TÉCNICAS MINIATURIZADAS Y DE MICROEXTRACCIÓN. APLICACIÓN EN MUESTRAS AMBIENTALES, ALIMENTARIAS Y PRODUCTOS DE CUIDADO PERSONAL

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LIDSA Laboratorio de Investigación y Desarrollo de Soluciones Analíticas

USC UNIVERSIDADE DE SANTIAGO DE COMPOSTELA

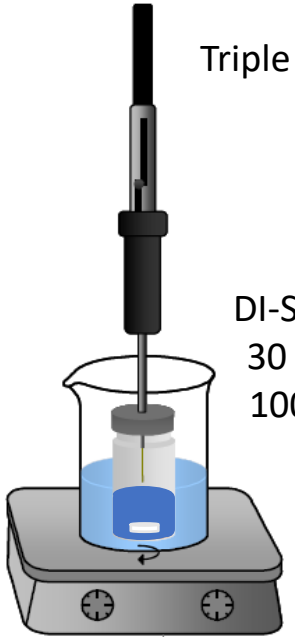
CRETUS Cross-Research in Environmental Technologies

1. Solid-phase microextraction (SPME). Applications

SAMPLE PREPARATION: WATER. DERIVATIZATION. COSMETICS.

María Llompart, María Celeiro

10 mL water
100 mg K_2CO_3
200 µL acetic anhydride
15 min, 100 °C



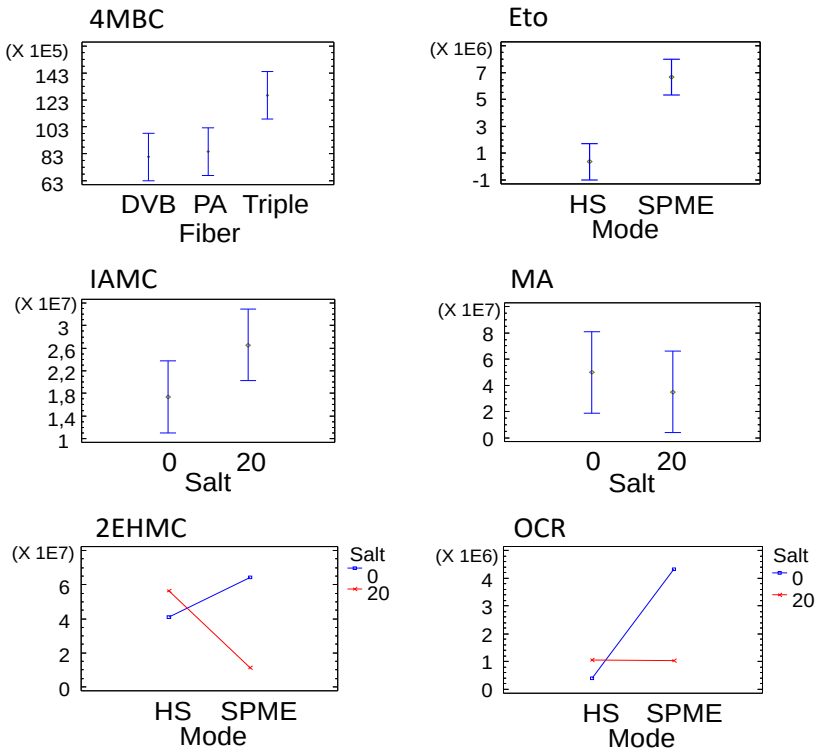
DI-SPME
30 min
100 °C

Thermal desorption (290°C)
GC-MS/MS analysis

Experimental design

- Fibre:
  PDMS/DVB
  Triple
  PA
- Mode:
  Headspace
  Direct
- Salt (%, w/v):
  0
  20

Categorical multi-factorial design ($3x2^2$)



Selected conditions

- ✓ **Triple fibre**
- ✓ **Direct immersion**
- ✓ **No salt addition**

ELSEVIER
Journal of Hazardous Materials
Volume 323, Part A, 5 February 2017, Pages 45-55

Simultaneous *in-vial* acetylation solid-phase microextraction followed by gas chromatography tandem mass spectrometry for the analysis of multiclass organic UV filters in water

Marlene Vila [a], Maria Celeiro [a], J. Pablo Lamas [a], Carmen Garcia-Jares [a], Thierry Dagnac [b], Maria Llompart [a]

ELSEVIER
Analytica Chimica Acta
Volume 1203, 22 April 2022, 339650

Development of a solid phase microextraction gas chromatography tandem mass spectrometry methodology for the analysis of sixty personal care products in hydroalcoholic gels - hand sanitizers - in the context of COVID-19 pandemic

Lua Vazquez [a], Maria Celeiro [a], Ana Castiñeira-Landeira [a], Thierry Dagnac [b], Maria Llompart [a]

M. Vila et al. J. Hazard. Mater. 323 (2017), 45.

1. Solid-phase microextraction (SPME). Applications

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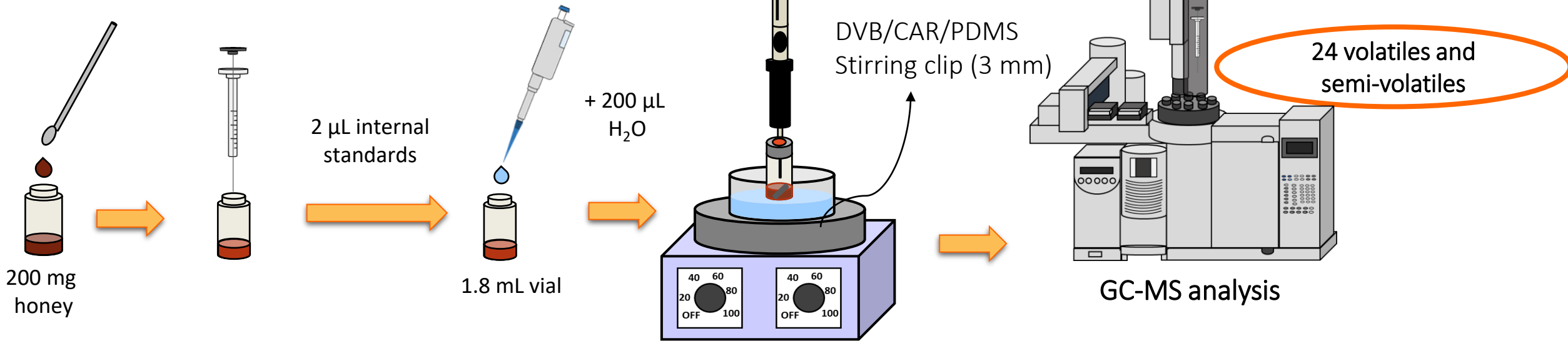
Miniaturization

Mini-SPME. Experimental procedure



Sustainable Chemistry and Pharmacy 21 (2021) 100451

Contents lists available at ScienceDirect

Sustainable Chemistry and Pharmacy

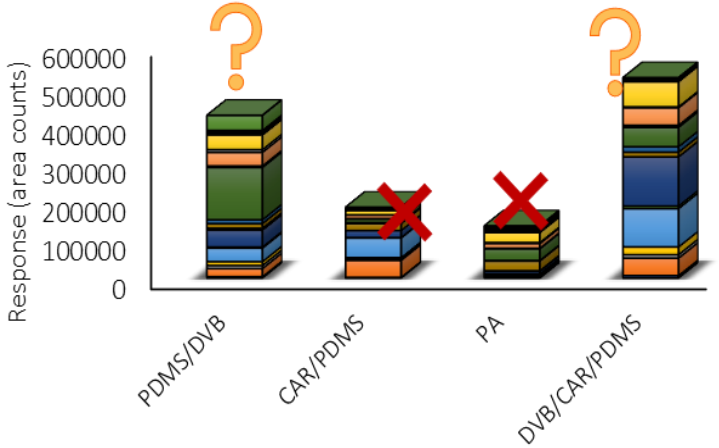
journal homepage: www.elsevier.com/locate/scp

Optimization of a miniaturized solid-phase microextraction method followed by gas chromatography mass spectrometry for the determination of twenty four volatile and semivolatile compounds in honey from Galicia (NW Spain) and foreign countries

Lua Vazquez [a], Maria Celeiro [a], Meruyert Sergazina [a,b], Thierry Dagnac [c], Maria Llompart [a,*]

HS-SPME mode
100 ºC
2 + 30 min

Fibre coating



- Dilution 1:10 (w/v)
- 1.2 g honey
- 30 min
- 60 ºC

2. Ultrasound-assisted emulsification microextraction (USAEME)

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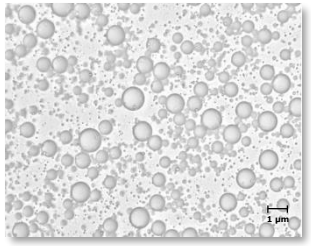
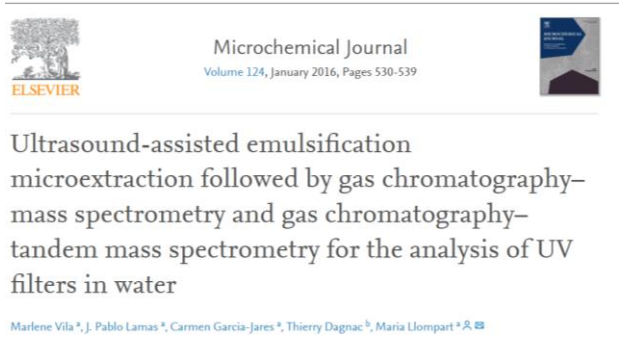
➢ **Emulsión: sistema heterogéneo formado por dos líquidos inmiscibles, en el que uno de ellos se encuentra disperso en el otro**




Microchemical Journal
Volume 124, January 2016, Pages 530-539
ELSEVIER
Ultrasound-assisted emulsification microextraction followed by gas chromatography–mass spectrometry and gas chromatography–tandem mass spectrometry for the analysis of UV filters in water
Marlene Vila [a], J. Pablo Lamas [a], Carmen Garcia-Jares [a], Thierry Dagnac [b], Maria Llompart [a]

Microextracción líquido-líquido (LLME) + **Ultrasonidos (US)** → **Emulsificación** → **USAEME**

✓ ↑↑ **Área de contacto**

✓ ↑ **Transferencia de masa**

PROCEDIMIENTO USAEME

10 mL water

100 µL of solvent

Ultrasounds
25 ˚C
5 min

Centrifugation
3500 rpm
10 min

Microdrop

3. Fabric-phase sorptive extraction (FPSE)

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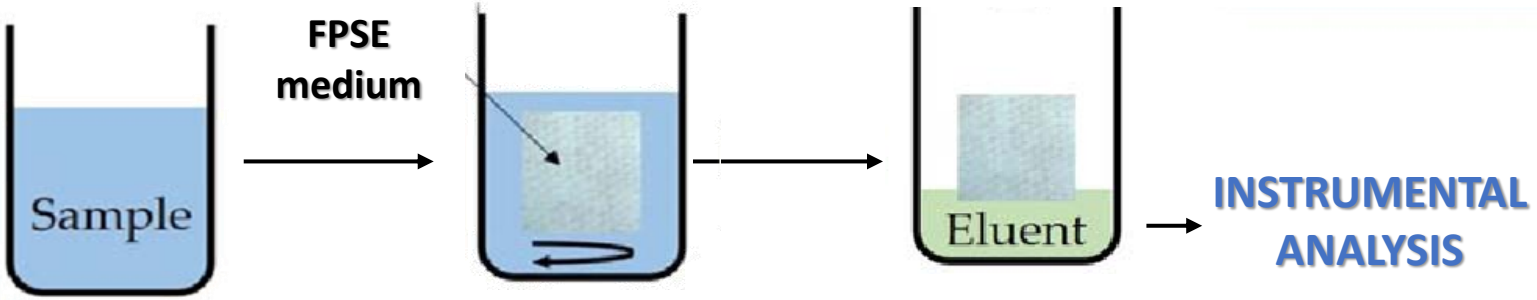


EXTRACTION STEP

Analytes are retained on the surface and also penetrate the interior

DESORTION STEP

Analytes are desorbed from the FPSE medium to an adequate solvent (few mL)

Analytica Chimica Acta: X 4 (2020) 100038

Contents lists available at ScienceDirect

Analytica Chimica Acta: X

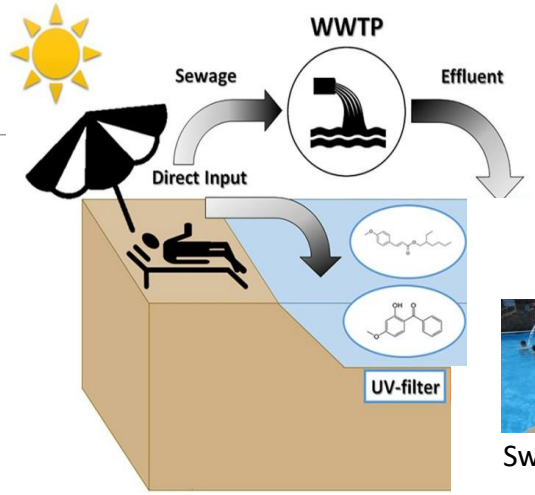
journal homepage: www.journals.elsevier.com/analytica-chimica-acta-x

Development of an analytical methodology based on fabric phase sorptive extraction followed by gas chromatography-tandem mass spectrometry to determine UV filters in environmental and recreational waters

Maria Celeiro [a], Ruben Acerbi [a], Abuzar Kabir [b], Kenneth G. Furton [b], Maria Llompart [a, *]

[a] CRETUS Institute, Department of Analytical Chemistry, Nutrition and Food Science, Faculty of Chemistry, Universidade de Santiago de Compostela, E-15782, Santiago de Compostela, Spain

[b] International Forensic Research Institute, Department of Chemistry and Biochemistry, Florida International University, Miami, FL-33199, USA



River

Sea

Swimming pool

Aquapark

Bathing areas

4. Micro matrix solid-phase dispersion (µMSPD)

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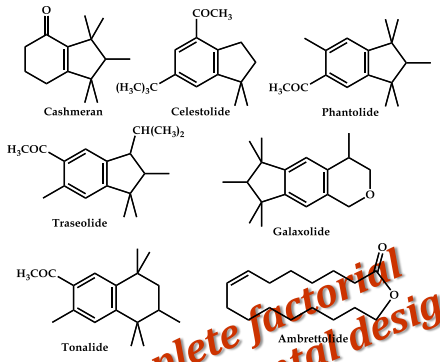
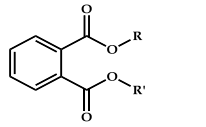
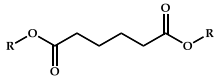
micro-MSPD

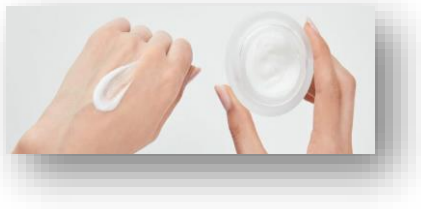




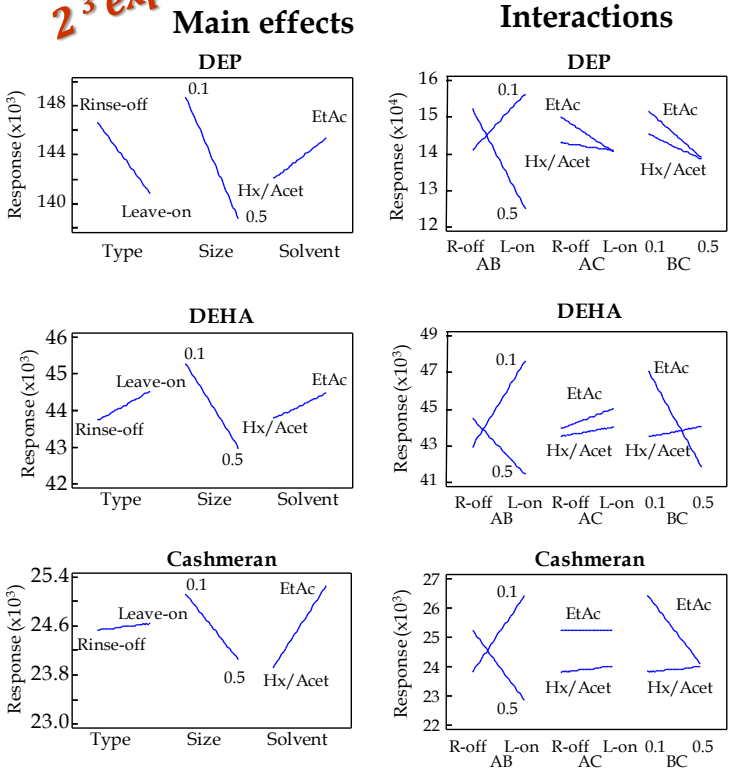
Phthalates



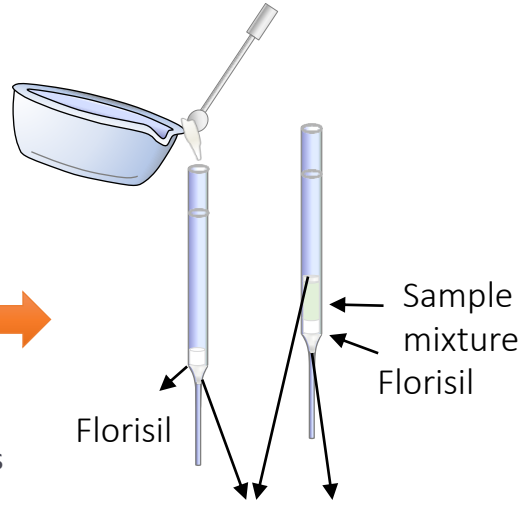
Adipates

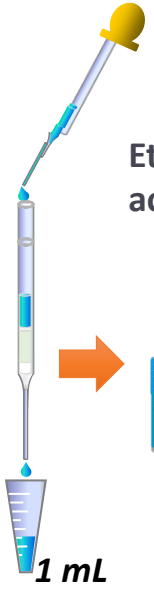


Complete factorial 2^3 experimental design



0.1 g sample + Florisil+ sodium sulphate anhydrous





GC-MS/MS

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Journal of Chromatography A
Volume 1293, 7 June 2013, Pages 10-19

Analysis of plasticizers and synthetic musks in cosmetic and personal care products by matrix solid-phase dispersion gas chromatography–mass spectrometry ☆

Maria Llompart, Maria Celeiro, J. Pablo Lamas, Lucia Sanchez-Prado, Marta Lores, Carmen Garcia-Jares

-Avoid plastic material ➔ Glass material ➔ Wash carefully

-Keep the material protected and baked at 230°C before their use

- Minimize extraction steps