

Supporting Information

Spatial differences among micropollutants in sewer overflows: A multisite analysis using passive samplers

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Contents

A.	Passive sampler installation setup	S4
B.	Time-weighted average (TWA) concentration estimates.....	S5
	Concentration distribution per contaminant	S7
C.	Spatial variability	S9
	ANOVA	S11
	Land use	S12
	Multiple regression	S14
D.	Risk quotient.....	S15
E.	Chemical analysis.....	S16
	QExactive: LC-HR-MS/MS	S16
	Agilent G6495A: LVI-LC-QQQ	S18

List of Figures

Figure SI 1. Installation of passive sampler mounting plates with triplicate SDB-RPS disks at the CSO (combined sewer overflow) discharge sites. The pictures are sorted by row by CSO ID.	S4
Figure SI 2. Summary of the TWA concentration ranges found in the sampled 20 CSO sites. The red × shows the environmental quality standard (EQS). Boxplot: Boxes represent the first and third quartile (Q1 and Q3), whiskers' lengths are max 1.5 x (Q3-Q1); see boxplot in R for details. 1,3-BTH-S: 1,3-benzothiazole-2-sulfonate. A. The number above gives the number of events > LOQ (limit of quantification) of 95 events. Only values > LOQ considered (version used in main paper). B. Values <LOQ replaced by 0.5 times LOQ, or if no measurement > LOQ at the site then values <LOQ replaced by 10 ⁻⁹ . C. Values < LOQ replaced by 10 ⁻⁷	S6
Figure SI 3. TWA concentration distribution over CSO sites per studied micropollutant, only value >LOQ considered. The red line indicates the environmental quality standard (EQS). The numbers above the graph show how many of the monitored events are above the EQS. 1,3-benzothiazole-2-sulfonate has no EQS value. Boxes represent the first and third quartile (Q1 and Q3), whiskers' lengths are max 1.5 x (Q3-Q1); see boxplot in R for details.	S7
Figure SI 4. Correlation between diuron and carbendazim time-weighted average (TWA) concentration.	S9
Figure SI 5. Overview on land use distribution in the 20 CSO catchments sorted in ascending order by fraction of industry (CSO ID 20: 100% industry). No data is available for CSO ID 17.	S10
Figure SI 6. Correlations between relative area of land use (building, urban green, street, other impervious areas) and TWA concentrations for 19 CSO sites (for CSO ID 17 no land use data was available). Only micropollutants shown with expected occurrence in selected urban land use category and highest found (if any) correlation	S12
Figure SI 7. Risk quotient (RQ) for all 20 CSO sites (TWA concentration divided by EQS) in comparison to event duration in minutes.....	S15

List of Tables

Table SI 1. Characteristics of the 20 CSO catchments and overflow structures. WWTP: wastewater treatment plant, CSO: combined sewer overflow	S9
Table SI 2. ANOVA p-value for the TWA concentration based on triplicates (not mean TWA concentration) to assess how much variability is due to differences between CSO sites in comparison to the variability between events. 1,3-BTH-2-S: 1,3-benzothiazole-2-sulfonate...S11	
Table SI 3. Summary results of multiple regression with survival package ¹ in R: model coefficient and p-value for each substance over all 20 catchments. Model: TWA concentration per substance = Intercept + Specific Volume + Duration + Building fraction+ Street fraction + Garden fraction + Remaining impervious surface fraction. Figure SI 5 shows the used relative land use distribution. 2,4-D and MCPA were not considered due to the few available data points. 1,3-BTH-2-S: 1,3-benzothiazole-2-sulfonate.....S14	
Table SI 4. Number of events with TWA concentration bigger than the EQS (Risk quotient; RQ > 1), only micropollutants shown with RQ > 1. Comparison of TWA concentration calculation strategies: 10% quantile, median and mean correction factor for estimation of $C_{TWA,corr}$ with non-corrected TWA concentration (C_{TWA}).....	S15
Table SI 5. HPLC gradient used for the chromatographic separation on an Atlantis T3 column. FA: formic acid.	S16
Table SI 6. Settings for the QExactive mass spectrometer	S17
Table SI 7. HPLC gradient used for the chromatographic separation on an Acquity UPLC HSS T3 column. FA: formic acid.	S18
Table SI 8. Settings for the Agilent G6495A mass spectrometer.....	S18
Table SI 9. Acquisition Method on Agilent G6495A triple quad mass spectrometer.....	S18
Table SI 10. Relative recoveries and LOQs for the different measurement series. Relative Recovery: recovery of analyte amount spiked into the sample considering the background concentration of the unspiked sample. LOQ determined with signal-to-noise ratio of 10. The LOQ of the less sensitive DRM transition is reported.....S20	

Supporting Information

Table SI 11. Quality control: measured concentration and relative recovery for three external references standards measured in one measurement sequence with Agilent 6495 TQ, direct injection. Used reference standards: 1) Neochema Pharma – Mix 17, STD-Nr. EGT-1552, 2) Dr. Ehrenstorfer Pesticide-Mix 1284, Product identification: 18001284, Lot No. 87269, 3) Dr. Ehrenstorfer Pesticide-Mix 1285, Product identification: 18001285, Lot No. 80529AL.....S20

Table SI 12. Measured concentrations [ng/disk] in field blinds. The field blinds were treated like all other passive sampler disks: preparation in laboratory and field, mounted but not exposed, stored and extracted. Field blinds were taken for different sites.S21

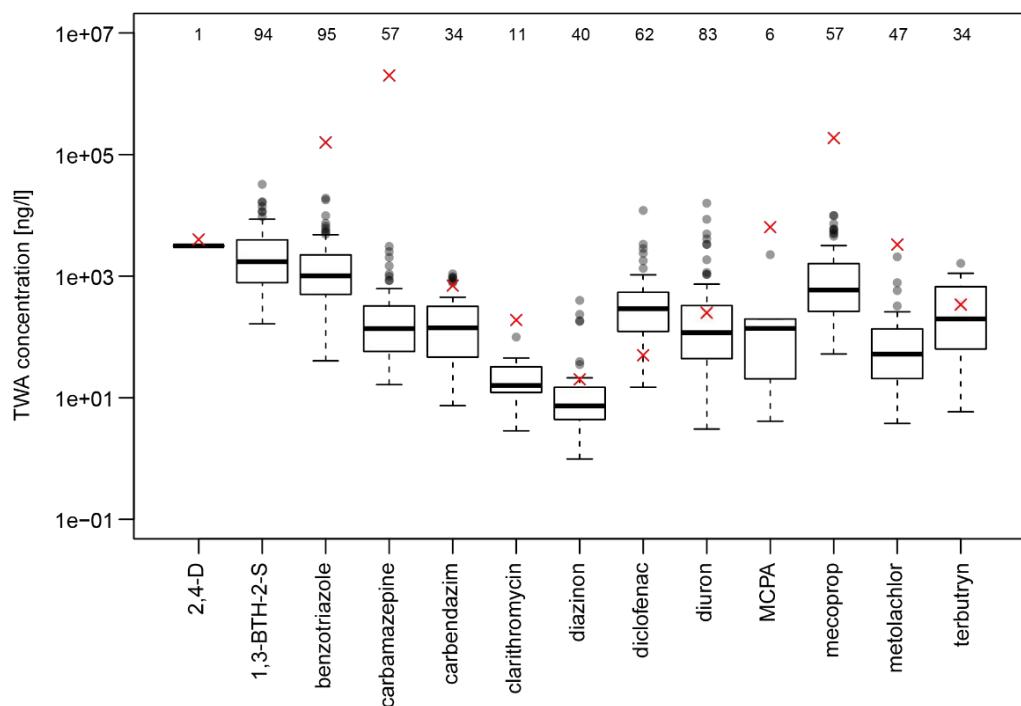
A. Passive sampler installation setup



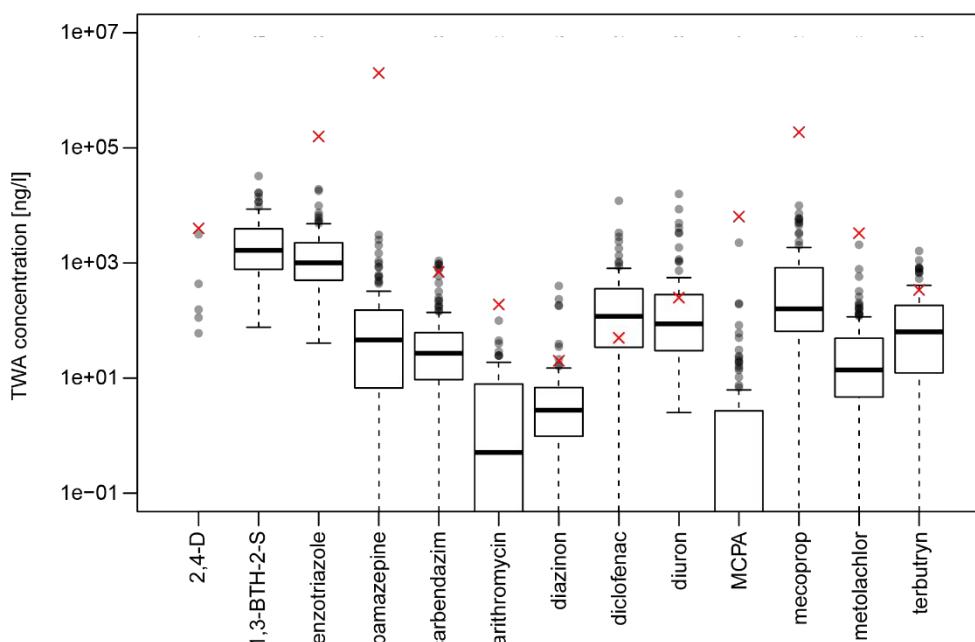
Figure SI 1. Installation of passive sampler mounting plates with triplicate SDB-RPS disks at the CSO (combined sewer overflow) discharge sites. The pictures are sorted by row by CSO ID.

B. Time-weighted average (TWA) concentration estimates

A.



B.



C.

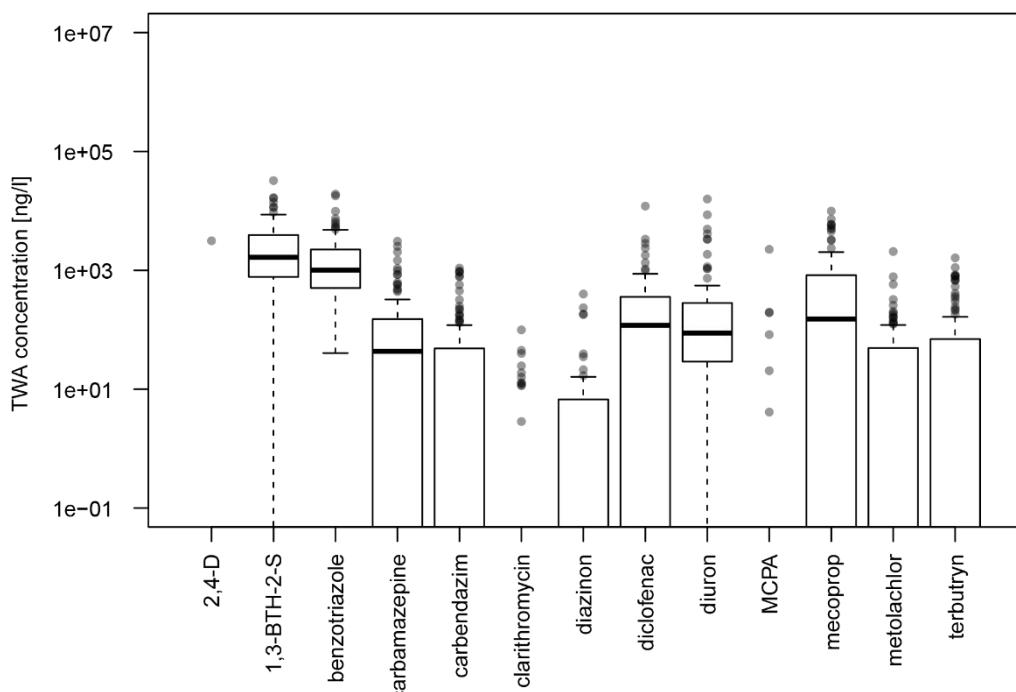
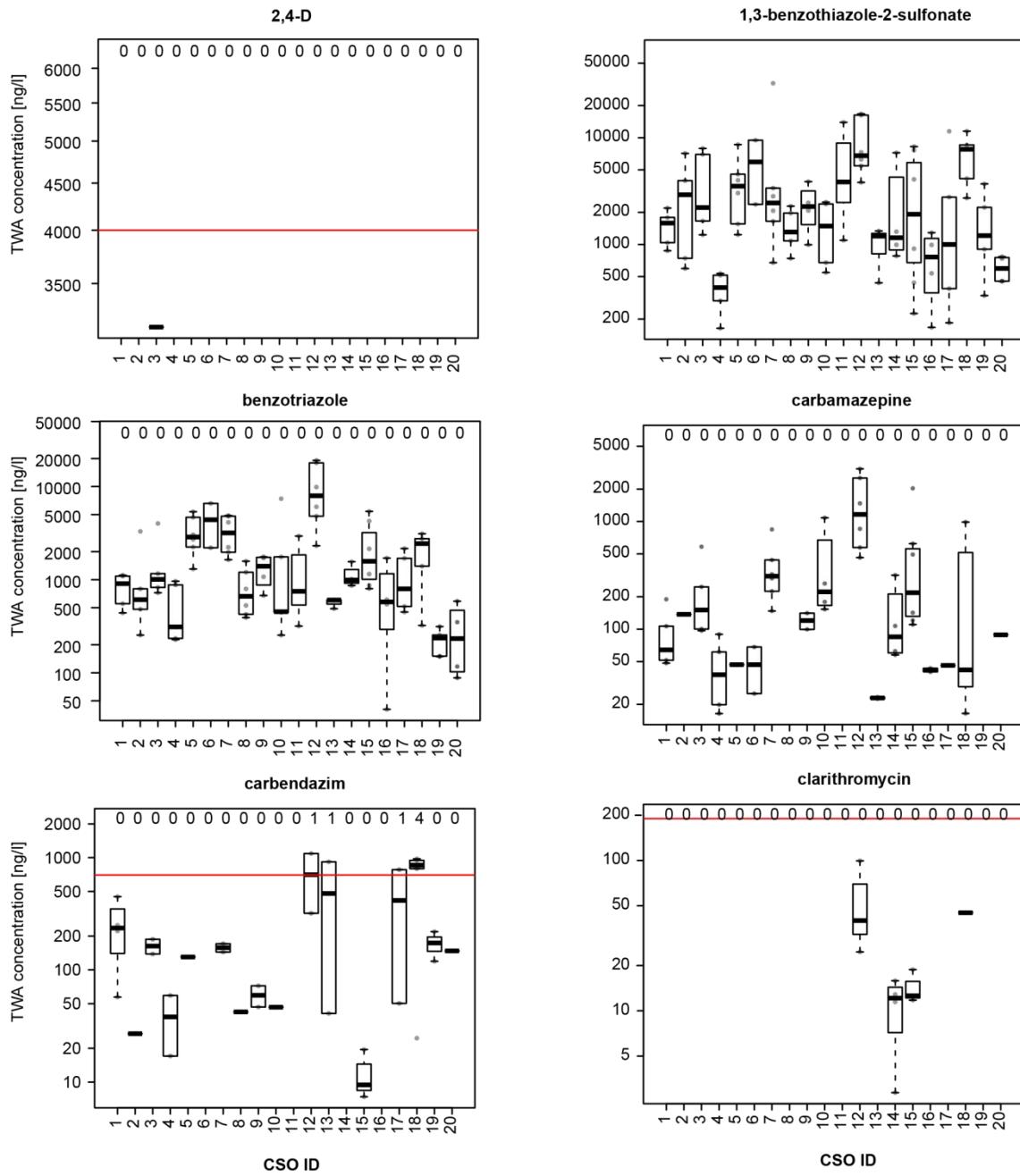


Figure SI 2. Summary of the TWA concentration ranges found in the sampled 20 CSO sites. The red × shows the environmental quality standard (EQS). Boxplot: Boxes represent the first and third quartile (Q1 and Q3), whiskers' lengths are max 1.5 x (Q3-Q1); see boxplot in R for details. 1,3-BTH-S: 1,3-benzothiazole-2-sulfonate. A. The number above gives the number of events > LOQ (limit of quantification) of 95 events. Only values > LOQ considered (version used in main paper). B. Values <LOQ replaced by 0.5 times LOQ, or if no measurement > LOQ at the site then values <LOQ replaced by 10^{-9} . C. Values < LOQ replaced by 10^{-7} .

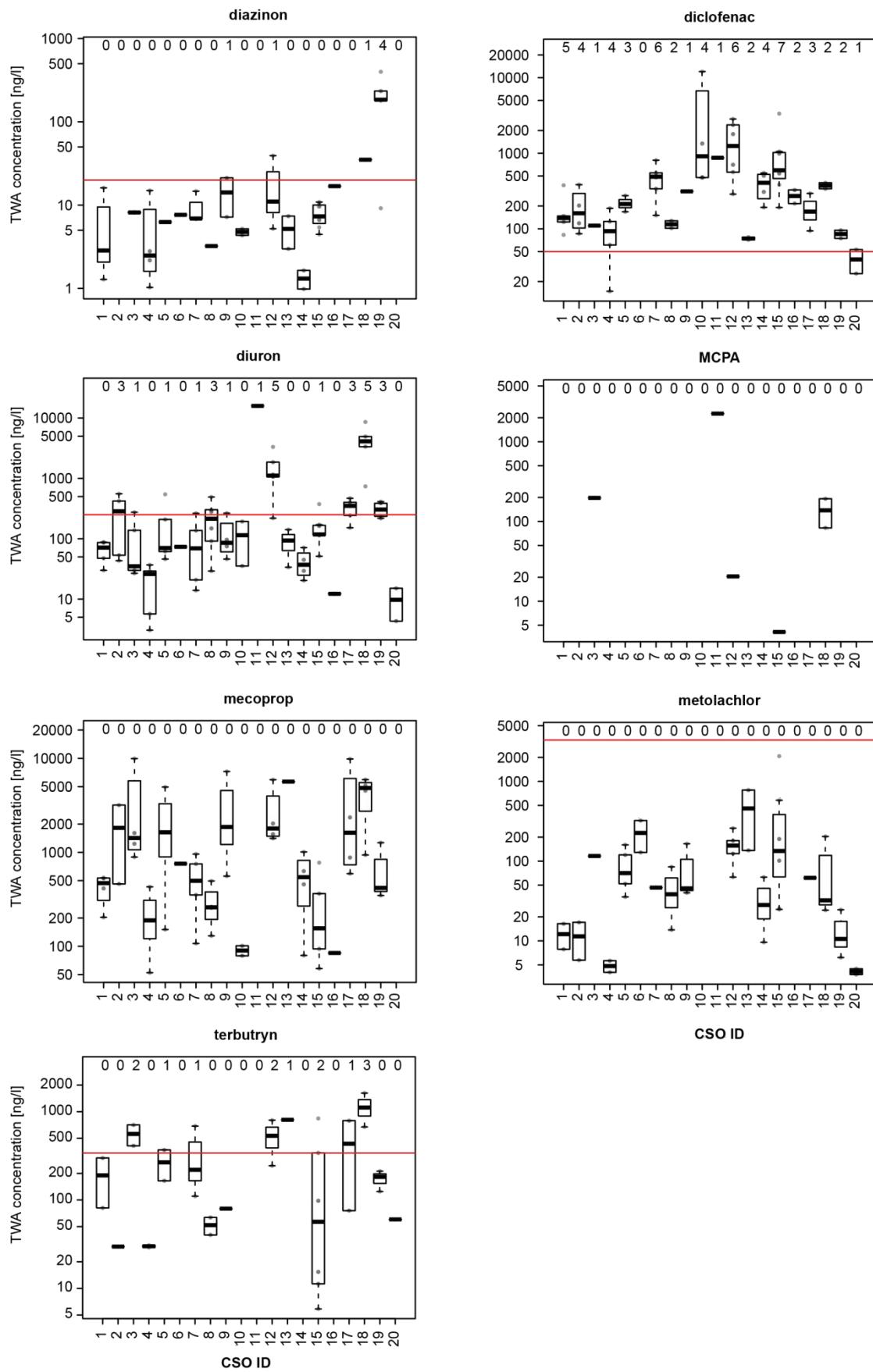
Supporting Information

CONCENTRATION DISTRIBUTION PER CONTAMINANT

Figure SI 3. TWA concentration distribution over CSO sites per studied micropollutant, only value >LOQ considered. The red line indicates the environmental quality standard (EQS). The numbers above the graph show how many of the monitored events are above the EQS. 1,3-benzothiazole-2-sulfonate has no EQS value. Boxes represent the first and third quartile (Q1 and Q3), whiskers' lengths are max 1.5 x (Q3-Q1); see boxplot in R for details.



Supporting Information



Supporting Information

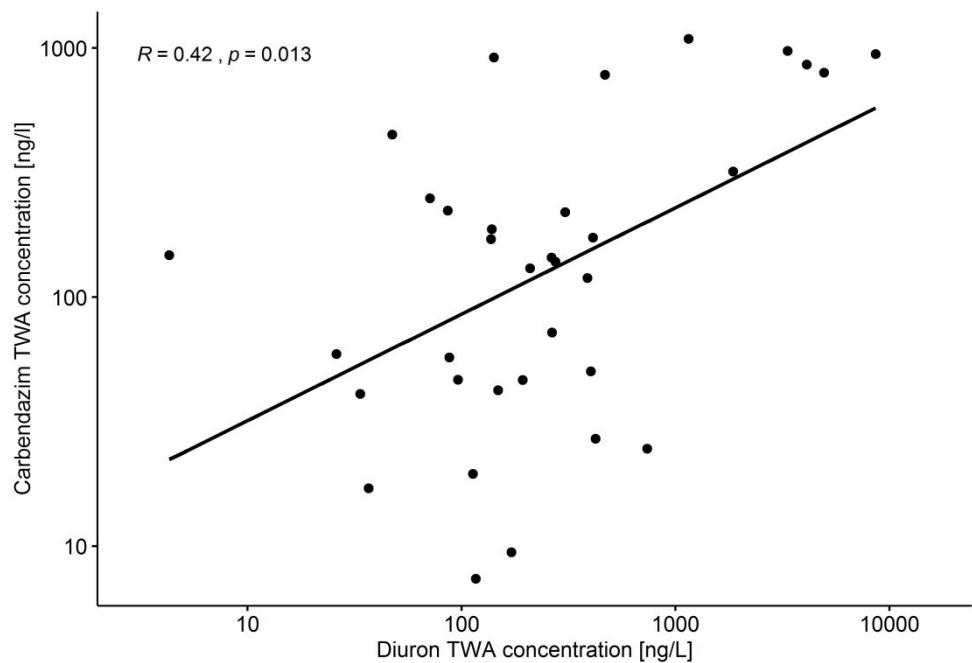


Figure SI 4. Correlation between diuron and carbendazim time-weighted average (TWA) concentration.

C. Spatial variability

Table SI 1. Characteristics of the 20 CSO catchments and overflow structures. WWTP: wastewater treatment plant, CSO: combined sewer overflow

CSO ID	Storage	Area impervious	Specific volume	Flow to
	volume m ³	ha _{imp}	m ³ /ha _{imp}	WWTP L/s
1	285	22.3	13	210
2	290	8.0	36	61
3	190	14.7	13	250
4	420	10.1	41	32
5 ^a	0	7.0	-	-
6 ^a	0	8.5	-	-
7	1,320	130.8	10	155
8	200	8.0	25	140
9	112	2.7	41	70
10 ^a	0	5.6	-	-
11 ^a	0	3.5	-	-
12	700	52.1	13	200
13	5,500	35.6	154	400
14	1,401	43.2	32	450
15	180	3.0	61	120
16	230	35.0	7	45
18	154	4.8	32	110
19	160	5.2	31	60
20	200	7.6	26	52
17	214	8.1	26	187

^adirect overflow without storage volume

Supporting Information

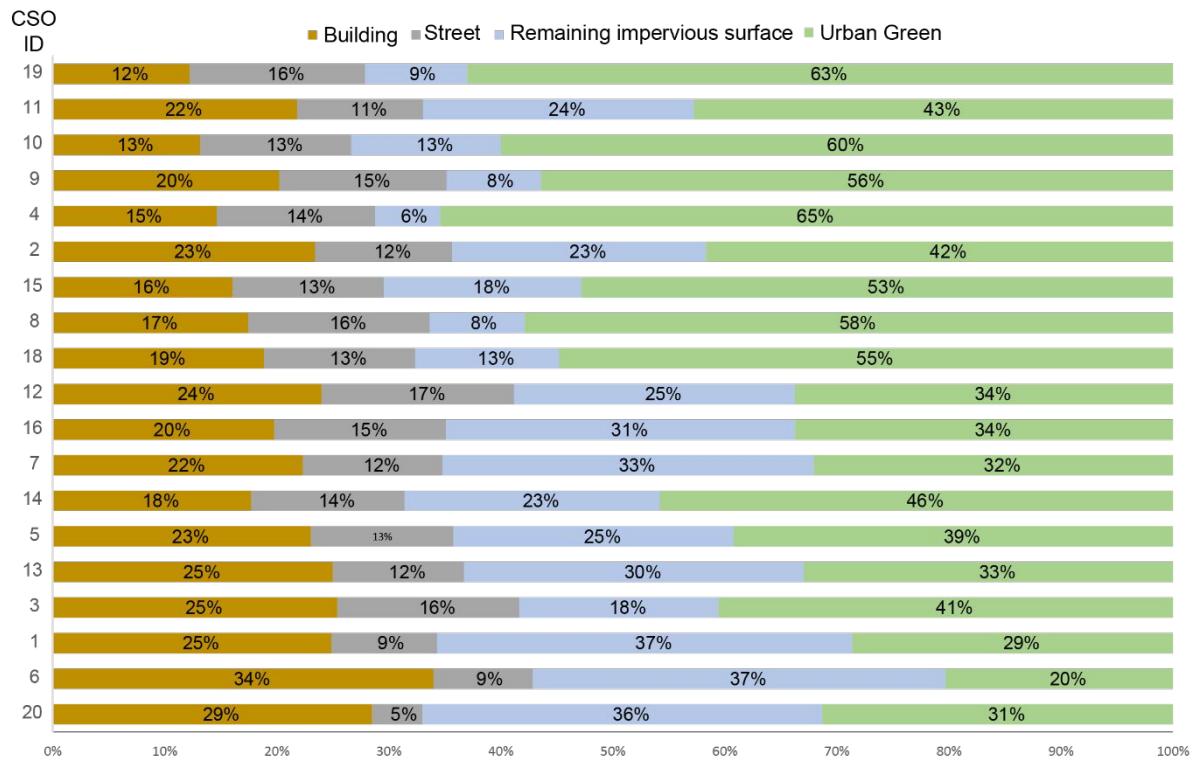


Figure SI 5. Overview on land use distribution in the 20 CSO catchments sorted in ascending order by fraction of industry (CSO ID 20: 100% industry). No data is available for CSO ID 17.

Supporting Information

ANOVA

Table SI 2. ANOVA p-value for the TWA concentration based on triplicates (not mean TWA concentration) to assess how much variability is due to differences between CSO sites in comparison to the variability between events. 1,3-BTH-2-S: 1,3-benzothiazole-2-sulfonate.

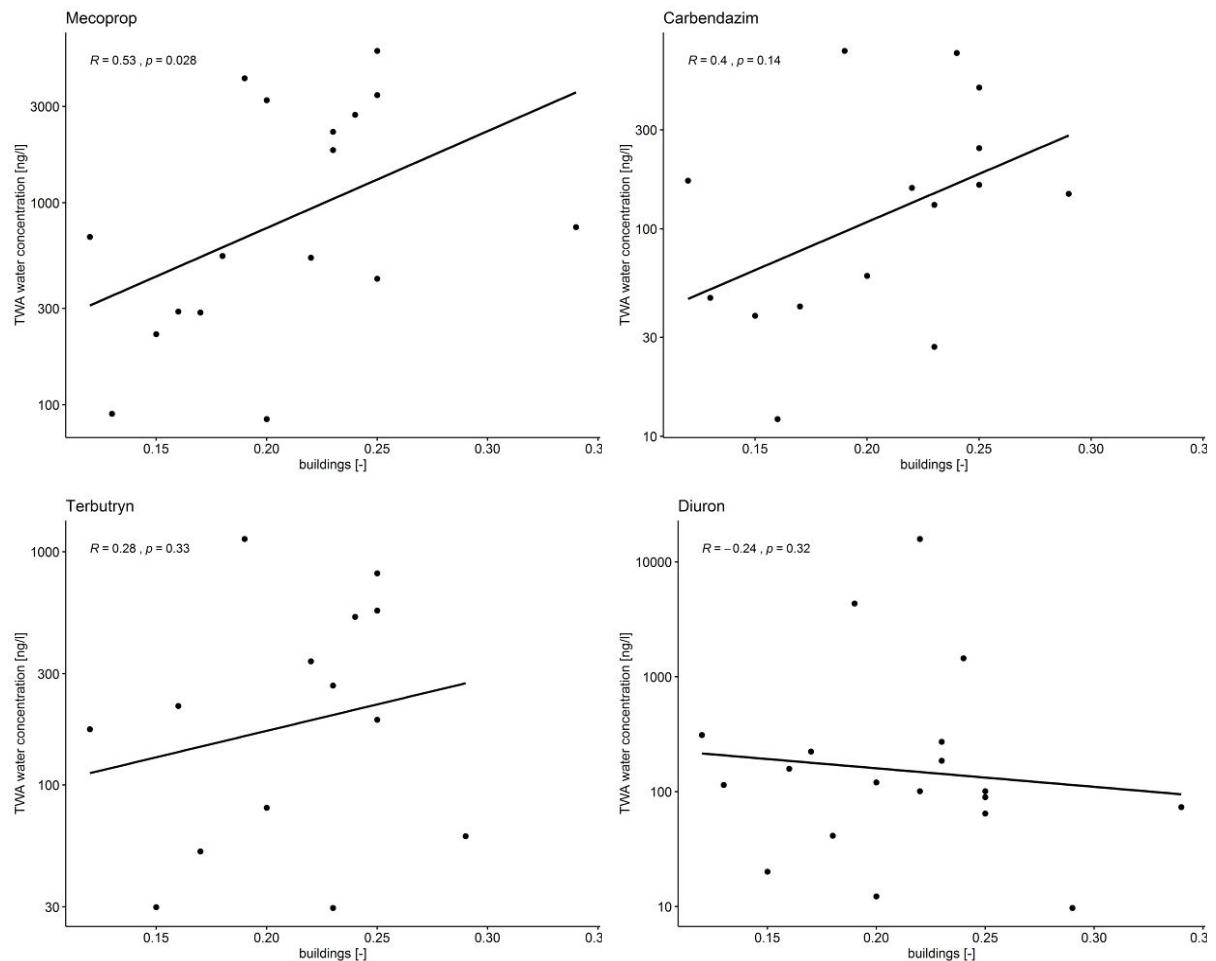
	p-value
1,3-BTH-S	2.8E-12
benzotriazole	5.1E-40
carbamazepine	9.6E-40
carbendazim	1.2E-17
clarithromycin	1.9E-14
diazinon	1.7E-38
diclofenac	4.7E-14
diuron	4.5E-21
mecoprop	4.6E-11
metolachlor	7.2E-18
terbutryn	1.5E-13

Supporting Information

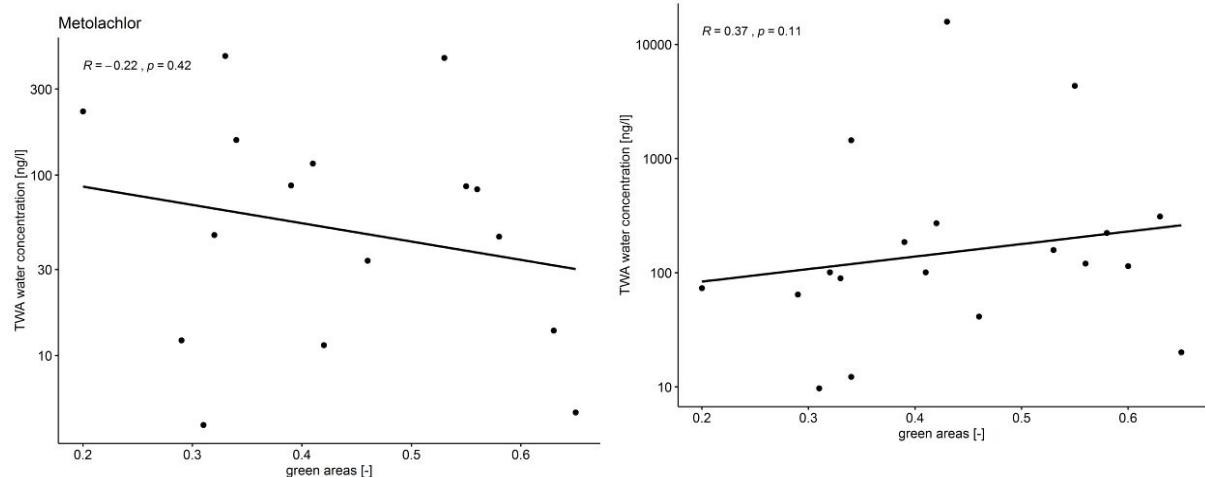
LAND USE

Figure SI 6. Correlations between relative area of land use (building, urban green, street, other impervious areas) and TWA concentrations for 19 CSO sites (for CSO ID 17 no land use data was available). Only micropollutants shown with expected occurrence in selected urban land use category and highest found (if any) correlation

Fraction of buildings

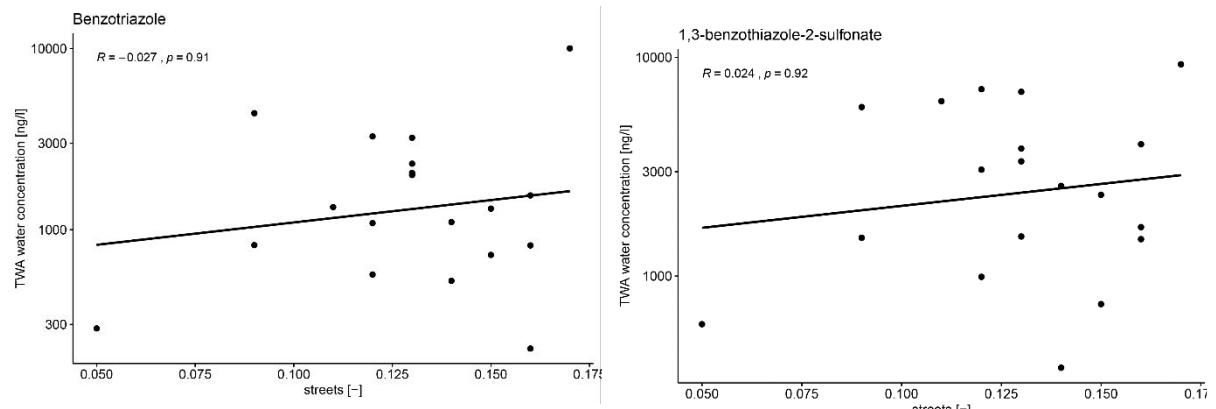


Fraction of urban green

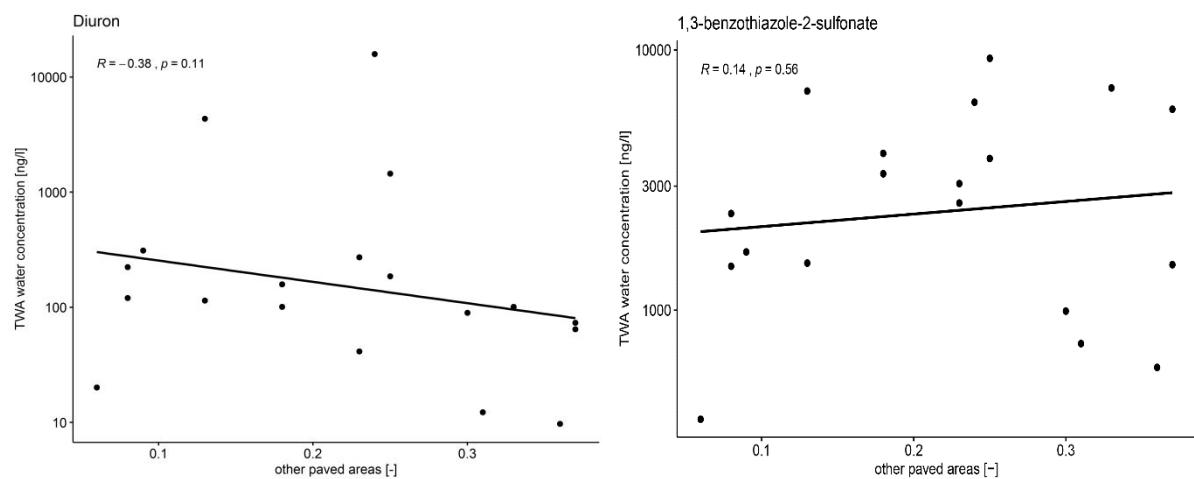


Supporting Information

Fraction of streets



Fraction of other impervious area



Supporting Information

MULTIPLE REGRESSION

Table SI 3. Summary results of multiple regression with survival package¹ in R: model coefficient and p-value for each substance over all 20 catchments. Model: TWA concentration per substance = Intercept + Specific Volume + Duration + Building fraction+ Street fraction + Garden fraction + Remaining impervious surface fraction. Figure SI 5 shows the used relative land use distribution. 2,4-D and MCPA were not considered due to the few available data points. 1,3-BTH-2-S: 1,3-benzothiazole-2-sulfonate.

	Intercept	Specific volume	Duration	Building	Street	Garden	Remaining impervious surfaces
coefficient							
benzotriazole	-1,700	-13	0.023	7,200	51,000	-8,800	0.0
carbamazepine	1,100	0.54	0.065	-3,800	9,200	-3,300	0.0
clarithromycin	-32	0.22	0.0095	-330	1,000	-190	0.0
diclofenac	10,000	-1.1	0.043	-28,000	-4,500	-8,600	0.0
1,3-BTH-S	-1,400	-15	-0.72	13,000	40,000	-5,700	0.0
carbendazim	-981	3.5	-0.18	2004	-469	893	0.0
diazinon	229	0.40	0.00	-1215	645	-236	0.0
diuron	-6,100	-0.62	-0.22	15,000	5,200	6,400	0.0
mecoprop	-13,000	9.7	-0.48	27,000	36,000	6,400	0.0
metolachlor	-580	4.3	-0.016	960	1,300	60	0.0
terbutryn	-1,100	3.0	-0.25	1,800	2,100	680	0.0
p-value							
benzotriazole	0.527	0.023	0.928	0.324	0.000	0.003	NA
carbamazepine	0.155	0.715	0.296	0.043	0.000	0.000	NA
clarithromycin	0.679	0.138	0.062	0.146	0.000	0.021	NA
diclofenac	0.000	0.778	0.791	0.000	0.360	0.000	NA
1,3-BTH-S	0.192	0.123	0.092	0.000	0.000	0.015	NA
carbamazepine	0.155	0.715	0.296	0.043	0.000	0.000	NA
carbendazim	0.108	0.001	0.052	0.194	0.746	0.132	NA
clarithromycin	0.679	0.138	0.062	0.146	0.000	0.021	NA
diazinon	0.019	0.022	0.608	0.000	0.022	0.019	NA
diclofenac	0.000	0.778	0.791	0.000	0.360	0.000	NA
diuron	0.007	0.895	0.281	0.017	0.000	0.008	NA
mecoprop	0.000	0.117	0.153	0.001	0.000	0.034	NA
metolachlor	0.197	0.000	0.685	0.384	0.209	0.889	NA
terbutryn	0.104	0.014	0.018	0.268	0.187	0.280	NA

D. Risk quotient

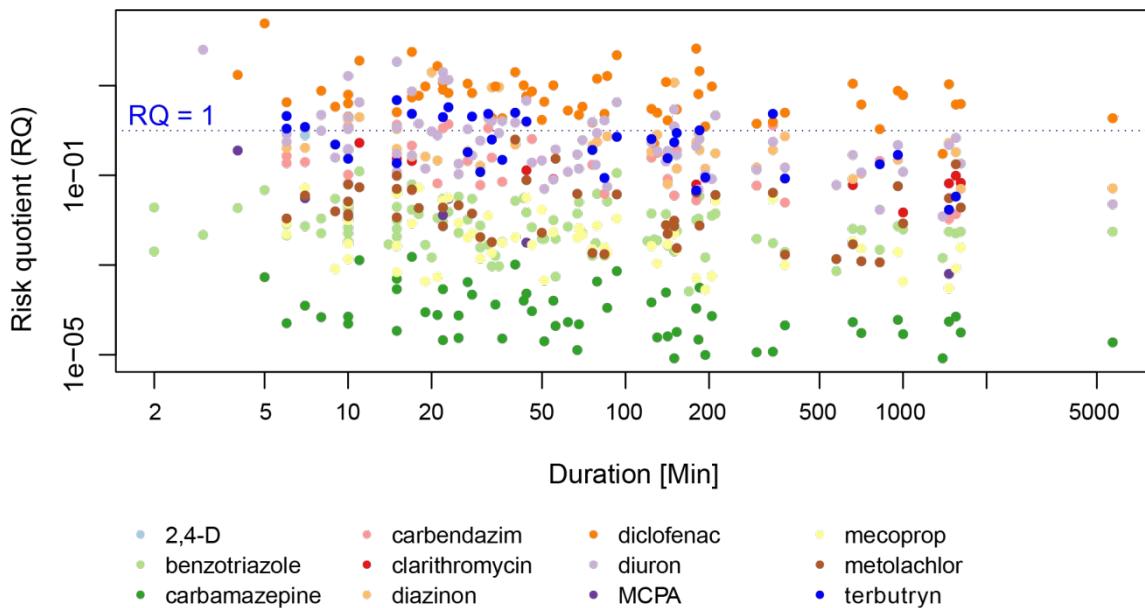


Figure SI 7. Risk quotient (RQ) for all 20 CSO sites (TWA concentration divided by EQS) in comparison to event duration in minutes.

Table SI 4. Number of events with TWA concentration bigger than the EQS (Risk quotient; RQ > 1), only micropollutants shown with RQ > 1. Comparison of TWA concentration calculation strategies: 10% quantile, median and mean correction factor for estimation of $C_{TWA,corr}$ with non-corrected TWA concentration (C_{TWA}).

	carbendazim	clarithromycin	diazinon	diclofenac	diuron	MCPA	terbutryn
90% $C_{TWA,corr}$	7	0	7	60	28	0	13
median $C_{TWA,corr}$	0	0	6	49	14	0	1
mean $C_{TWA,corr}$	0	0	5	41	15	0	1
C_{TWA}	0	1	6	56	21	1	0

E. Chemical analysis

Two methods with comparable performance were applied: i) LC-HR-MS/MS and ii) LVI-LC-QQQ:

- i) LC-HRMS/MS: Full scan MS detection was performed with a QExactive mass spectrometer (resolution R =70,000 at m/z 200) with electrospray ionization (ESI). Data-dependent acquisition was used to acquire five MS/MS (Thermo Fisher Orbitrap MS Q Exactive, resolution R =17,500 at m/z 200) scans using a inclusion list for the target analytes. Samples were analyzed in two separate runs in positive and negative ionization mode. Column: Waters Atlantis T3, 4.6x150 mm, 3 μ m particle size. Large volume injection (LVI): 100 μ L.
- ii) LC-QQQ: Agilent 6495 triple quad mass spectrometer using electrospray as ionization source. Data acquisition was achieved in dynamic MRM mode. Column: Waters Acquity UPLC HSS T3, 3x100mm, 1.8 μ m particle size. Large volume injection (LVI): 100 μ L.

QEXACTIVE: LC-HR-MS/MS

Table SI 5. HPLC gradient used for the chromatographic separation on an Atlantis T3 column. FA: formic acid.

Time [min]	H ₂ O + 0.1% FA [%]	MeOH + 0.1 % FA [%]	Flow [μ L/min]
0.0	95	5	300
1.5	95	5	300
17.5	5	95	300
25.0	5	95	300
25.5	95	5	300
29.5	95	5	300

Supporting Information

Table SI 6. Settings for the QExactive mass spectrometer

Parameter	positive and negative
<i>ESI settings</i>	
Spray voltage	4000 V (positive) / 3000 V (negative)
Capillary temperature	350 °C
S-Lens RF voltage	50 V
Probe Heater Temp	40°C
Sheath Gas Flow (Arb)	40
Aux Gas Flow (Arb)	10
Spare Gas Flow (Arb)	0
<i>Orbitrap settings</i>	
MS1	
Scan range (m/z)	100 - 1000
Mass resolution (m/z 200)	70,000
Automatic Gain Control	5e5
Max Injection time	100 ms
MS2	
Mass resolution (m/z 200)	17,500
Automatic Gain Control	1e5
Max Injection time	80 ms
Acquisition type	data-dependent
Data- dependent loop count	1
Isolation window (m/z)	1
Collision energy normalized	compound-optimized
Dynamic exclusion	2 s

Arb: arbitrary units

ms: milli seconds

s: seconds

Supporting Information

AGILENT G6495A: LVI-LC-QQQ

Table SI 7. HPLC gradient used for the chromatographic separation on an Acquity UPLC HSS T3 column. FA: formic acid.

Time [min]	H ₂ O + 0.1% FA [%]	MeOH + 0.1 % FA [%]	Flow [µL/min]
0.0	100	0	500
18.5	5	95	500
22.0	5	95	500
22.5	100	0	500

Table SI 8. Settings for the Agilent G6495A mass spectrometer

Parameter	Positive and negative
<i>ESI settings</i>	
Gas Temp	250 °C
Gas Flow	15 L/min
Nebulizer	25 psi
Sheath Gas Heater	380 °C
Sheath Gas Flow	12 L/min
Capillary	3500 V (positive) 3000 V (negative)
<i>QQQ settings</i>	
Cycle time	650 ms
Acquisition type	DynamicMRM
MRM Repeats	3
Collision energy normalized	compound-optimized

Table SI 9. Acquisition Method on Agilent G6495A triple quad mass spectrometer

C Name	ISTD	Precursor Ion [m/z]	MS1 Res	Product Ion [m/z]	MS2 Res	Fragment or (V)	Collision Engergy (V)	Ret Time (min)	Polarity
1,3-Benzothiazole-2-sulfonic acid	No	216.0	Unit	134.1	Unit	380	16	9.0	Pos
1,3-Benzothiazole-2-sulfonic acid	No	216.0	Unit	90.1	Unit	380	20	9.0	Pos
2,4-D	No	219.0	Unit	161.0	Unit	380	8	15.3	Neg
2,4-D	No	219.0	Unit	125.1	Unit	380	32	15.3	Neg
2,4-D-D3	Yes	222.0	Unit	164.0	Unit	380	8	15.3	Neg
2,4-D-D3	Yes	222.0	Unit	127.2	Unit	380	28	15.3	Neg
Benzotriazole	No	120.1	Unit	92.1	Unit	380	16	9.5	Pos
Benzotriazole	No	120.1	Unit	65.2	Unit	380	20	9.5	Pos
Benzotriazole-D4	Yes	124.1	Unit	96.1	Unit	380	18	9.4	Pos
Benzotriazole-D4	Yes	124.1	Unit	69.1	Unit	380	26	9.4	Pos
Carbamazepine	No	237.1	Unit	194.0	Unit	380	22	14.3	Pos
Carbamazepine	No	237.1	Unit	193.1	Unit	380	38	14.3	Pos
Carbamazepine-D8	Yes	245.2	Unit	202.1	Unit	380	30	14.2	Pos
Carbamazepine-D8	Yes	245.2	Unit	201.2	Unit	380	34	14.2	Pos
Carbendazim	No	192.1	Unit	160.1	Unit	380	20	8.1	Pos
Carbendazim	No	192.1	Unit	65.1	Unit	380	56	8.1	Pos
Carbendazim-D4	Yes	196.1	Unit	164.1	Unit	380	24	8.0	Pos

Supporting Information

C Name	ISTD	Precursor	MS1	Product	MS2 Res	Fragment	Collision	Ret	Polarity
		Ion [m/z]	Res	Ion [m/z]	or	(V)	Engergy	Time	(min)
							(V)		
Carbendazim-D4	Yes	196.1	Unit	69.2	Unit	380	60	8.0	Pos
Clarithromycin	No	748.5	Unit	590.4	Unit	380	18	15.3	Pos
Clarithromycin	No	748.5	Unit	158.1	Unit	380	26	15.3	Pos
Clarithromycin-D3	Yes	751.5	Unit	593.4	Unit	380	14	15.3	Pos
Clarithromycin-D3	Yes	751.5	Unit	161.3	Unit	380	30	15.3	Pos
Diazinon	No	305.1	Unit	169.1	Unit	380	20	18.1	Pos
Diazinon	No	305.1	Unit	153.2	Unit	380	20	18.1	Pos
Diazinon-D10	Yes	315.2	Unit	170.1	Unit	380	28	18.1	Pos
Diazinon-D10	Yes	315.2	Unit	154.1	Unit	380	24	18.1	Pos
Diclofenac	No	296.0	Unit	249.9	Unit	380	10	17.6	Pos
Diclofenac	No	296.0	Unit	214.1	Unit	380	30	17.6	Pos
Diclofenac	No	296.0	Unit	215.0	Unit	380	20	17.6	Pos
Diclofenac-D4	Yes	300.1	Unit	254.0	Unit	380	10	17.64	Pos
Diclofenac-D4	Yes	300.1	Unit	219.1	Unit	380	20	17.64	Pos
Diclofenac-D4	Yes	300.1	Unit	218.0	Unit	380	34	17.64	Pos
Diuron	No	233.0	Unit	72.2	Unit	380	28	15.35	Pos
Diuron	No	233.0	Unit	46.3	Unit	380	20	15.35	Pos
Diuron-D6	Yes	239.1	Unit	78.1	Unit	380	40	15.3	Pos
Diuron-D6	Yes	239.1	Unit	52.4	Unit	380	20	15.3	Pos
MCPA	No	199.0	Unit	141.1	Unit	380	20	15.6	Neg
MCPA 37Cl	No	201.0	Unit	143.0	Unit	380	12	15.6	Neg
MCPA-D3	Yes	202.0	Unit	144.0	Unit	380	16	15.6	Neg
MCPA 37Cl-D3	Yes	204.0	Unit	146.0	Unit	380	12	15.6	Neg
Metolachlor	No	284.1	Unit	252.1	Unit	380	12	17.5	Pos
Metolachlor	No	284.1	Unit	176.2	Unit	380	28	17.5	Pos
Metolachlor-D6	Yes	290.2	Unit	258.2	Unit	380	16	17.5	Pos
Metolachlor-D6	Yes	290.2	Unit	182.2	Unit	380	28	17.5	Pos
Terbutryn	No	242.2	Unit	186.1	Unit	380	20	15.3	Pos
Terbutryn	No	242.2	Unit	68.1	Unit	380	52	15.3	Pos
Terbutryn-D5	Yes	247.2	Unit	191.1	Unit	380	20	15.2	Pos
Terbutryn-D5	Yes	247.2	Unit	69.1	Unit	380	48	15.2	Pos

Supporting Information

Table SI 10. Relative recoveries and LOQs for the different measurement series. Relative Recovery: recovery of analyte amount spiked into the sample considering the background concentration of the unspiked sample. LOQ determined with signal-to-noise ratio of 10. The LOQ of the less sensitive DRM transition is reported

Used mass spectrometer & injection/enrichment mode	Thermo Scientific QExactive Plus, direct injection*	Agilent 6495 TQ, direct injection**		
	Rel. recovery [%]	LOQ [µg/L]	Rel. recovery [%]	LOQ [ng/L]
1,3-Benzothiazole-2-sulfonic acid	130-202	1-2	108-134	13-30
2,4-D	108-120	1.9-2	107-128	5-20
Benzotriazole	79-126	1-2	99-113	30-50
Carbamazepine	113-135	0.3-2	107-124	1.4-8
Carbendazim	103-116	0.3-0.5	79-96	0.6-2
Clarithromycin	115-131	1.8-2	100-150	0.5-13
Diazinon	112-116	0.3-0.7	100-137	0.6-6
Diclofenac	94-116	0.7-1	102-103	1-13
Diuron	101-119	0.6-2	114-116	1.9-17
MCPA	92-111	0.8-1.5	75-114	30-40
Mecoprop	117-130	0.7-2	74-102	2.6-12
Metolachlor	115-128	0.6-1	99-112	5.3
Terbutryn	107-122	1.4	99-109	0.6-11

* Three measurement series were conducted with this setup, therefore a range of relative recovery and LOQ values is given

** Two measurement series were conducted with this setup, therefore a range of relative recovery and LOQ values is given

Table SI 11. Quality control: measured concentration and relative recovery for three external reference standards measured in one measurement sequence with Agilent 6495 TQ, direct injection. Used reference standards: 1) Neochema Pharma – Mix 17, STD-Nr. EGT-1552, 2) Dr. Ehrenstorfer Pesticide-Mix 1284, Product identification: 18001284, Lot No. 87269, 3) Dr. Ehrenstorfer Pesticide-Mix 1285, Product identification: 18001285, Lot No. 80529AL.

Compound	Measured concentration			Relative recovery		
	Neochema	Ehrenstorfer 1284	Ehrenstorfer 1285	Neomix	Ehrenstorfer 1284	Ehrenstorfer 1285
	ng/L	ng/L	ng/L	%	%	%
2,4-D			20,69			103
Benzotriazole	9,244*			92		
Carbamazepine	2,070			104		
Carbendazim		1,716			86	
Clarithromycin	1,883			94		
Diazinon		1,822			91	
Diclofenac	2,027			101		
Diuron		2,180			109	
MCPA			2,104			105
Mecoprop	1,863		1,686	93		84
Metolachlor		1,996			100	

*Concentration for benzotriazole factor 5 higher (10,000 ng/L) in external reference standard

Supporting Information

Table SI 12. Measured concentrations [ng/disk] in field blinds. The field blinds were treated like all other passive sampler disks: preparation in laboratory and field, mounted but not exposed, stored and extracted. Field blinds were taken for different sites.

CSO ID	2,4-D	1,3-BTH-2-S	Benzotriazole	Carbamazepine	Carbendazim	Clarithromycin	Diazinon	Diclofenac	Diuron	MCPA	Mecoprop	Metolachlor	Terbutryne
5	NA	NA	0.5	NA	NA	0.8	NA	NA	NA	NA	NA	NA	NA
7	NA	NA	0.3	NA	NA	0.8	NA	NA	NA	NA	NA	NA	NA
1	NA	NA	0.2	NA	NA	0.9	NA	NA	NA	NA	NA	NA	NA
15	NA	1.8	0.6	NA	NA	0.7	NA	NA	NA	NA	NA	NA	NA
12	NA	1.5	0.3	NA	NA	0.8	NA	NA	NA	NA	NA	NA	NA
13	NA	1.7	0.3	NA	NA	0.8	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	0.5	NA	NA	1.8	NA	NA	NA	NA	NA	NA	NA
17	NA	NA	0.3	NA	NA	0.8	NA	NA	NA	NA	NA	NA	NA

References

- Therneau, T. *A Package for Survival Analysis in S*, version 2.38; <https://CRAN.R-project.org/package=survival>, 2015.