

# **Matrix Solid-Phase Dispersion Procedure for Determination of Antibiotics and Metabolites in Mussels: Application in Exposure Bioassays**

**Carmen Mejías <sup>1</sup>, Tainá G. Fonseca <sup>2</sup>, Noelia García-Criado <sup>1</sup>, Julia Martín <sup>1</sup>, Juan Luis Santos <sup>1</sup>, Irene Aparicio <sup>1</sup> and Esteban Alonso <sup>1,\*</sup>**

<sup>1</sup> Departamento de Química Analítica, Escuela Politécnica Superior, Universidad de Sevilla, C/Virgen de África, 7, E-41011 Seville, Spain; cmpadilla@us.es (C.M.); ngarcia5@us.es (N.G.-C.); jbueno@us.es (J.M.); jlsantos@us.es (J.L.S.); iaparicio@us.es (I.A.)

<sup>2</sup> Centre for Marine and Environmental Research—CIMA/ARNET—Infrastructure Network in Aquatic Research, University of Algarve, Campus de Gambelas, 8000-139 Faro, Portugal; tgfonseca@ualg.pt

\* Correspondence: ealonso@us.es; Tel.: +34-954-55-28-58

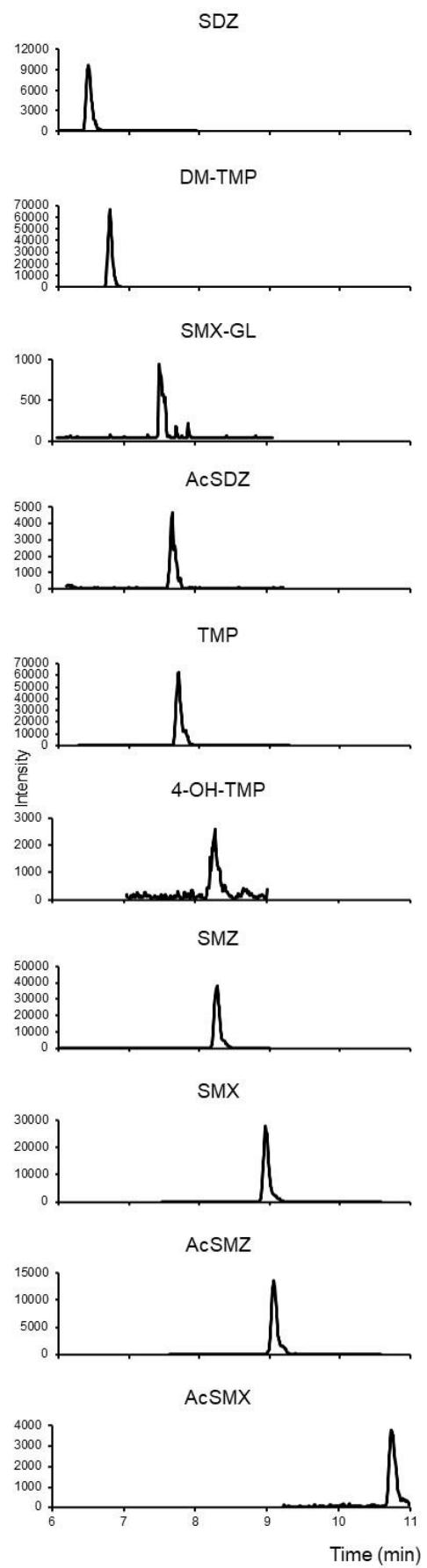
**Table S1.** Box–Behnken design matrix for the optimisation of clean-up sorbent amount.

Experiment	C18 amount (g)	PSA amount (g)	Florisil® amount (g)
1	0.8	0.4	0.8
2	0.8	0	0.4
3	0.4	0.4	0.4
4	0.4	0.8	0
5	0	0.8	0.4
6	0	0.4	0
7	0.8	0.8	0.4
8	0.4	0	0
9	0	0	0.4
10	0.4	0.4	0.4
11	0.4	0	0.8
12	0.4	0.8	0.8
13	0.4	0.4	0.4
14	0	0.4	0.8
15	0.8	0.4	0

**Table S2.** Method application to mussels (expressed as ng g<sup>-1</sup> dm).

Compound	Mussels			
	S <sub>F</sub>	S <sub>0</sub>	S <sub>14</sub>	S <sub>28</sub>
<b>TMP</b>	-	-	62.3	77.5
4-OH-TMP	-	-	-	-
DM-TMP	-	-	-	-
<b>SMX</b>	-	-	1.17	1.26
AcSMX	-	-	-	-
SMX-GL	-	-	-	-
<b>SDZ</b>	-	-	-	-
AcSDZ	-	-	-	-
<b>SMZ</b>	-	-	-	-
AcSMZ	-	-	-	-

-: lower than MDL; Parent compounds are marked in bold; S<sub>F</sub>: field sample; S<sub>0</sub>: sample at time 0 (after acclimatisation period, before spiking); S<sub>14</sub>: sample at time 14 days of exposure to SMX and TMP (1 µg L<sup>-1</sup>); S<sub>28</sub>: sample at time 28 days of exposure to SMX and TMP (1 µg L<sup>-1</sup>).



**Figure S1.** LC-MS/MS chromatogram of a 10 ng g<sup>-1</sup> d.m. spiked mussel sample.