Development of a sampling and analysis method using SPE and GC-MS for the determination of unmetabolized styrene in urine

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Introduction

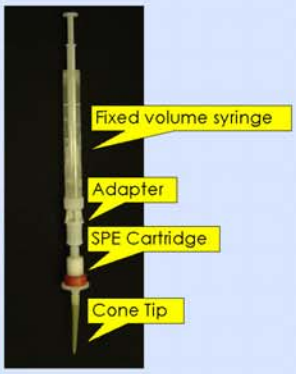
Biological monitoring of unmetabolized styrene in urine provides a useful method for determination of the individual's uptake of styrene [1]

In field studies, urine is generally sampled in glass or plastics containers, refrigerated, and delivered to the laboratory where samples are transferred to vials for analysis

Drawbacks
- Difficulty for collection, transport and storage of samples
- Losses of the volatile analytes of interest
- Analysis must be carried out as soon as possible

A new sampling system is being developed to overcome the drawbacks [2]

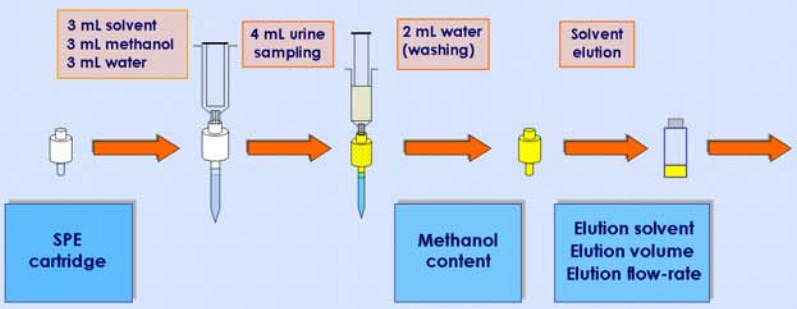
The system combines sampling, transportation and preservation of biological fluids. It consists on a special syringe joined to an SPE cartridge by means an adapter

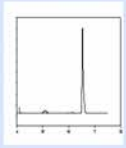


Objectives

- Development of a sampling and analysis method for the determination of styrene in urine
- Estimation of the variables that could affect the SPE extraction efficiency

Experimental





1 µL of extract injected into GC/MS

CG HP6890 - MSD HP5973
Column: HP1-MS 50mx0.25mmx1µm
Carrier gas: helium, 1.2 mL/min
Injection: splitless, 1µl sample
Inlet temperature: 250 °C
Oven temperature: 120 °C
Adquisition mode: SIM. M/z 104

Experimental design

Half fraction screening design
Experimental factors: 5
Number of runs: 32
Replicated design: 1
Randomized: Yes
Response: Recovery (%)

Factors	Low	High
Sorbent	C18	OASIS
Eluting Solvent	EA	DCM
Elution volume (ml)	1.0	4.0
Elution flow rate (ml/min)	0.5	4.0
Methanol fraction (%)	0	5

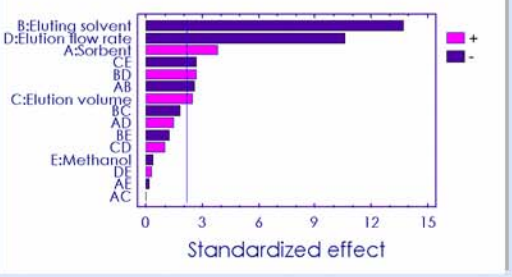
EA: Ethyl acetate / DCM: Dichloromethane

Results and conclusions

Fractional factorial design matrix and results of screening experiment

Sorbent	Eluting solvent	Elution vol (mL)	Elution flow (mL/min)	Methanol (%)	Recovery (%)	
OASIS	DCM	1	0.5	5	54.3	58.2
C18	DCM	1	0.5	0	59.5	50.5
OASIS	DCM	1	4.0	0	33.3	45.6
OASIS	EA	4	0.5	5	90.0	97.8
C18	EA	4	0.5	0	93.2	83.3
C18	EA	1	0.5	5	82.8	83.6
OASIS	EA	4	4.0	0	74.8	77.7
OASIS	DCM	4	0.5	0	54.8	64.3
C18	EA	1	4.0	0	43.2	46.3
C18	DCM	4	4.0	0	40.9	43.8
OASIS	EA	1	0.5	0	89.8	83.3
C18	DCM	1	4.0	5	41.9	31.8

Standardized Pareto Chart for Recovery



Repeatability

Spiked urine samples: 174.7µg/L
Sorbent: OASIS
Eluent : 1.5 mL of ethyl acetate
Flow-rate: 0.5 mL/min

	µg/L extracted urinary styrene	Recovery %
	145.0	83.0
	145.2	83.1
	154.7	88.5
	145.6	83.4
	173.9	99.5
Mean	152.9	87.5
RSD%	8.1	8.1

Conclusions

- The eluting solvent and the elution flow-rate are the more significant factors affecting the styrene extraction from urine
- The use of ethyl acetate and the OASIS sorbent improves the efficiency of styrene extraction
- The increase of the elution flow-rate has a negative influence on the response
- The presence of methanol in washing solvent has no effect on the styrene recovery
- The increase in the elution volume has a minor positive effect, however low elution volumes allows the enrichment of the styrene concentration in the eluate
- The obtained results indicate that this methodology could be satisfactorily used for biomonitoring of styrene in urine

[1] Ibarra, I.; PhD Thesis. Universidad de Murcia. 2002.
[2] Simon P. et al. Sixth International Symposium on Biological Monitoring in Occupational and Environmental Health, Heidelberg 6-8 September 2004