# DETERMINATION OF NITRO MUSKS IN ENVIRONMENTAL WATERS BY STIR BAR SORPTIVE DISPERSIVE MICROEXTRACTION FOLLOWED BY THERMAL DESORPTION-GAS CHROMATOGRAPHY-MASS SPECTROMETRY


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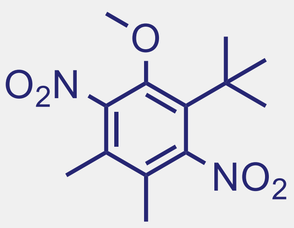
## INTRODUCTION

### AIM

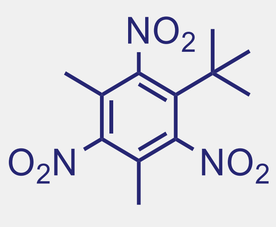
In this work, an analytical method for the determination of the complete family of nitro musks in environmental waters is presented. It is the first time that nitro musks are determined by using magnetic materials

Despite their pleasant aroma, **nitro musks** have shown health risks related to dermatitis, carcinogenic effects and endocrine disruption. They are indirectly released into the environment via wastewater treatments plants, or directly from swimming activities

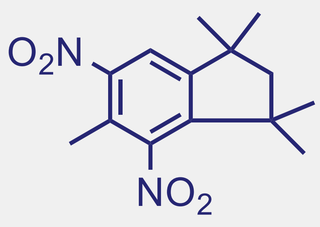
**Stir bar sorptive dispersive microextraction (SBSDME)** [1] is a hybrid microextraction approach (stir bar sorptive extraction (SBSE) plus dispersive solid phase extraction (DSPE)) where a **magnetic material** used as extraction phase is dispersed by magnetic stirring and retrieved onto the stir bar surface due to its magnetic properties



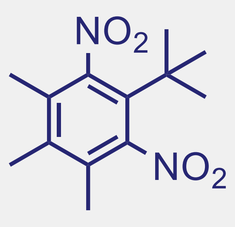
**Musk ambrette (MA)**



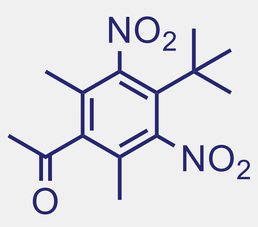
**Musk xylene (MX)**



**Musk moskene (MM)**



**Musk tibetene (MT)**



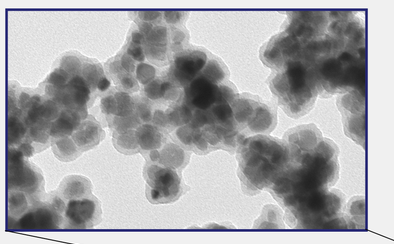
**Musk ketone (MK)**

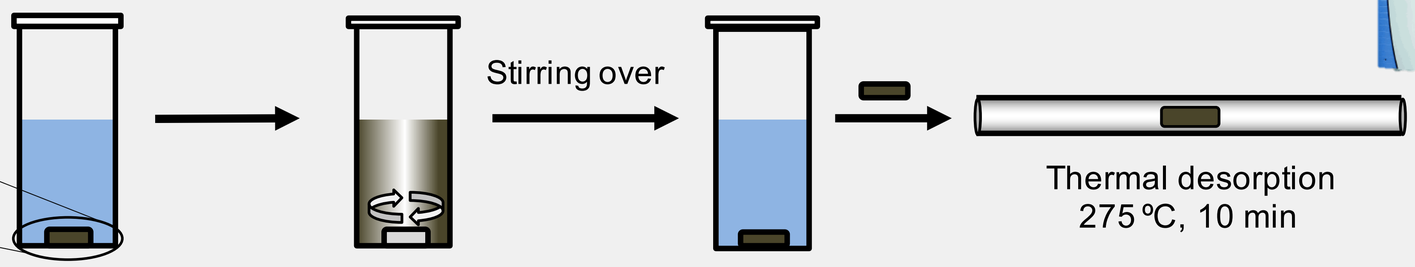
Cobalt ferrite magnetic nanoparticles coated with polydopamine (**$CoFe_2O_4$@pDA MNPs**) were used as extraction material

The analytes are desorbed by **thermal desorption**, as an effective and solvent-free desorption step, and subsequently subjected to GC-MS

## EXPERIMENTAL

TEM image of $CoFe_2O_4$@pDA MNPs






**GC-MS**

### GC-MS conditions

- Inlet temperature: 280 °C
- Transfer line temperature: 280 °C
- Ion source temperature: 250 °C
- Helium flow rate: 1 mL $min^{-1}$
- Column: HP-5MS Ultra Inert (30 m x 0.25 mm, 0.25 µm)

## RESULTS AND DISCUSSION

### Figures of merit of the proposed method

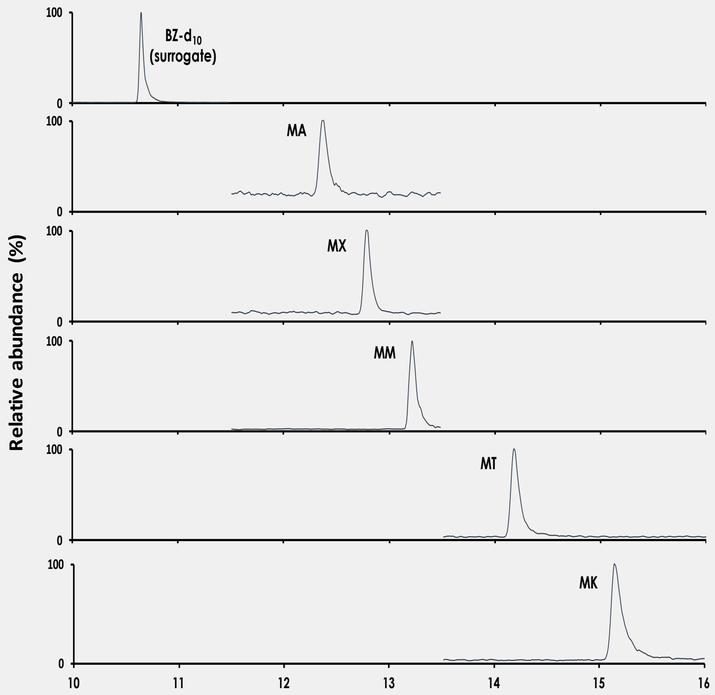
| Nitro musk | Regression coeficient, $R^2$ | MLOD (ng $L^{-1}$) | MLOQ (ng $L^{-1}$) | Enrichment factor | Repeatability (% RSD) | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Intra-day | | Inter-day | |
| | | | | | 20 ng $L^{-1}$ | 40 ng $L^{-1}$ | 20 ng $L^{-1}$ | 40 ng $L^{-1}$ |
| **MA** | 0.997 | 2.8 | 0.5 | 284 ± 10 | 3.9 | 4.5 | 9.7 | 10.2 |
| **MX** | 0.9995 | 1.9 | 6.5 | 178 ± 10 | 4.8 | 5.4 | 8.6 | 9.6 |
| **MM** | 0.998 | 0.1 | 0.5 | 640 ± 70 | 3.5 | 3.7 | 7.8 | 12.3 |
| **MT** | 0.9990 | 0.9 | 3.0 | 303 ± 30 | 5.8 | 6.6 | 9.8 | 11.5 |
| **MX** | 0.995 | 0.3 | 1.0 | 378 ± 30 | 5.3 | 5.9 | 10.5 | 10.1 |

- High level of **linearity**, that reached at least 100 ng mL-1, was obtained for all compounds
- Low **limits of detection** (0.1 - 2.8 ng L-1) and good values of **precision** (< 11 %) and **enrichment factors** (178 - 640) were achieved

### Relative recoveries (%)

| Nitro musk | Relative recovery values (%) | | | |
|---|---|---|---|---|
| | Sea water | | River water | |
| | 20 ng $L^{-1}$ | 40 ng $L^{-1}$ | 20 ng $L^{-1}$ | 40 ng $L^{-1}$ |
| **MA** | 102 ± 10 | 114 ± 6 | 111 ± 10 | 101 ± 9 |
| **MX** | 106 ± 10 | 102 ± 6 | 101 ± 5 | 96 ± 7 |
| **MM** | 116 ± 10 | 97 ± 10 | 101 ± 3 | 109 ± 10 |
| **MT** | 98 ± 2 | 99 ± 8 | 108 ± 6 | 110 ± 4 |
| **MK** | 110 ± 10 | 112 ± 10 | 113 ± 10 | 93 ± 10 |

The results of the **recovery studies** (80 - 116 %) at two concentration levels show that the matrices under consideration do not significantly affect the SBSDME approach




**Chromatogram of a standard solution containing the target analytes at 100 ng $L^{-1}$ obtained by the developed method**

## CONCLUSIONS

- The proposed SBSDME-TD-GC-MS method contributes to the development of sensitive methods for the determination of nitro musks in environmental waters at trace levels
- The work expands the analytical potential of SBSDME to other analytes


[1] J.L. Benedé, A. Chisvert, D.L. Giokas, A. Salvador, J. Chromatography A 1362 (2014) 25-33



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Consulta otras comunicaciones presentadas por el GICAPC en la **XXII Reunión de la Sociedad Española de Química Analítica:**

*Determination of nitro musks in environmental waters by stir bar sorptive dispersive microextraction followed by thermal desorption-gas chromatography-mass spectrometry*. J.L. Benedé, A. Chisvert, A. Salvador. Flash Communication. Ver comunicación.

*Stir bar sorptive-dispersive microextraction mediated by a magnetic nanoparticles-metal organic framework composite for the determination of n-nitrosamines in cosmetic products*. P. Miralles, I. Van Gemert, A. Chisvert, A. Salvador. Flash Communication. Ver comunicación.

*Development of an analytical method for the determination of acrylamide in cosmetic products based on dispersive liquid-liquid microextraction.* L. Schettino, J.L. Benedé, A. Chisvert, A. Salvador. Flash Communication. Ver comunicación.

*Determination of hydroxylated ingredients with preservative activity in cosmetic products by gas chromatography-mass spectrometry*. C. Azorín, J.L. Benedé, A. Chisvert, A. Salvador. Ver comunicación.

*A green analytical method for the determination of hydroxyethoxyphenyl butanone in cosmetic products*. P. Miralles, J.L. Benedé, A. Mata-Martín, A. Chisvert, A. Salvador. Ver comunicación.

*Determination of polycyclic aromatic hydrocarbons in cosmetics by stir bar sorptive dispersive microextraction and gas chromatography-mass spectrometry*. Vállez-Gomis, J. Grau, J.L. Benedé, A. Chisvert, A. Salvador. Ver comunicación.

*Reversed-phase dispersive liquid-liquid microextraction prior to liquid chromatography-tandem mass spectrometry for the determination of acrylamide in cosmetic products*. L. Fernández, J.L. Benedé, A. Chisvert, A. Salvador. Ver comunicación.

*Development of dispersive liquid-solid microextraction: application to the determination of cortisone and cortisol in human saliva*. J. Grau, J.L. Benedé, A. Chisvert, A. Salvador. Ver comunicación.