Supporting information

Multi-residue determination of micropollutants in Nigerian fish from Lagos lagoon using ultrasound assisted extraction, solid phase extraction and ultra-high-performance liquid chromatography tandem mass spectrometry

Idera Fabunmi[a,b,c], Natalie Sims[a], Kathryn Proctor[a] , Aderonke Oyeyiola[b], Temilola Oluseyi[b*] , Kehinde Olayinka[b], Barbara Kasprzyk-Hordern[a*]

[a]Department of Chemistry, University of Bath, Bath BA2 7AY, UK
[b]Department of Chemistry, University of Lagos, Lagos, Nigeria
[c]Nigerian Institute for Oceanography and Marine Research

*Corresponding author: b.kasprzyk-hordern@bath.ac.uk
**Corresponding author: temilolaoluseyi@gmail.com, toluseyi@unilag.edu.ng

The supporting information contains three tables:

Table S1. Instrument performance data for studies micropollutants (ordered by micropollutant class) as described by Petrie *et al.*[1]

Table S2. Instrument details including chromatographic retention time and mass spectrometry parameters used in the determination of micropollutants (ordered by micropollutant class) as described by Petrie *et al.*[1]

Table S3. Instrument parameter: Intra- and inter-day precision for target micropollutants at 10, 100 and 500 ng mL^{-1} (ordered by micropollutant class) as described by Petrie *et al.*[1]

Table S4. Instrument parameter: Intra- and inter-day accuracy for target micropollutants at 10, 100 and 500 ng mL^{-1} (ordered by micropollutant class) as described by Petrie *et al.*[1]

Table S5. Method parameter: Precision (%) of the UAE-SPE-UPLC-MSMS method in Sole fish (*Solea solea)* (ordered by micropollutant class)

Table S6. Method parameter: Accuracy (%) of the UAE-SPE-UPLC-MSMS method in Sole fish (*Solea solea)* (ordered by micropollutant class)

Table S7. Concentrations (ng g^{-1}) of micropollutants in fish species from the Lagos lagoon (ordered by micropollutant class)

Table S1. Instrument performance data for studies micropollutants (ordered by micropollutant class) as described by Petrie et al. [1]

Micro-pollutant class	Micro-pollutant	Rt (min)	ESI mode	Corresponding internal standard[b]	Linearity Range (ng mL^{-1})	Linearity r^2	$IDL_{S/N}$ (ng mL^{-1})	$IQL_{S/N}$ (ng mL^{-1})	Intra-day instrument performance[a] Precision (%)	Intra-day instrument performance[a] Accuracy (%)	Inter-day instrument performance[a] Precision (%)	Inter-day instrument performance[a] Accuracy (%)
Anaesthetic	Ketamine	10.6	+	Ketamine D_4	0.05-500	0.999	0.01	0.05	1.3	93.6	1.8	92.5
Analgesics and metabolites	Methadone	17.6	+	Methadone D_9	0.05-400	0.998	0.01	0.05	1.4	100.2	1.5	98.7
	Normorphine	3..4	+	Morphine D_3	1.00-500	0.999	0.30	1.00	2.2	99.8	1.5	100.9
	Tramadol	11.0	+	Metoprolol D_7	1.00-500	0.999	0.01	1.00	1.9	98.4	1.6	100.1
	N-desmethyltramadol	11.9	+	Cocaine-D_3	0.50-500	0.999	0.01	0.50	2.2	94.4	2.5	92.5
	O-desmethyltramadol	8.3	+	MDA-D_5	1.00-400	0.997	0.01	1.00	4.9	98.5	3.3	95.3
Anti-bacteria/antibiotics	Clarithromycin	18.9	+	Methadone D_9	0.06-500	1.000	0.01	0.06	2.4	101.8	2.6	99.8
	Trimethoprim	8.4	+	Methamphetamine D_5	0.10-500	0.999	0.03	0.10	2.2	99.5	3.0	96.9
	Sulfamethoxazole	9.6	+	Benzoylecgonine D_8	0.10-500	0.999	0.03	0.10	2.4	96.0	3.5	95.1
Anti-cancer	Ifosfamide	12.7	+	Metoprolol D_7	0.05-500	0.999	0.01	0.05	2.7	95.3	2.4	93.6
	Tamoxifen	22.4	+	Tamoxifen $^{13}C_2$ ^{15}N	0.03-1000	0.998	0.01	0.03	2.4	96.8	4.0	96.0
Anti-depressants and metabolite	Fluoxetine	18.4	+	Fluoxetine D_5	0.05-1000	0.999	0.01	0.05	1.8	98.3	1.7	96.8
	Sertraline	19.2	+	Sertraline D_3	0.05-500	1.000	0.01	0.05	1.7	95.7	1.6	95.3
	Mirtazapine	13.5	+	Mirtazapine D_3	0.05-500	0.999	0.01	0.05	2.7	97.6	3.4	94.8
	Citalopram	15.1	+	Citalopram D_6	0.50-500	0.999	0.05	0.50	2.6	101.8	0.7	101.2
	Venlafaxine	14.1	+	Metoprolol D_7	0.04-500	1.000	0.01	0.04	1.7	90.5	2.5	91.2
	Desmethylvenlafaxine	10.8	+	Metoprolol D_7	0.10-500	0.998	0.03	0.10	2.1	102.3	2.8	101.3
Anti-epileptic and metabolites	Carbamazepine	16.2	+	Carbamazepine $^{13}C_6$	0.05-500	1.000	0.01	0.05	1.6	92.7	2.0	91.7
	Carbamazepine10,11-epoxide	13.5	+	Carbamazepine $^{13}C_6$	0.10-500	0.998	0.03	0.10	2.1	89.9	1.6	88.9
	10,11 dihydro 10 hydroxycarbamazepine	13.5	+	Carbamazepine $^{13}C_6$	0.50-1000	0.997	0.05	0.50	5.6	93.8	2.8	92..2
Antihistamine	Cetirizine	18.7	+	Temazepam D_5	0.08-500	1.000	0.02	0.08	1.3	100.8	1.3	100.5
Beta-blocker	Metoprolol	11.2	+	Metoprolol D_7	0.05-500	0.999	0.01	0.05	2.0	96.1	1.3	96.8
	Propranolol	15.1	+	Propranolol D_7	0.09-500	0.999	0.03	0.09	1.0	106.2	2.0	105.4
Calcium channel blocker	Diltiazem	16.7	+	Carbamazepine $^{13}C_6$	0.10-500	0.999	0.01	0.10	2.3	93.6	2.3	92.7
Anti-diabetes	Gliclazide	17.8	+	Carbamazepine $^{13}C_6$	0.05-500	0.998	0.01	0.05	2.8	95.3	2.1	93.2
Drug precursor	Ephedrine/pseudoephedrine	7.2	+	1S,2R-(+) Ephedrine D_3	0.10-500	1.000	0.03	0.10	3.4	97.3	4.1	94.0
Nicotine metabolites	Cotinine	7.2	+	Cotinine D_3	0.05-500	0.999	0.01	0.05	1.5	98.8	1.5	98.4
	1,7 dimethylxantine	6..8	+	Cotinine D_3	1.00-500	0.999	0.30	1.00	9.9	94.9	6.0	94.3
Hypertension	Lisinopril	7.1	+	Amphetamine D_5	0.93-400	0.995	0.09	0.93	7.2	95.2	2.2	97.2
Hypnotic	Temazepam	18.2	+	Temazepam D_5	0.05-500	0.999	0.01	0.05	1.6	97.9	1.0	97.0

Table S1. (Continued)

Micro-pollutant class	Micro-pollutant	Rt (min)	ESI mode	Corresponding internal standard[b]	Linearity Range (ng mL^{-1})	r^2	$IDL_{S/N}$ (ng mL^{-1})	$IQL_{S/N}$ (ng mL^{-1})	Intra-day instrument performance[a] Precision (%)	Intra-day instrument performance[a] Accuracy (%)	Inter-day instrument performance[a] Precision (%)	Inter-day instrument performance[a] Accuracy (%)
Anti-inflammatory	Acetaminophen	5.1	+	Acetaminophen D_4	0.54-500	0.999	0.11	0.54	2.6	99.0	1.6	97.4
Stimulants and metabolites	Amphetamine	8.4	+	Amphetamine D_5	0.10-500	0.999	0.03	0.10	1.6	100.7	4.4	100.8
	Methamphetamine	8.5	+	Methamphetamine D_5	0.10-500	0.999	0.03	0.10	1.3	101.1	2.2	101.0
	MDMA	8.6	+	MDMA D_5	0.05-500	1.000	0.01	0.05	1.7	99.8	1.3	99.2
	MDA	8.6	+	MDA D_5	0.10-500	0.999	0.03	0.10	0.7	100.0	1.1	98.4
	6-acetylmorphine	7..7	+	Cotinine D_3	0.10-500	0.997	0.03	0.10	5.1	100.1	6.1	95.3
	Cocaethylene	12.9	+	Cocaethylene D_3	0.05-500	1.000	0.01	0.05	1.7	94.7	2.8	95.1
	Cocaine	11.3	+	Cocaine D_3	0.05-500	1.000	0.01	0.05	1.5	99.0	2.2	97.2
	Benzoylecgonine	9.7	+	Benzoylecgonine D_8	0.05-500	0.998	0.01	0.05	0.9	103.2	2.4	103.4
	Mephedrone	9.8	+	Mephedrone D_3	0.05-500	0.998	0.01	0.05	2.9	85.7	1.8	87.1
UV filter	Benzophenone-3	21.2	+	Methadone D_9	0.05-400	0.995	0.01	0.05	4.5	86.8	3.2	84.9

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine

[a]Instrument performance was determined at concentration 10, 100, 500 ng mL^{-1}

Table S2. Instrument details including chromatographic retention time and mass spectrometry parameters used in the determination of micropollutants (ordered by micropollutant class) as described by Petrie et al. [1]

Micropollutant class	Micropollutant	t_R (min)	Precursor ion (m/z)	Product ion 1 (m/z)	CV (V)	CE (eV)	Product ion 2 (m/z)	CV (V)	CE (eV)	Ion ratio[b]
Anaesthetic	Ketamine	10.59	238.1	125.0	31	27	220.1	31	15	2.93 ±0.04
	Ketamine D_4	10.59	242.1	129.1	31	27	-	-	-	-
Analgesics and metabolites	Methadone	17.59	310.2	265.1	31	15	105.1	31	28	1.50 ±0.05
	Methadone D_9	17.60	319.3	268.2	31	15	-	-	-	-
	Normorphine	3.41	272.1	165.0	45	43	152.1	45	49	1.16 ±0.05
	Morphine D_3	3.43	289.1	152.1	53	56	-	-	-	-
	Tramadol	10.97	264.0	58.0	28	45	120.7	28	46	270 ±21.4
	Metoprolol D_7	11.23	275.4	123.1	44	20	-	-	-	-
	N-desmethyltramadol	11.88	250.1	44.0	25	12	232.1	25	8	38.6 ±0.06
	Cocaine-D_3	11.40	307.2	185.1	40	20	-	-	-	-
	O-desmethyltramadol	8.29	250.2	58.0	30	18	232.1	30	10	181 ±6.40
	MDA-D_5	8.66	185.1	168.1	21	11	-	-	-	-
Anti-bacteria/antibiotics	Clarithromycin	18.87	748.5	158.1	40	31	590.4	40	20	3.71 ±0.03
	Methadone D_9	17.60	319.3	268.2	31	15	-	-	-	-
	Trimethoprim	8.43	291.2	230.2	26	26	123.1	26	36	1.07 ±0.03
	Methamphetamine D_5	8.56	155.1	91.8	28	18	-	-	-	-
	Sulfamethoxazole	9.56	254.1	92.2	36	30	156.1	36	20	1.44 ±0.03
	Benzoylecgonine D_8	9.68	298.2	171.1	38	19	-	-	-	-
Anti-cancer	Ifosfamide	12.69	261.0	92.0	40	28	154.0	40	22	1.89 ±0.03
	Metoprolol D_7	11.23	275.4	123.1	44	20	-	-	-	-
	Tamoxifen	22.39	372.2	72.0	50	25	129.0	50	28	30.6 ±0.91
	Tamoxifen $^{13}C_2$ ^{15}N	22.36	375.1	75.0	50	25	-	-	-	-
Anti-depressants and metabolite	Fluoxetine	18.35	310.2	44.1	34	10	148.1	34	10	14.9 ±0.54
	Fluoxetine D_5	18.32	315.3	153.2	26	8	-	-	-	-
	Sertraline	19.22	306.0	159.0	23	27	275.0	23	10	1.26 ±0.02
	Sertraline D_3	19.23	309.1	159.0	23	27	-	-	-	-
	Mirtazapine	13.52	266.1	195.0	44	26	72.0	44	18	1.05 ±0.03
	Mirtazapine D_3	13.40	269.0	194.9	35	25	-	-	-	-
	Citalopram	15.06	325.1	262.1	46	18	109.9	46	26	21.8 ±3.45
	Citalopram D_6	15.16	331.0	109.0	46	28	-	-	-	-
	Venlafaxine	14.14	278.2	58.1	27	40	260.1	27	12	1.78 ±0.02
	Metoprolol D_7	11.23	275.4	123.1	44	20	-	-	-	-
	Desmethylvenlafaxine	10.79	264.0	107.1	25	24	246.3	25	20	2.00 ±0.04
	Metoprolol D_7	11.23	275.4	123.1	44	20	-	-	-	-
Anti-epileptic and metabolites	Carbamazepine	16.15	237.0	194.1	40	20	179.1	40	38	10.6 ±0.15
	Carbamazepine $^{13}C_6$	16.22	243.1	200.1	40	20	-	-	-	-
	Carbamazepine10,11-epoxide	13.47	253.1	180.1	39	25	210.1	39	12	1.96 ±0.04
	Carbamazepine $^{13}C_6$	16.22	243.1	200.1	40	20	-	-	-	-
	10,11 dihydro 10 hydroxycarbamazepine	13.49	255.1	194.1	20	20	179.1	20	40	9.75 ±0.29

Table S2. (Continued)

Micropollutant class	Micropollutant	t_R (min)	Precursor ion (m/z)	Product ion 1 (m/z)	CV (V)	CE (eV)	Product ion 2 (m/z)	CV (V)	CE (eV)	Ion ratio[b]
	Carbamazepine $^{13}C_6$	16.22	243.1	200.1	40	20	-	-	-	-
Antihistamine	Cetirizine	18.68	389.1	201.0	32	21	166.0	32	40	2.51 ±0.05
	Temazepam D_5	18.12	306.7	260.1	37	21	-	-	-	-
Beta-blocker	Metoprolol	11.17	268.3	116.1	42	20	121.1	42	22	2.36 ±0.04
	Metoprolol D_7	11.23	275.4	123.1	44	20	-	-	-	-
	Propranolol	15.14	260.2	183.1	42	18	116.1	42	16	1.88 ±0.09
	Propranolol D_7	15.12	267.0	188.8	40	18	-	-	-	-
Calcium channel blocker	Diltiazem	16.72	415.0	178.0	40	25	310.1	40	25	23.4 ±1.30
	Carbamazepine $^{13}C_6$	16.22	243.1	200.1	40	20	-	-	-	-
Anti-diabetes	Gliclazide	17.80	324.1	127.0	41	20	110.0	41	20	0.98 ±0.04
	Carbamazepine $^{13}C_6$	16.22	243.1	200.1	40	20	-	-	-	-
Drug precursor	Ephedrine/pseudoephedrine	7.22	166.1	148.1	23	12	133.1	23	21	6.58 ±0.12
	1S,2R-(+) Ephedrine D_3	7.21	169.2	151.0	23	18	-	-	-	-
Nicotine metabolites	Cotinine	7.22	177.1	80.0	34	21	98.1	34	22	2.91 ±0.05
	Cotinine D_3	7.21	180.1	80.0	44	24	-	-	-	-
	1,7 dimethylxantine	6.78	181.0	124.1	54	21	-	-	-	-
	Cotinine D_3	7.21	180.1	80.0	44	24	-	-	-	-
Hypertension	Lisinopril	7.14	406.2	84.0	38	27	246.1	38	22	9.42 ±0.30
	Amphetamine D_5	8.42	141.1	92.8	20	14	-	-	-	-
Hypnotic	Temazepam	18.16	301.1	255.1	37	21	283.1	37	14	2.21 ±0.05
	Temazepam D_5	18.12	306.7	260.1	37	21	-	-	-	-
Anti-inflammatory	Acetaminophen	5.11	151.9	110.0	26	16	92.9	26	24	5.54 ±0.13
	Acetaminophen D_4	5.22	156.0	114.0	26	16	-	-	-	-
Stimulants and metabolites	Amphetamine	8.39	136.2	91.1	18	16	119.1	18	8	1.20 ±0.04
	Amphetamine D_5	8.42	141.1	92.8	20	14	-	-	-	-
	Methamphetamine	8.51	150.2	91.1	24	19	119.1	24	10	1.73 ±0.03
	Methamphetamine D_5	8.56	155.1	91.8	28	18	-	-	-	-
	MDMA	8.62	194.1	163.1	24	13	105.1	24	24	2.34 ±0.03
	MDMA D_5	8.69	199.1	165.1	26	13	-	-	-	-
	MDA	8.59	180.0	163.1	21	11	105.1	21	22	2.73 ±0.06
	MDA D_5	8.66	185.1	168.1	21	11	-	-	-	-
	6-acetylmorphine	7.65	328.1	165.1	52	39	211.1	52	26	1.46 ±0.04
	Cotinine D_3	7.21	180.1	80.0	44	24	-	-	-	-
	Cocaethylene	12.91	318.2	196.2	38	20	82.1	38	30	1.90 ±0.05
	Cocaethylene D_3	12.98	321.2	199.1	40	22	-	-	-	-
	Cocaine	11.32	304.2	182.1	40	20	82.1	40	31	2.75 ±0.09
	Cocaine D_3	11.40	307.2	185.1	40	20	-	-	-	-
	Benzoylecgonine	9.68	290.2	168.1	38	19	105.1	38	30	1.95 ±0.03
	Benzoylecgonine D_8	9.68	298.2	171.1	38	19	-	-	-	-
	Mephedrone	9.79	178.1	160.1	10	12	145.0	10	22	1.55 ±0.06
	Mephedrone D_3	9.86	181.1	148.0	30	22	-	-	-	-
UV filter	Benzophenone-3	21.23	229.0	151.0	35	18	105.0	35	20	1.58 ±0.04
	Methadone D_9	17.60	319.3	268.2	31	15	-	-	-	-

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine; Key: CV, cone voltage; CE, collision energy; ESI, electrospray ionisation; [b]MRM ratio: Product ion 1/Product ion 2 ratio average over the entire calibration range

Table S3. Instrument parameter: Intra- and inter-day precision for target micropollutants at 10, 100 and 500 ng mL^{-1} (ordered by micropollutant class) as described by Petrie et al. [1]

Micropollutant class	Micropollutant	Intra-day precision (%)			Inter-day precision (%)		
		10 ng mL^{-1}	100 ng mL^{-1}	500 ng mL^{-1}	10 ng mL^{-1}	100 ng mL^{-1}	500 ng mL^{-1}
		Chemicals determined in ESI + mode					
Anaesthetic	Ketamine	0.4	1.8	1.6	2.7	1.7	1.1
Analgesics and metabolites	Methadone	1.7	1.2	-	0.7	2.3	-
	Normorphine	3.6	1.1	1.9	2.2	1.5	0.9
	Tramadol	1.2	2.6	1.9	1.7	1.7	1.5
	N-desmethyltramadol	3.2	1.2	2.2	3.4	3.0	1.1
	O-desmethyltramadol	3.1	6.7	-	2.0	4.6	-
Anti-bacteria/antibiotics	Clarithromycin	4.0	2.0	1.2	5.1	1.5	1.3
	Trimethoprim	1.6	1.8	3.2	1.4	3.5	4.1
	Sulfamethoxazole	0.7	2.2	4.3	2.0	3.0	5.6
Anti-cancer	Ifosfamide	4.0	3.3	0.7	2.9	3.1	1.3
	Tamoxifen	2.2	0.7	4.3	4.5	1.8	5.6
Anti-depressants and metabolite	Fluoxetine	2.2	1.3	1.9	1.5	2.1	1.4
	Sertraline	0.7	1.7	2.7	3.0	1.0	0.9
	Mirtazapine	2.2	3.0	2.9	3.9	4.2	2.1
	Citalopram	1.7	4.9	1.2	0.5	0.8	0.8
	Venlafaxine	1.4	1.3	2.4	3.9	2.0	1.6
	Desmethylvenlafaxine	2.4	2.4	1.4	4.0	2.3	2.0
Anti-epileptic and metabolites	Carbamazepine	2.4	1.1	1.3	2.6	1.7	1.6
	Carbamazepine10,11-epoxide	2.0	2.4	2.0	0.9	0.1	3.7
	10,11 dihydro 10 hydroxycarbamazepine	5.1	3.8	7.9	3.7	2.0	2.8
Antihistamine	Cetirizine	0.8	0.4	2.6	2.0	0.9	0.8
Beta-blocker	Metoprolol	0.9	3.2	1.8	2.0	1.2	0.6
	Propranolol	0.4	0.7	1.9	2.1	1.5	2.5
Calcium channel blocker	Diltiazem	3.2	2.2	1.4	0.4	1.5	4.9
Anti-diabetes	Gliclazide	2.5	5.0	0.8	0.8	4.4	1.1
Drug precursor	Ephedrine/pseudoephedrine	2.5	4.1	3.5	4.5	3.6	4.1
Nicotine metabolites	Cotinine	1.5	1.2	1.8	0.8	2.2	1.6
	1,7 dimethylxantine	6.8	9.0	13.9	13.0	2.9	2.1
Hypertension	Lisinopril	13.0	1.4	-	0.9	3.4	-
Hypnotic	Temazepam	1.1	1.4	2.4	1.7	1.0	0.4
Anti-inflammatory	Acetaminophen	4.3	0.9	2.7	1.6	1.6	1.6
Stimulants and metabolites	Amphetamine	1.3	1.3	2.2	0.8	2.1	1.5
	Methamphetamine	1.2	0.3	2.4	1.6	0.2	1.3
	MDMA	3.3	0.5	1.2	0.6	1.2	2.1
	MDA	0.6	0.4	1.1	1.4	1.1	0.7
	6-acetylmorphine	3.5	3.4	8.3	1.1	9.6	7.5
	Cocaethylene	2.0	1.2	1.8	5.9	0.8	1.6
	Cocaine	1.1	1.1	2.2	2.2	2.7	1.7
	Benzoylecgonine	0.7	1.1	0.9	1.4	3.3	2.5
	Mephedrone	4.9	3.0	0.9	1.8	0.9	2.8
UV filter	Benzophenone-3	4.3	4.6	-	2.5	3.8	-

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine

Table S4. Instrument parameter: Intra- and inter-day accuracy for target micropollutants at 10, 100 and 500 ng mL^{-1} (ordered by micropollutant class) as described by Petrie et al. [1]

Micropollutant class	Micropollutant	Intra-day accuracy (%)			Inter-day accuracy (%)		
		10 ng mL^{-1}	100 ng mL^{-1}	500 ng mL^{-1}	10 ng mL^{-1}	100 ng mL^{-1}	500 ng mL^{-1}
Chemicals determined in ESI+ mode							
Anaesthetic	Ketamine	77.3	105.3	98.2	75.6	103.6	98.5
Analgesics and metabolites	Methadone	101.1	99.3	-	100.4	97.1	-
	Normorphine	98.6	102.3	98.5	101.1	102.7	99.1
	Tramadol	99.0	99.0	97.3	100.6	100.6	99.0
	N-desmethyltramadol	94.7	93.9	94.7	91.1	92.8	93.6
	O-desmethyltramadol	94.2	102.8	-	92.6	97.9	-
Anti-bacteria/antibiotics	Clarithromycin	102.6	99.3	103.6	97.5	99.9	102.0
	Trimethoprim	104.6	94.2	99.6	104.5	96.8	89.3
	Sulfamethoxazole	106.1	93.6	88.3	104.3	91.6	89.3
Anti-cancer	Ifosfamide	96.4	92.5	96.9	94.5	90.6	95.6
	Tamoxifen	93.0	105.2	92.3	88.8	106.1	93.2
Anti-depressants and metabolite	Fluoxetine	92.3	103.0	99.6	91.1	101.1	98.1
	Sertraline	84.3	101.9	100.9	84.0	100.8	101.1
	Mirtazapine	94.0	101.6	97.3	90.3	99.0	95.0
	Citalopram	102.0	101.3	102.1	101.6	100.5	101.7
	Venlafaxine	72.9	96.7	101.7	74.6	96.2	102.7
	Desmethylvenlafaxine	105.9	102.2	98.9	101.4	101.3	101.3
Anti-epileptic and metabolites	Carbamazepine	86.3	94.5	97.2	84.1	94.8	96.1
	Carbamazepine10,11-epoxide	94.0	80.5	95.1	94.8	80.4	91.3
	10,11 dihydro 10 hydroxycarbamazepine	94.8	98.6	88.1	94.6	96.5	85.4
Antihistamine	Cetirizine	103.8	99.2	99.4	101.8	99.2	100.4
Beta-blocker	Metoprolol	90.9	96.7	100.7	91.3	98.1	101.1
	Propranolol	125.4	98.4	94.8	122.5	96.8	97.0
Calcium channel blocker	Diltiazem	94.0	94.6	92.1	93.8	93.4	91.0
Anti-diabetes	Gliclazide	87.3	97.2	101.5	86.7	92.5	100.3
Drug precursor	Ephedrine/pseudoephedrine	99.5	95.1	97.4	92.3	95.7	94.1
Nicotine metabolites	Cotinine	95.1	100.9	100.3	94.4	99.6	101.3
	1,7 dimethylxantine	90.0	98.4	96.3	84.2	99.9	98.7
Hypertension	Lisinopril	103.0	87.3	-	103.6	90.7	-
Hypnotic	Temazepam	93.8	98.7	101.1	92.3	98.0	100.6
Anti-inflammatory	Acetaminophen	98.1	99.3	99.5	96.4	98.0	97.7
Stimulants and metabolites	Amphetamine	100.8	101.6	99.8	100.3	102.9	99.2
	Methamphetamine	100.4	101.5	101.3	99.0	101.7	102.3
	MDMA	103.2	97.9	98.3	102.5	96.7	98.3
	MDA	100.0	100.8	99.3	98.4	99.6	97.3
	6-acetylmorphine	105.0	83.0	112.3	104.7	75.0	106.3
	Cocaethylene	88.3	101.2	94.5	88.2	101.8	95.2
	Cocaine	98.2	100.4	98.4	96.7	98.1	96.8
	Benzoylecgonine	105.2	103.2	101.4	104.3	101.6	104.4
	Mephedrone	75.9	89.9	91.4	77.3	89.9	94.4
UV filter	Benzophenone-3	84.4	89.2	-	83.4	86.3	-

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine

Table S5. Method parameter: Precision (%) of the UAE-SPE-UPLC-MSMS method in Sole fish (*Solea solea)* (ordered by micropollutant class)

Micropollutant class	Micropollutant	Spike level		
		10 ng g^{-1}	50 ng g^{-1}	100 ng g^{-1}
Anaesthetic	Ketamine	5.5	0.8	3.0
Analgesics and metabolites	Methadone	9.7	0.6	11.1
	Normorphine	0.0	0.0	0.0
	Tramadol	7.5	1.7	4.9
	N-desmethyltramadol	8.7	15.5	1.2
	O-desmethyltramadol	6.4	16.9	11.2
Anti-bacteria/antibiotics	Clarithromycin	3.5	8.7	17.7
	Trimethoprim	4.6	3.4	11.0
	Sulfamethoxazole	3.2	9.2	5.1
Anti-cancer	Ifosfamide	31.1	12.7	61.8
	Tamoxifen	0.0	1.0	1.7
Anti-depressants and metabolite	Fluoxetine	8.7	3.0	21.0
	Sertraline	0.0	2.1	0.4
	Mirtazapine	2.3	1.2	0.1
	Citalopram	4.2	5.6	3.1
	Venlafaxine	13.4	1.6	1.9
	Desmethylvenlafaxine	9.9	2.7	6.5
Anti-epileptic and metabolites	Carbamazepine	10.9	5.4	11.2
	Carbamazepine10,11-epoxide	12.1	7.4	8.4
	10,11 dihydro 10 hydroxycarbamazepine	16.0	13.1	7.8
Antihistamine	Cetirizine	1.0	7.4	30.4
Beta-blocker	Metoprolol	5.9	0.7	2.8
	Propranolol	4.5	1.8	0.4
Calcium channel blocker	Diltiazem	2.8	10.0	9.5
Anti-diabetes	Gliclazide	14.8	2.4	7.8
Drug precursor	Ephedrine/pseudoephedrine	5.0	3.8	5.2
Nicotine metabolites	Cotinine	3.9	1.6	3.2
	1,7 dimethylxantine	14.3	97.9	8.3
Hypertension	Lisinopril	5.8	0.2	4.0
Hypnotic	Temazepam	7.3	2.7	9.3
Anti-inflammatory	Acetaminophen	2.2	0.7	1.3
Stimulants and metabolites	Amphetamine	4.6	6.8	0.9
	Methamphetamine	3.7	3.1	2.9
	MDMA	2.1	0.7	0.4
	MDA	0.6	0.5	1.2
	6-acetylmorphine	86.8	1.2	35.7
	Cocaethylene	4.3	2.1	2.8
	Cocaine	0.6	2.6	6.5
	Benzoylecgonine	3.2	8.0	8.8
	Mephedrone	0.0	4.7	4.1
UV filter	Benzophenone-3	7.8	4.8	93.7

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine

Table S6. Method parameter: Accuracy (%) of the UAE-SPE-UPLC-MSMS method in Sole fish (*Solea solea)* (ordered by micropollutant class)

Micropollutant class	Micropollutant	Spike level		
		10 ng g^{-1}	**50 ng g^{-1}**	**100 ng g^{-1}**
Anaesthetic	Ketamine	83.0	104.3	100.7
Analgesics and metabolites	Methadone	83.7	117.6	103.4
	Normorphine	0.0	0.0	84.8
	Tramadol	135.4	104.3	91.5
	N-desmethyltramadol	88.7	122.5	117.1
	O-desmethyltramadol	139.2	118.3	94.7
Anti-bacteria/antibiotics	Clarithromycin	108.1	158.5	182.4
	Trimethoprim	117.1	150.1	113.8
	Sulfamethoxazole	94.0	88.8	87.7
Anti-cancer	Ifosfamide	62.7	107.4	174.3
	Tamoxifen	-10.4	90.7	91.9
Anti-depressants and metabolite	Fluoxetine	40.6	86.7	97.1
	Sertraline	-11.7	95.1	91.4
	Mirtazapine	74.0	108.1	103.4
	Citalopram	85.0	90.7	92.9
	Venlafaxine	95.1	95.1	93.3
	Desmethylvenlafaxine	90.5	116.3	94.9
Anti-epileptic and metabolites	Carbamazepine	108.6	118.2	107.7
	Carbamazepine10,11-epoxide	74.3	89.5	91.3
	10,11 dihydro 10 hydroxycarbamazepine	158.3	141.0	113.8
Antihistamine	Cetirizine	158.1	167.2	150.2
Beta-blocker	Metoprolol	110.0	117.5	116.4
	Propranolol	97.2	100.8	80.3
Calcium channel blocker	Diltiazem	74.2	113.9	90.8
Anti-diabetes	Gliclazide	95.5	85.8	116.4
Drug precursor	Ephedrine/pseudoephedrine	119.7	115.1	100.3
Nicotine metabolites	Cotinine	126.3	104.5	100.9
	1,7 dimethylxantine	133.7	52.0	68.2
Hypertension	Lisinopril	105.8	85.8	115.8
Hypnotic	Temazepam	97.3	97.3	97.4
Anti-inflammatory	Acetaminophen	143.7	123.1	116.2
Stimulants and metabolites	Amphetamine	115.0	93.4	96.3
	Methamphetamine	103.3	101.9	96.9
	MDMA	98.3	103.1	103.2
	MDA	90.3	104.4	102.2
	6-acetylmorphine	58.7	109.9	91.1
	Cocaethylene	101.0	104.0	95.9
	Cocaine	98.3	97.9	98.1
	Benzoylecgonine	108.3	132.1	122.7
	Mephedrone	0.0	94.1	93.3
UV filter	Benzophenone-3	77.1	54.1	116.7

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine

Table S7. Concentrations (ng g^{-1}) of micropollutants in fish species from the Lagos lagoon (ordered by micropollutant class)

	Micropollutants	Sole			Tilapia			Grouper			Catfish			Silver catfish			Croaker			Red snapper		
		Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes
Anaesthetic	Ketamine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Analgesics and metabolites	Methadone	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Normorphine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Tramadol	nd	nd	nd	nd	nd	0.8*	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	N-desmethyltramadol	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	O-desmethyltramadol	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Antibacterials/antibiotics	Clarithromycin	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Trimethoprim	nd	nd	nd	nd	nd	nd	nd	nd	0.1*	nd	nd	nd	nd	nd	1.8*	nd	19.2	7.4	nd	nd	1.4*
	Sulfamethoxazole	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Anti-cancer	Tamoxifen	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Anti-depressants and metabolite	Fluoxetine	3.7	12.4	27.1	3.4	nd	nd	16.3	4.5	12.9	7.4	10.8	17.2	3.2	14.3	9.0	7.1	12.2	13.4	8.2	7.6	4.4
	Sertraline	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Mirtazapine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Citalopram	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Venlafaxine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Desmethylvenlafaxine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Anti-epileptic and metabolites	Carbamazepine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Carbamazepine 10,11 epoxide	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Antihistamine	Cetirizine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd

Table S7. (Continued)

Micropollutant class	Micropollutants	Sole			Tilapia			Grouper			Catfish			Silver catfish			Croaker			Red snapper		
		Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes	Tissue	Gills	Eyes
Beta-blocker	Metoprolol	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Propranolol	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Calcium channel blocker	Diltiazem	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Anti-diabetes	Gliclazide	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Drug precursor	Ephedrine/pseudoephedrine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Nicotine metabolites	Cotinine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Hypertension	Lisinopril	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Hypnotic	Temazepam	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Anti-inflammatory	Acetaminophen	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Stimulants and metabolites	Amphetamine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Methamphetamine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	MDMA	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	MDA	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Cocaethylene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Cocaine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Benzoylecgonine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
	Mephedrone	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd

MDMA, 3,4-methylenedioxy-methamphetamine; MDA, 3,4-methylenedioxyamphetamine
nd, not detected;*Concentration <MQL

Reference

1 B. Petrie, J. Youdan, R. Barden and B. Kasprzyk-Hordern, *J. Chromatogr. A*, 2016, **1431**, 64–78.