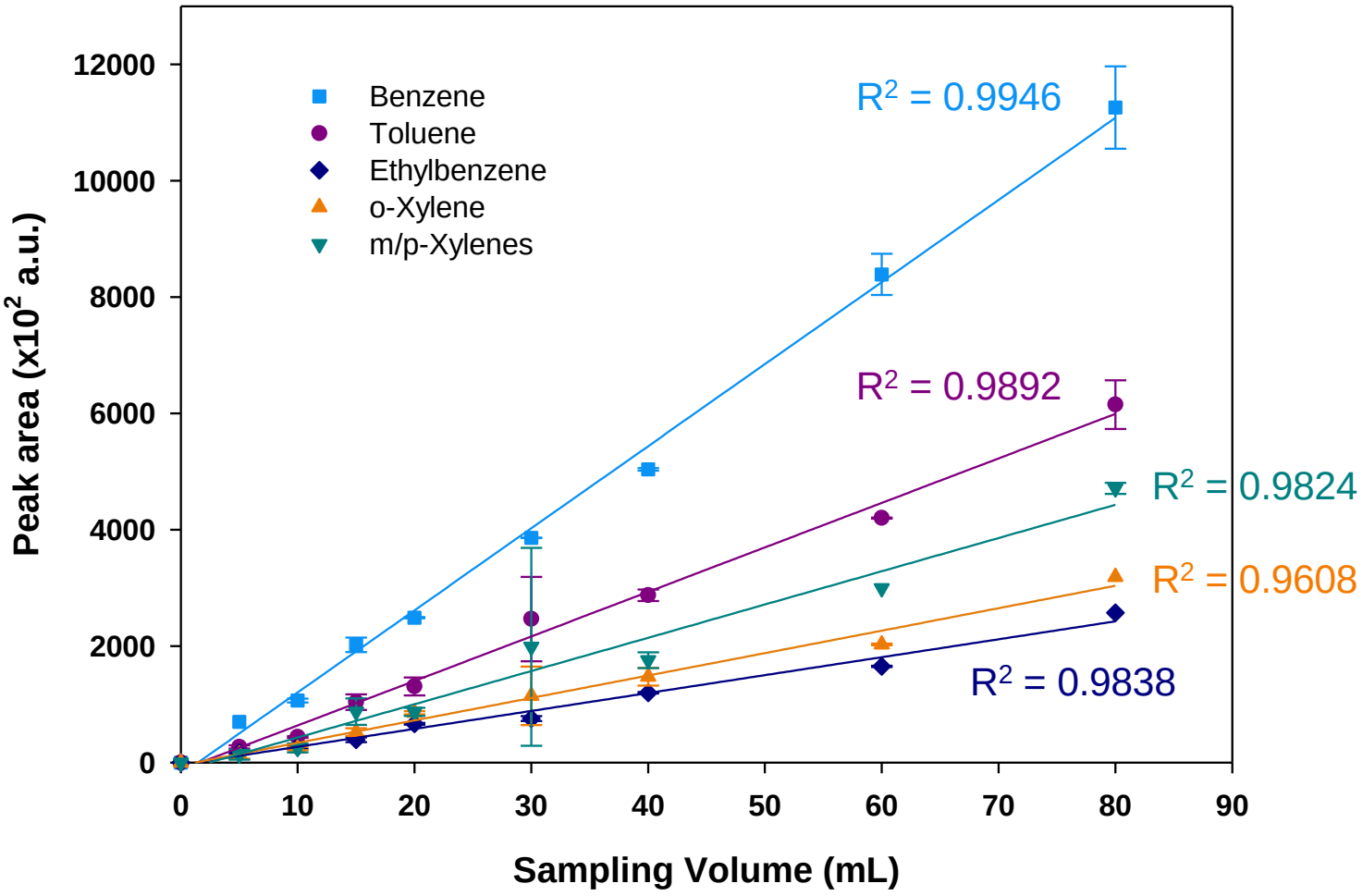
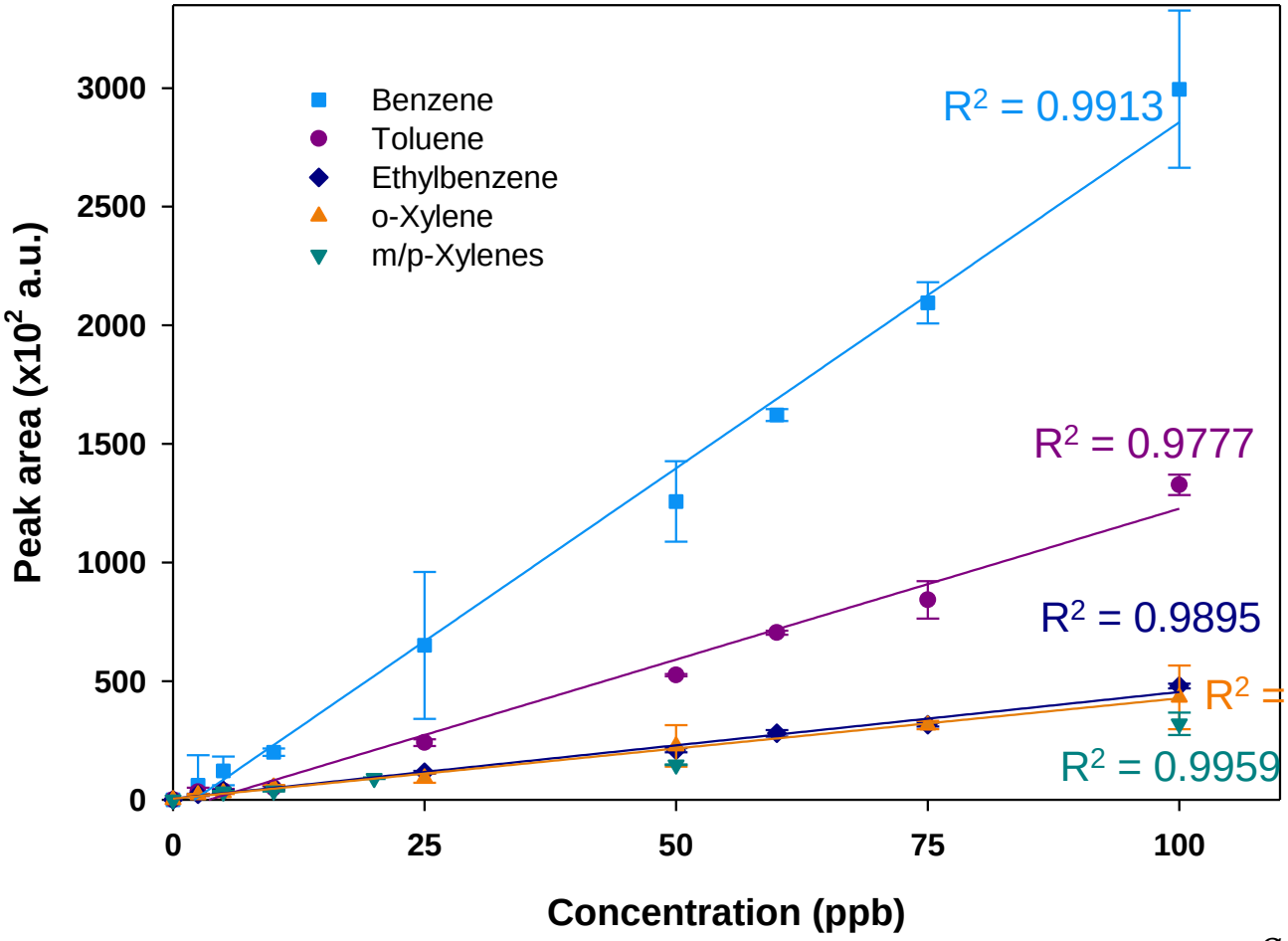


Concentration = 100 ppb

Lara-Ibeas et al. (2019), *Micromachines*, **10**, 187.

4. Results and discussion | *1st prototype of preconcentrator*

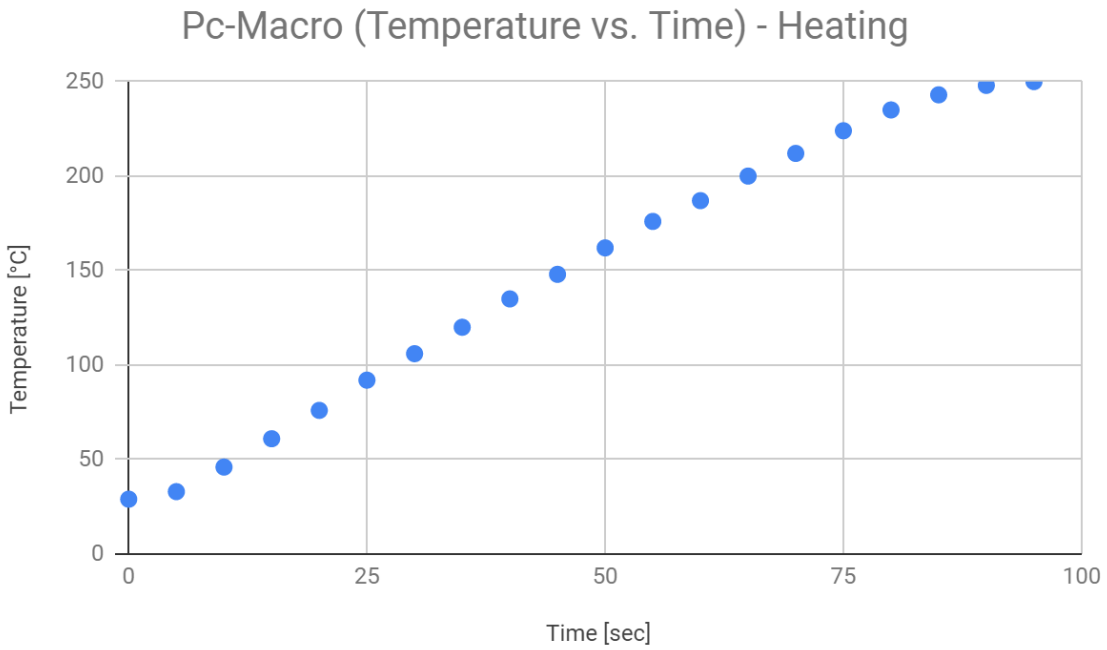


Compound	LOD (ppb)
Benzene	**0.20**
Toluene	**0.26**
Ethylbenzene	**0.49**
m/p-Xylenes	**0.80**
o-Xylene	**1.70**

Sampling volume = 20 mL

Lara-Ibeas et al. (2019), *Micromachines*, **10**, 187.

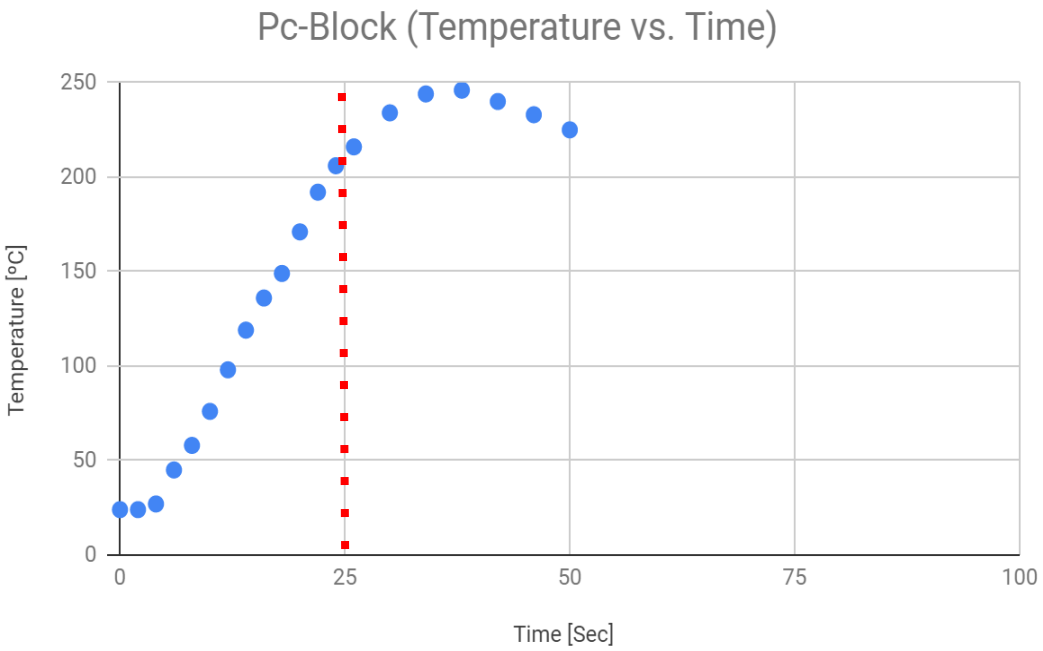
The first prototype of preconcentrator takes 65 seconds to rise the temperature up to 200 °C from room temperature (25°C). While the energy consumption remains constant at 210 Watts.



Heating	Time [Sec]	Temperature [°C]	Power[w]
	0	29	210
	5	33	210
	10	46	210
	15	61	210
	20	76	210
	25	92	210
	30	108	210
	35	120	210
	40	135	210
	45	148	210
	50	162	210
	55	176	210
	60	187	210
	65	200	210
	70	212	210
	75	224	210
	80	235	210
	85	243	210
	90	248	210
	95	250	210

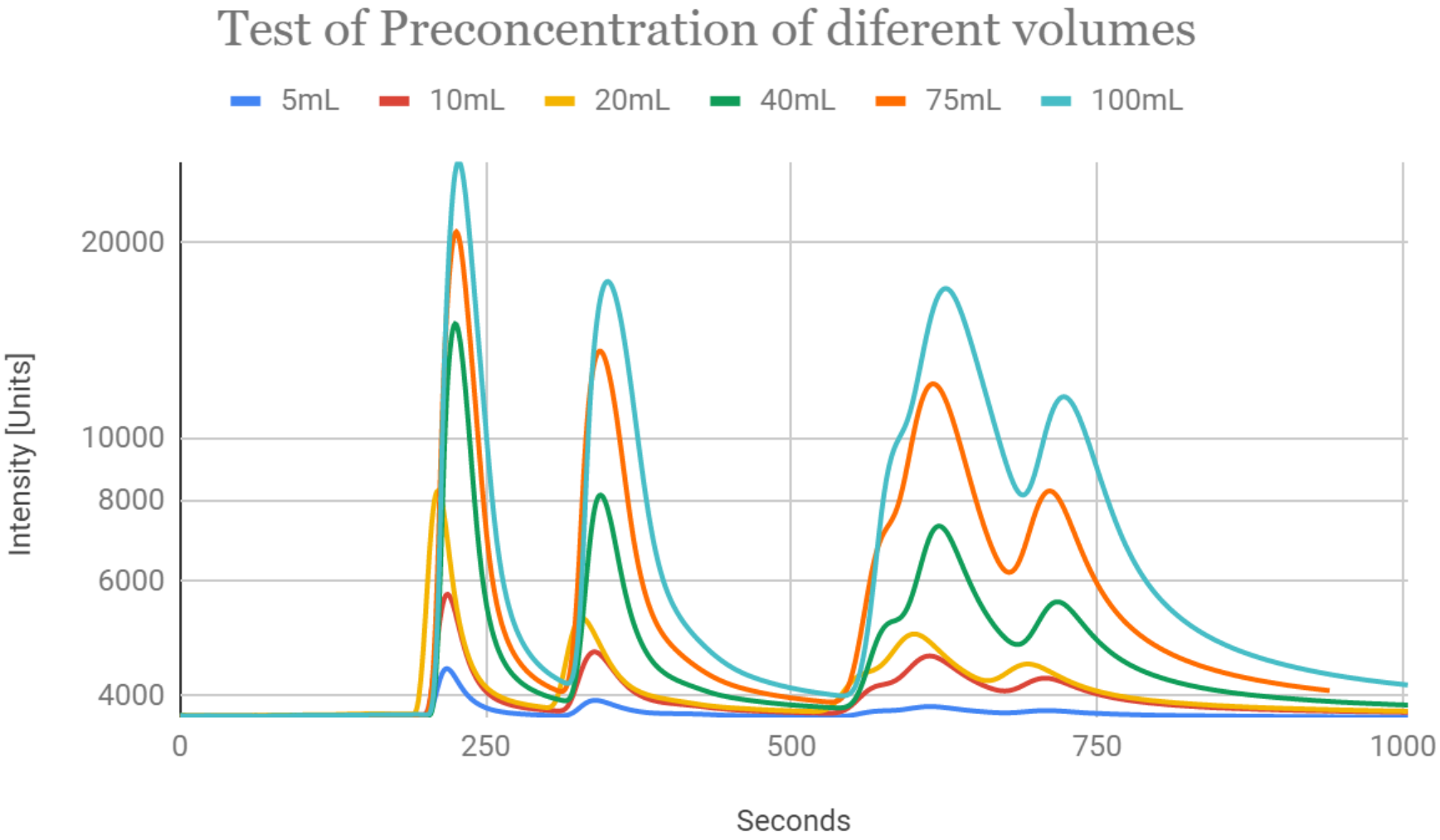
Rodriguez et al. (2019), *to be submitted.*

The second prototype of preconcentrator takes 23 seconds to rise the temperature up to 200 °C from room temperature (25°C). While, in this case the energy consumption varies from 42.9 Watts to 22.15 Watts .



Heating	Time [Sec]	Temperature [°C]	Voltage [Volt]	Current [Amp]	Power [Watt]
	0	24	14	3.06	42.9
	2	24	14	2.39	33.47
	4	27	14	2.28	31.89
	6	45	14	2.02	28.28
	8	58	14	1.92	26.89
	10	76	14	1.85	25.93
	12	98	14	1.79	25.09
	14	119	14	1.73	24.18
	16	136	14	1.69	23.60
	18	149	14	1.65	23.06
	20	171	14	1.61	22.54
	22	192	14	1.58	22.15
	24	**206**			
	26	216			
	30	234			
	34	244			
	38	246			
	42	240			
	46	233			
	50	225			

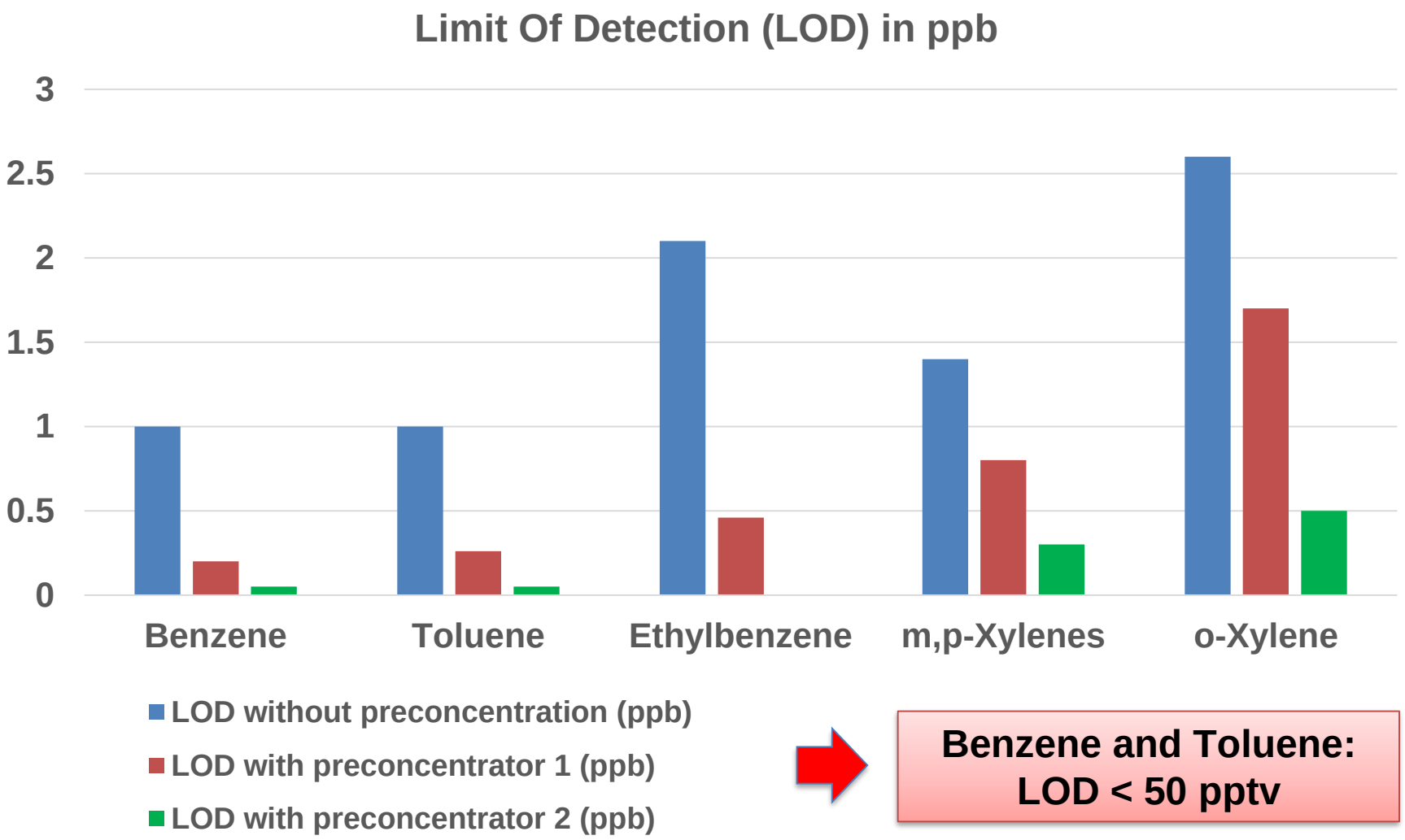
Rodriguez et al. (2019), *to be submitted.*



Rodriguez et al. (2019), *to be submitted*.

5. Conclusions

Comparison of sensitivity



Benzene and Toluene: LOD < 50 pptv

Nasreddine et al. (2016), *Sensors and Actuators B*, **224**, 159-169.

Lara-Ibeas et al. (2019), *Micromachines*, **10**, 187.

Rodriguez et al. (2019), *to be submitted*.

Acknowledgements

Financial supports

Smart In'Air
Monitoring