Determination of ketones in urine by SPME for biological monitoring application

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Introduction

- The solid-phase microextraction technique (SPME) can be very useful in biological control of organic solvents exposure by determining the concentration of unmetabolised solvent either in exhaled air or in urine.
- Compounds such as acetone (AC), methyl ethyl ketone (MEK) and methyl isobutyl ketone (MIBK), are components of lacquers and paints, as well as solvents for these products, and it is common to find them in work environments.
- Biological limit values have been established for the unmetabolized ketones in urine specimen [1]. So, it is interesting to develop methods that provide routine biological control for these compounds.
- The effect of variables such as fiber type, incubation time, temperature and time of extraction and the addition of salt on the ketone extraction have been previously evaluated [2]. The selected conditions were: Carboxen (75 μm), 1 min of incubation time, 50 ºC of extraction temperature, extraction time of 20 min, and urine saturated with salt.

The aim of this work was to develop a method, using SPME, that allows the simultaneous determination of urinary AC, MEK, and MIBK since coexposure to mixtures of these substances is frequent in occupational environments.

Experimental

Urine samples

1

Sample volume	2.5 mL
Equilibrium time	1 min
Equilibrium temperature	50ºC
NaCl	0.9 g

Extraction

2

Fibre 75 μm Carboxen/PDMS	
Headspace	
Stirred	
Extraction temperature	50 ºC
Extraction time	20 min

Desorption

Desorption temperature	300ºC
Desorption time	4 min
Inlet liner	0.75 mm

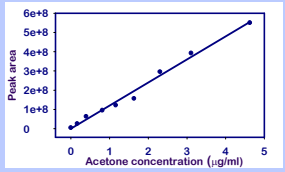
Analysis

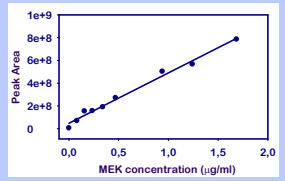
3

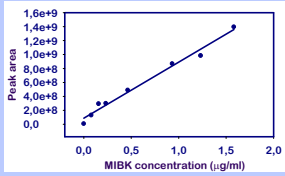
CG HP 6890A – MSD HP5973
He, 1.4 mL/min
HP-1 60m x 0.25mm x 1μm
Splitless
Oven: 4 min at 40ºC, to 140ºC at 20ºC/min and held for 2 min
SIM mode detecting *m/z* 43, 58 for AC
m/z 43, 72, and 57 for MEK
m/z 58, 85, and 100 for MIBK

Results and discussion

Calibration curves, limits of detection and precision of the HS-SPME method



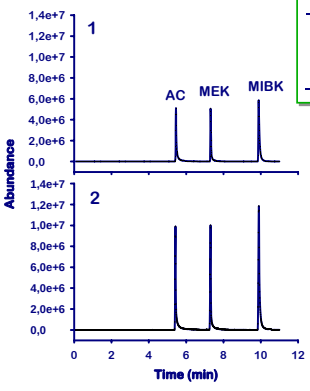




Parameters obtained from the calibration curves

Compound	Correlation coefficient	LOD (μg/ml)
AC	0.995	0.32
MEK	0.994	0.15
MIBK	0.992	0.19

- There is a linear relationship between the amount of analyte extracted by the fiber and the urinary concentration
- The precision of the method was between 3 and 11%
- Under the studied conditions, the limits of detection were low enough to quantify the three ketones at occupational levels



Concentation (μg/ml)	1	2
AC	1.17	3.95
MEK	0.24	0.80
MIBK	0.23	0.80

Intra-day precision (% RSD), n=6

Concentration level	RSD (%)		
	AC	MEK	MIBK
1	10.6	9.3	9.3
2	9.1	2.6	4.3

Mass chromatograms of m/z 43, 58 for AC
m/z 43, 72, and 57 for MEK
m/z 58, 85, and 100 for MIBK at 1 and 2 concentration levels.

The developed method is suitable to quantify ketones in urine at occupationally exposed levels.

The method can be used on a routine basis for biomonitoring of acetone, methylethyl ketone and methyl isobutyl ketone.

References

1. Límites de exposición profesional para Agentes Químicos en España. 2008. Instituto Nacional de Seguridad e Higiene en el Trabajo. INSHT
2. C. Prado, P. Marín, J. Alcaraz, J.F. Periago. XVII Congreso Español de Toxicología. Santiago de Compostela 2007.