

# New approach to time-integrated diffusive sampling of environmental contaminants based on SPME

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## Introduction

Passive sampling is a well established methodology for the evaluation of occupational exposures to environmental volatile organic compounds (VOC's) (1)

Solid-phase microextraction (SPME) technique has demonstrated to be a reliable mean of sampling VOC's in air, as well as in biological fluids (2,3)

Currently, SPME is also being applied, as passive sampler, to determine the time-weighted average (TWA) exposure of VOC's at work atmospheres

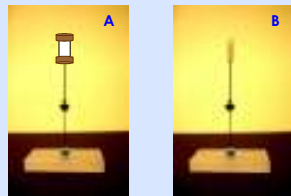
## Objectives

- Evaluate of a SPME passive sampler based on radial diffusion of the analytes to the sorbent
- Study the effect of concentration and time on the uptake rate
- Evaluate the performance of the diffusive sampler in short sampling time under fluctuating concentrations

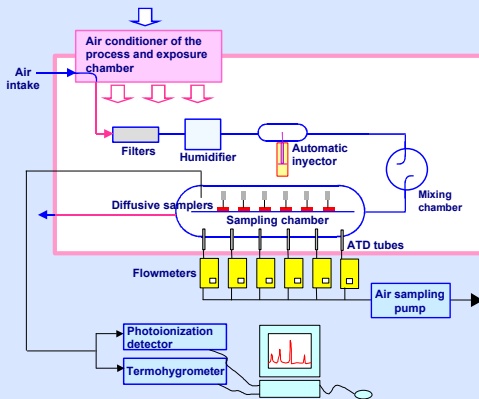
## Experimental

### Diffusive sampling device

- SPME fiber assembly, short, 23 GA, metal
- 80  $\mu\text{m}$  Carboxen/PDMS
- Diffusion cell: hydrophobic PE cylinder, 7-12  $\mu\text{m}$  porosity, 1.7 wall thickness, 17.4 mm length and capped with holed septa (A)
- The diffusion cell is replaced by a teflon cap after sampling (B)



### Evaluation of SPME-passive sampler performance



#### Determination of diffusive uptake rate

- Toluene concentration: 19 – 38  $\text{mg}/\text{m}^3$
- Exposure time: 15 – 60 min
- T: 20  $^{\circ}\text{C} \pm 2^{\circ}\text{C}$
- HR: 50 %  $\pm$  5%

- Atmosphere concentration checked by:
  - dynamic sampling with ATD tubes (Tenax)
  - Photoionization detector (PID) MiniRAE 2000

#### Fluctuating toluene concentration

Patterns:

- 10 min at 0.1 TLV, 5 min at TLV, 5 min at 0.5 TLV and 10 min at 0.1 TLV concentration
- 5 min at 0.01 TLV, 3 min at TLV, 10 min at 0 concentration and 3 min at TLV concentration

### Chromatographic analysis

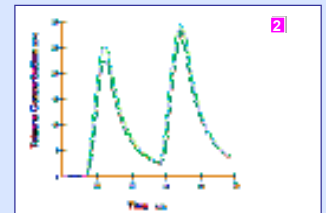
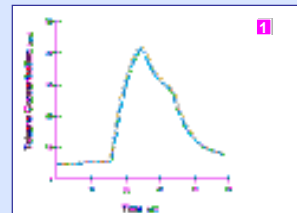
- HP 6890N chromatograph, splitless mode, FID
- SPME desorption at 280 $^{\circ}\text{C}$ , 2 min.
- HP-5, 30 m x 0.32 mm, 0.25  $\mu\text{m}$ , 35  $^{\circ}\text{C}$  hold for 2 min, 10  $^{\circ}\text{C}/\text{min}$  to 80  $^{\circ}\text{C}$ , hold 2 min.
- Carrier gas Helium at 1.2 ml/min

## Results

Concentration ( $\text{mg}/\text{m}^3$ )	Time (min)	U (mL/min)	
19.24	15	2.21	2.15
		2.07	2.04
		2.17	
19.00	60	2.22	2.28
		2.14	2.02
		2.10	2.16
36.02	15	2.11	2.00
		1.98	2.08
		2.02	2.09
38.02	30	1.98	2.28
		2.21	1.96
		2.02	2.15
38.01	30	2.06	2.25
		2.28	2.11
		2.16	2.15

Mean diffusive uptake rate,  $U = 2.12 \text{ mL}/\text{min}$   
Standard deviation = 0.0925

Intraday repeatability between 2.5 to 6.4%  
Interday repeatability 5.3%



Obtained concentration ( $\text{mg}/\text{m}^3$ ) (U= 2.12 mL/min)	
SPME-TWA	57.2
	64.1
	58.3
	55.4
	55.0
	56.8
Average	57.8
Standard deviation	3.3
Coef. of variation (%)	5.7
Theoretical	59.9
PID average	61.7
Active sampling	60.1

Obtained concentration ( $\text{mg}/\text{m}^3$ ) (U= 2.12 mL/min)	
SPME-TWA	34.3
	32.5
	32.6
	38.1
	33.8
	34.0
Average	34.2
Standard deviation	2.0
Coef. of variation (%)	6.0
Theoretical	36.3
PID average	34.9
Active sampling	33.7

No statistical significant effect both of concentration and sampling time on uptake rate were observed under the studied conditions

The results shows good response characteristics of the diffusive sampler under fluctuating vapour concentrations

The obtained results are very promising in order to use this new solid-phase microextraction device for diffusive monitoring of environmental contaminants

The developed method can be of special interest for assessing short-term exposures to environmental VOC's (4)

[1] C. Prado, I. Ibarra, J.F. Periago, J. Chromatogr. A, 778 (1997) 255

[2] C. Prado, P. Marín, J.F. Periago, J. Chromatogr. A, 1011 (2003) 125

[3] C. Prado, J. Garrido, J.F. Periago, J. Chromatogr. B, 804 (2004) 255

[4] Council Directive 98/24/EC on the protection of the health and safety of workers from the risks related to chemical agents at work.

### Acknowledgements

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