

10.1071/EN18030_AC

©CSIRO 2018

Environmental Chemistry 2018, 15(6), 362-371

SUPPLEMENTARY MATERIAL

Environmental chiral analysis of β -blockers: evaluation of different n-alkyl-modified SBA-15 mesoporous silicas as sorbents in solid phase extraction

Mariana Silva,^A Sonia Morante-Zarcelero,^A Damián Pérez-Quintanilla,^A María Luisa Marina^B and Isabel Sierra^{A,C}

^ADepartamento de Tecnología Química y Ambiental, E.S.C.E.T, Universidad Rey Juan Carlos, C/ Tulipán s/n, 28933 Móstoles, Madrid, Spain.

^BDepartamento de Química Analítica, Química Física e Ingeniería Química, Facultad de Biología, Ciencias Ambientales y Química, Universidad de Alcalá, Ctra. Madrid-Barcelona Km 33.600, 28871 Alcalá de Henares, Madrid, Spain.

^CCorresponding author. Email: isabel.sierra@urjc.es

Table S1. Comparative study of reported methods for the SPE extraction of β -blockers from waters

Ace: Acebutolol, Al: Alprenolol, Ate: Atenolol, Bet: Betaxolol, Bis: Bisoprolol, Car: Carazolol, Cel: Celiprolol, Lab: Labetalol, Met: Metoprolol, Nad: Nadolol, Neb: Nebivolol, Ox: Oxprenolol, Pin: Pindolol, Prop: Propranolol, Sot: Sotalol, PF: preconcentration factor

β -blockers analysed	SPE sorbent	Sample	Recovery (%)	PF	Method	Comments	Ref.
Ace, Ate, Nad, Met, Prop	Strata X-C [®] (200 mg)	Tap water (250 mL)	67-125	83	CG-MS	Water sample adjusted at pH: 3. Not chiral analysis.	Caban et al. (2015)
Ate, Prop, Met	Oasis MCX [®] (60 mg)	River water (1000 mL)	60-110	2000	UPLC–MS/MS	Water sample adjusted at pH: 2.5. Not chiral analysis. Internal standard calibration.	Kasprzyk-Hordern et al. (2007)
Ace, Al, Ate, Bis, Labe, Met, Nad, Pin, Prop, Sot, Tim	Oasis MCX [®] (150 mg)	Effluent wastewater (250 mL)	91-108	250	LC–MS/MS	Water sample adjusted at pH: 3. Not chiral analysis. Matrix matched calibration	Lee et al. (2007)
Ace, Al, Ate, Bis, Met, Nad, Pin, Prop, Sot, Tim	Oasis MCX [®] (60 mg)	Influent wastewater (400 mL)	10-68	1000	HPLC-MS	Water sample acidified. Not chiral analysis. Matrix matched calibration	Piram et al. (2008)
Ate, Prop, Met	Oasis MCX [®] (60 mg)	Effluent wastewater (250 mL)	17-84	500	UPLC–ESI-MS/MS	Water sample adjusted at pH: 2. Not chiral analysis. Internal standard calibration.	Kasprzyk-Hordern et al. (2008)
		Influent wastewater (250 mL)	14-76	500			
		Surface water (1000 mL)	40-90	2000			

Ate, Met	Oasis MCX [®] (60 mg)	River water (150 mL) Effluent wastewater (100 mL)	71-74 82-87	750 500	LC-ESI- MS/MS	Water sample adjusted at pH: 2. Not chiral analysis. Internal standard calibration.	Al-Odaini et al. (2010)
Ate, Nad, Met, Bis, Bet	Oasis MCX [®] (150 mg)	Ground water (100 mL)	79-114	200	LC-TOF-MS	Water sample adjusted at pH: 3. Not chiral analysis.	Galera et al. (2011)
Ace, Ate, Met, Prop, Tim, Nad, Ox, Pin, Al	Oasis MCX [®] (60 mg)	Effluent wastewater (200 mL)	25-97	200	LC-ESI- MS/MS	Water sample pH not specified. Not chiral analysis. Internal standard calibration.	Salem et al. (2012)
Ace, Ate, Met, Nad, Pin, Prop	Strata X [®] (200 mg)	Effluent wastewater (250 mL)	62-96 28-92	50	GC-FID or GC-MS	Water sample pH not adjusted (pH: 8). Not chiral analysis. Matrix matched calibration	Caban et al. (2012)
Ace, Ate, Nad, Met, Prop	Strata X [®] (200 mg)	Tap water (250 mL)	63-113	50	CG-MS	Water sample pH not specified. Not chiral analysis.	Caban et al. (2015)
Ate, Prop	Oasis HLB [®] (200 mg)	Effluent wastewater (100 mL)	87-97	100	LC-MS/MS	Water sample adjusted at pH: 7. Not chiral analysis. Matrix matched calibration	Gómez et al. (2006)

Ox, Met, Prop, Bis, Bet	Oasis HLB® (60 mg)	River water (500 mL)	94-103	500	CG-MS	Water sample adjusted at pH: 7.5. Not chiral analysis.	Miège et al. (2006)																		
Ace, Ate, Met, Sot	Oasis HLB® (60 mg)	Effluent wastewater (250 mL)	78-101	500	LC-MS/MS	Water sample pH not specified. Not chiral analysis. Internal standard calibration.	Vieno et al. (2006)																		
		Influent wastewater (100 mL)	64-108	200																					
		Surface water (500 mL)	62-105	1000																					
		Ground water (1000 mL)	76-93	2000																					
Ate, Met, Nad, Pin, Prop, Sot	Oasis HLB® (60 mg)	Effluent wastewater (500 mL)	50-115	100	HPLC-	Water sample pH not adjusted (pH: 7). Chiral separation. Matrix matched calibration	MacLeod et al. (2007)																		
		Influent wastewater (100 mL)	56-110	20	MS/MS			Neb, Met, Ate, Bis	Oasis HLB® (200 mg)	Surface water (50 mL)	73-101	100	HILIC-	Water sample adjusted at pH: 7. Not chiral analysis. Internal standard calibration.	van Nuijs et al. (2010)	Influent wastewater (50 mL)	65-104	MS/MS	Met, Prop, Ate	Oasis HLB® (500 mg)	Effluent wastewater (200 mL)	69-102	200	UPLC-	Water sample adjusted at pH: 2.5. Not chiral analysis
Neb, Met, Ate, Bis	Oasis HLB® (200 mg)	Surface water (50 mL)	73-101	100	HILIC-	Water sample adjusted at pH: 7. Not chiral analysis. Internal standard calibration.	van Nuijs et al. (2010)																		
		Influent wastewater (50 mL)	65-104		MS/MS			Met, Prop, Ate	Oasis HLB® (500 mg)	Effluent wastewater (200 mL)	69-102	200	UPLC-	Water sample adjusted at pH: 2.5. Not chiral analysis	Yuan et al. (2014)	Influent wastewater (200 mL)	69-86	MS/MS							
Met, Prop, Ate	Oasis HLB® (500 mg)	Effluent wastewater (200 mL)	69-102	200	UPLC-	Water sample adjusted at pH: 2.5. Not chiral analysis	Yuan et al. (2014)																		
		Influent wastewater (200 mL)	69-86		MS/MS																				

Met, Prop, Sot	Oasis HLB [®] (not specified)	Wastewater (50 mL)	21-150	100	LC-MS/MS	Water sample pH not specified. Chiral analysis. Internal standard calibration	Evans et al. (2015)
Ate, Sot, Pin, Tim, Met, Car, Prop, Bet	MIP4SPE [™] (not specified)	Effluent wastewater (25 mL) Influent wastewater (25 mL)	50-110 40-112	25	LC-QqLIT- MS	Water sample neutral pH (not adjusted). Not chiral analysis. Internal standard calibration.	Gros et al. (2008)
Prop	SupelMIP [™] (not specified)	River water (100 mL)	97	100	HPLC-DAD	Water sample neutral pH (not adjusted). Chiral separation. Matrix matched calibration	Morante-Zarcero and Sierra (2012a)
Met, Pin, Prop, Ate	SupelMIP [™] (not specified)	River water (100 mL)	97	100	HPLC-DAD	Water sample neutral pH (not specified). Simultaneous chiral separation. Matrix matched calibration	Morante-Zarcero and Sierra (2012b)
Met, Prop, Bis, Bet, Nad, Car, Tim	C18-end capped (500 mg)	Ground water (1000 mL)	26-125	250	CG-MS	Water sample adjusted at pH: 7.5. Not chiral analysis.	Ternes et al. (1998)
Ate, Met, Nad, Bet, Bis, Car, Cel, Prop, Sot	Bakerbond C18 [®] (not specified)	Effluent wastewater (100 mL) Influent wastewater (200 mL) River water (1000 mL)	31-84 15-49 36-92	100 200 1000	LC-MS/MS	Water sample pH not adjusted. Not chiral analysis. Matrix matched calibration	Scheurer et al. (2010)
Ace, Ate, Nad, Met, Prop	Strata C18-EC [®] (200 mg)	Tap water (250 mL)	20-81	50	CG-MS	Water sample pH not adjusted. Not chiral analysis.	Caban et al. (2015)

Ate, Nad, Pin, Tim, Bis, Bet	MCM-41	River water (100 mL)	67-98	100	Micro-LC-MS/MS	Water sample adjusted at pH: 2. Not chiral analysis. Standard addition calibration.	Dahane et al (2016)
Pin, Ate, Prop, Met	SBA-15-C18 (100 mg)	Tap water (150 mL)	72 - 118	300	CE-DAD	Water sample pH not adjusted. Simultaneous chiral separation. Matrix matched calibration	Silva et al. (2017)
		River water (150 mL)	66-106				
Pin, Ate, Prop, Met	SBA-15-C8 (200 mg)	Ground water (150 mL)	62-105	500	CE-DAD	Water sample pH not adjusted. Simultaneous chiral separation. Matrix matched calibration	This work
		River water (250 mL)	91- 98				
		Effluent wastewater (250 mL)	86 - 98				

References

- <jrn>Al-Odaini NA, Zakaria MP, Yaziz MI, Surif S (2010). Multiresidue analytical method for human pharmaceuticals and synthetic hormones in river water and sewage effluents by solid-phase extraction and liquid chromatography–tandem mass spectrometry. *Journal of Chromatography A* **1217**, 6791–6806. [doi:10.1016/j.chroma.2010.08.033](https://doi.org/10.1016/j.chroma.2010.08.033)</jrn>
- <jrn>Evans SE, Davies P, Lubben A, Kasprzyk-Hordern B (2015). Determination of chiral pharmaceuticals and illicit drugs in wastewater and sludge using microwave-assisted extraction, solid-phase extraction and chiral liquid chromatography coupled with tandem mass spectrometry. *Analytica Chimica Acta* **882**, 112–126. [doi:10.1016/j.aca.2015.03.039](https://doi.org/10.1016/j.aca.2015.03.039)</jrn>
- <jrn>Galera MM, Vázquez PP, Vázquez MDMP, García MDG, Amate CF (2011). Analysis of β -blockers in groundwater using large-volume injection coupled-column reversed-phase liquid chromatography with fluorescence detection and liquid chromatography time-of-flight mass spectrometry. *Journal of Separation Science* **34**, 1796–1804. [doi:10.1002/jssc.201100117](https://doi.org/10.1002/jssc.201100117)</jrn>
- <jrn>Gómez MJ, Petrović M, Fernández-Alba AR, Barceló D (2006). Determination of pharmaceuticals of various therapeutic classes by solid-phase extraction and liquid chromatography–tandem mass spectrometry analysis in hospital effluent wastewaters. *Journal of Chromatography A* **1114**, 224–233. [doi:10.1016/j.chroma.2006.02.038](https://doi.org/10.1016/j.chroma.2006.02.038)</jrn>
- <jrn>Gros M, Pizzolato TM, Petrović M, de Alda MJL, Barceló D (2008). Trace level determination of β -blockers in waste waters by highly selective molecularly imprinted polymers extraction followed by liquid chromatography–quadrupole-linear ion trap mass spectrometry. *Journal of Chromatography A* **1189**, 374–384. [doi:10.1016/j.chroma.2007.10.052](https://doi.org/10.1016/j.chroma.2007.10.052)</jrn>
- <jrn>Lee HB, Sarafin K, Peart TE (2007). Determination of β -blockers and β 2-agonists in sewage by solid-phase extraction and liquid chromatography–tandem mass spectrometry. *Journal of Chromatography A* **1148**, 158–167. [doi:10.1016/j.chroma.2007.03.024](https://doi.org/10.1016/j.chroma.2007.03.024)</jrn>

<jrn>MacLeod SL, Sudhir P, Wong CS (2007). Stereoisomer analysis of wastewater-derived β -blockers, selective serotonin re-uptake inhibitors, and salbutamol by high-performance liquid chromatography–tandem mass spectrometry. *Journal of Chromatography A* **1170**, 23–33. [doi:10.1016/j.chroma.2007.09.010](https://doi.org/10.1016/j.chroma.2007.09.010)</jrn>

<jrn>Van Nuijs AL, Tarcomnicu I, Simons W, Bervoets L, Blust R, Jorens PG, Covaci A (2010). Optimization and validation of a hydrophilic interaction liquid chromatography–tandem mass spectrometry method for the determination of 13 top-prescribed pharmaceuticals in influent wastewater. *Analytical and Bioanalytical Chemistry* **398**, 2211–2222. [doi:10.1007/s00216-010-4101-1](https://doi.org/10.1007/s00216-010-4101-1).</jrn>

<jrn>Yuan X, Qiang Z, Ben W, Zhu B, Liu J (2014). Rapid detection of multiple class pharmaceuticals in both municipal wastewater and sludge with ultrahigh-performance liquid chromatography–tandem mass spectrometry. *Journal of Environmental Sciences* **26**, 1949–1959. [doi:10.1016/j.jes.2014.06.022](https://doi.org/10.1016/j.jes.2014.06.022)</jrn>