Supporting Information

High levels of antibiotics released by a pharmaceutical manufacturer negatively impacted wastewater treatment plant performance

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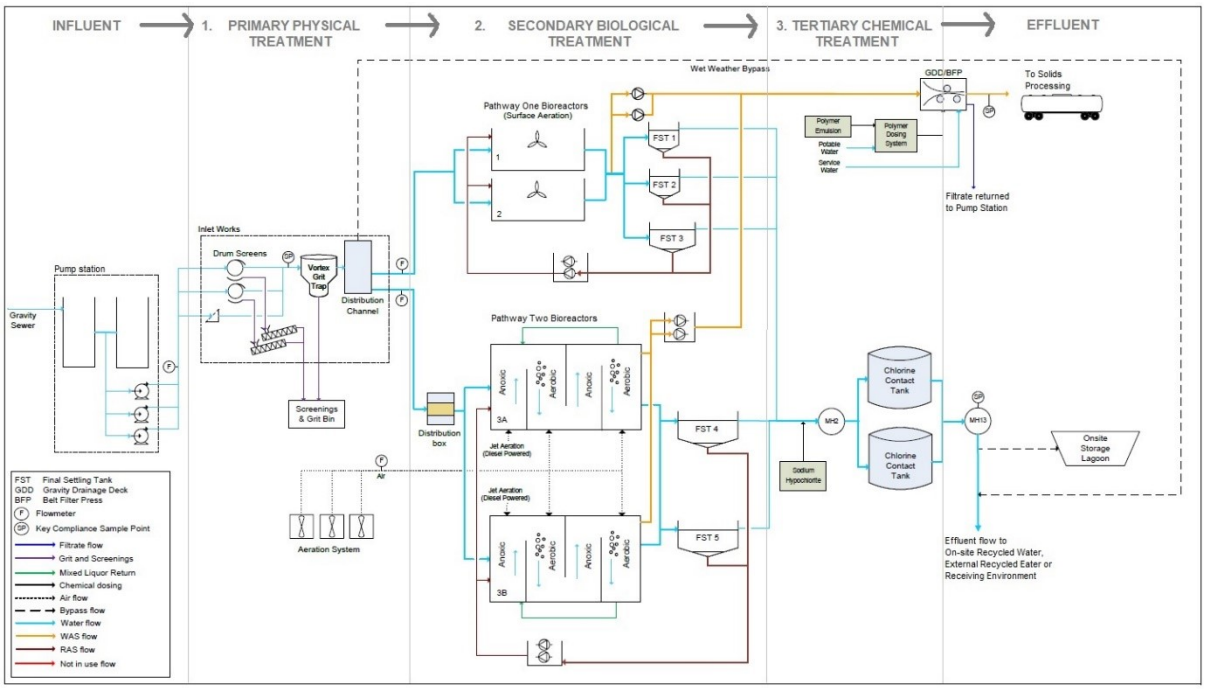

Figure S1: WWTP process flow diagram. The influent flows in through primary physical treatment, secondary biological treatment and tertiary chemical treatment before being discharged as effluent.

Materials and Methods SI - Chemical standard preparation and analytical method

Stock solutions of all analytes and IS were prepared to 1000 mg/L in methanol (MeOH, HPLC-grade, Merck, Darmstadt, Germany) or dimethyl sulfoxide (DMSO, Merck, Darmstadt, Germany) and stored at −20 °C. Amoxicillin, penicillin V, meropenem, gentamicin, neomycin, and colistin were prepared from standard powders 48 h before experimentation, due to stability considerations of solutions. Formic acid (FA) was procured from VWR Chemicals (Tingalpa, QLD, Australia), while hydrochloric acid (HCl) was procured from Merck (Kilsyth, VIC, Australia). Ultra-pure water of 18.2 MΩ cm^{-1} purity was obtained from a Milli-Q system (Merck Millipore, Bedford, MA, USA). Regenerated cellulose syringe filters (0.2 µm RC filters) were procured from Agilent (Mulgrave, VIC, Australia).

An ultra-high performance liquid chromatography system (Nexera series-LC 40, Shimadzu, Kyoto, Japan) coupled to a tandem mass spectrometer (SCIEX Triple Quad 7500 System, AB SCIEX, Framingham, MA, USA) was used for sample analysis. Two transitions were used for each analyte with the optimised collision energy (CE) and collision exit cell potential (CXP) for each transition. Entrance potential was set to +10 V for all positive mode transitions and -10 V for all negative mode transitions. The ion source temperature was set at 550 °C (accommodating an LC flow rate of 0.4 mL min^{-1}) and electrospray ionisation voltages (ISV) were optimised for each analyte, while the ion source gases 1 and 2 were set at 60 psi and curtain gas at 40 psi. Q0 dissociation (Q0D) was optimised for each transition. The mass spectrometer was run in scheduled multiple reaction monitoring (sMRM) mode and in switching positive and negative ion modes. Dwell times were used as default values of the SCIEX OS 2.1.6 software (AB SCIEX, Framingham, MA, USA). Instrument details and parameters of each transition are shown in Li et al. (2023)[25].

A Hypersil Gold C18 selectivity column (100 × 2.1 mm, 1.9 µm, 175 Å, Thermo Fisher Scientific, Waltham, MA, USA) with a Gemini NX-C18 guard column (4 × 2 mm, Phenomenex, Torrance, CA, USA) was used to achieve chromatographic separation. A Kinetex EVO C18 column (30 × 2.1 mm, 5 µm, 100 Å, Phenomenex, Torrance, CA, USA) was used as a pre-injection column. Mobile phases consisted of 95:5 (v/v) Milli-Q water: methanol with 0.2% formic acid (mobile phase A) and 95:5 (v/v) methanol: Milli-Q water with 0.2% formic acid (mobile phase B). The flow rate was set at 0.4 mL min^{-1}.

Mobile phase B was initially set at 5%, followed by a linear increase to 40% over 4 min, a linear increase to 100% over 3 min, held for 3 min, finally returned to 5% over 0.1 min, and kept steady for 2 min to equilibrate the system. The total run time was 12 min. Data were acquired and processed using SCIEX OS 2.1.6.

Table S1 Chemical information, ordered by class groupings.

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
1	Amoxicillin	AMX	Penicillin	parent	Human/Vet	26787-78-0	Trihydrate	365.10	C16H19N3O5S	HPC
2	Amoxicilloic acid	AMXa	Penicillin	TP		42947-63-7	Sodium salt	383.12	C16H21N3O6S	TRC
3	Ampicillin	AMP	Penicillin	parent	Human/Vet	69-53-4	Trihydrate	349.11	C16H19N3O4S	HPC
4	Cloxacillin	CLX	Penicillin	parent	Vet	61-72-3	Sodium salt	435.07	C19H18ClN3O5S	TRC
5	Dicloxacillin	DLX	Penicillin	parent	Human	3116-76-5	Sodium Monohydrate	469.03	C19H17Cl2N3O5S	LGC
6	Flucloxacillin	FLX	Penicillin	parent	Human	5250-39-5	Sodium salt	453.06	C19H17ClFN3O5S	TRC
7	Penicillin V	PenV	Penicillin	parent	Human	87-08-1		350.09	C16H18N2O5S	TRC
8	Penicillin V acid	PenVa	Penicillin	TP		1049-84-9	Hydrate	368.10	C16H20N2O6S	Biosynth
9	Piperacillin	PIP	Penicillin	parent	Human	61477-96-1	Sodium salt	517.16	C23H27N5O7S	HPC
10	Cefaclor	CFC	Cephalosporin	parent	Human	53994-73-3	Hydrate	367.04	C15H14ClN3O4S	TRC
11	Cefalexin	CFX	Cephalosporin	parent	Human/Vet	15686-71-2	Hydrate	347.09	C16H17N3O4S	TRC
12	Cephalothin	CFL	Cephalosporin	parent	Human	153-61-7		396.05	C16H16N2O6S2	TRC
13	Cefazolin	CFZ	Cephalosporin	parent	Human	25953-19-9		454.03	C14H14N8O4S3	TRC
14	Cefepime	CFP	Cephalosporin	parent	Human	88040-23-7	Dihydrochloride monohydrate	480.12	C19H24N6O5S2	HPC
15	Ceftiofur	CTF	Cephalosporin	parent	Vet	80370-57-6		523.03	C19H17N5O7S3	TRC

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
16	Ceftriaxone	CTR	Cephalosporin	parent	Human	73384-59-5	Disodium salt hemiheptahydrate	554.05	C18H18N8O7S3	HPC
17	Cefuroxime	CRX	Cephalosporin	parent	Human/Vet	55268-75-2	Sodium salt	424.07	C16H16N4O8S	LGC
18	Ciprofloxacin	CIP	Quinolone	parent/TP	Human	85721-33-1	Hydrochloride hydrate	331.13	C17H18FN3O3	HPC
19	Desethylene ciprofloxacin	deCIP	Quinolone	TP		103222-12-4	Hydrochloride	305.12	C15H16FN3O3	TRC
20	Enrofloxacin	ENR	Quinolone	parent	Vet	93106-60-6		359.16	C19H22FN3O3	HPC
21	Moxifloxacin	MOX	Quinolone	parent	Human	151096-09-2		401.18	C21H24FN3O4	TRC
22	Moxifloxacin sulfate	MOX-SO_4	Quinolone	TP		234080-64-9	Disodium salt	481.13	C21H24FN3O7S	TRC
23	Norfloxacin	NOR	Quinolone	parent	Human	70458-96-7		319.13	C16H18FN3O3	TRC
24	Desethylene norfloxacin	deNOR	Quinolone	TP		75001-77-3	Hydrochloride	293.12	C14H16FN3O3	TRC
25	Ofloxacin	OFL	Quinolone	parent	Human	82419-36-1		361.14	C18H20FN3O4	LGC
26	Desmethyl ofloxacin	dmOFL	Quinolone	TP		82419-52-1		347.13	C17H18FN3O4	TRC
27	Sarafloxacin	SAR	Quinolone	parent	Vet[a]	98105-99-8		385.12	C20H17F2N3O3	TRC
28	Oxolinic acid	OXO	Quinolone	parent	Vet[a]	14698-29-4		261.06	C13H11NO5	TRC
29	Sulfachlorpyridazine	SCP	Sulfonamide	parent	Human/Vet[a]	80-32-0		284.01	C10H9ClN4O2S	HPC
30	Sulfadiazine	SDZ	Sulfonamide	parent	Human/Vet	68-35-9		250.05	C10H10N4O2S	HPC
31	Acetyl sulfadiazine	aSDZ	Sulfonamide	TP		127-74-2		292.06	C12H12N4O3S	TRC
32	Sulfadimethoxine	SDM	Sulfonamide	parent	Human/Vet[a]	122-11-2		310.07	C12H14N4O4S	HPC
33	Sulfamerazine	SMR	Sulfonamide	parent	Vet	127-79-7		264.07	C11H12N4O2S	HPC

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
34	Acetyl sulfamerazine	aSMR	Sulfonamide	TP		127-73-1		306.08	C13H14N4O3S	TRC
35	Sulfamethazine	SMZ	Sulfonamide	parent	Vet	57-68-1		278.08	C12H14N4O2S	HPC
36	Acetyl sulfamethazine	aSMZ	Sulfonamide	TP		100-90-3		320.09	C14H16N4O3S	TRC
37	Sulfamethizole	SMT	Sulfonamide	parent	Human/Vet [a]	144-82-1		270.02	C9H10N4O2S2	HPC
38	Sulfamethoxazole	SMX	Sulfonamide	parent	Human	723-46-6		253.05	C10H11N3O3S	HPC
39	Acetyl sulfamethoxazole	aSMX	Sulfonamide	TP		21312-10-7		295.06	C12H13N3O4S	TRC
40	Sulfapyridine	SPY	Sulfonamide	parent/TP	Human/Vet [a]	144-83-2		249.06	C11H11N3O2S	LGC
41	Acetyl sulfapyridine	aSPY	Sulfonamide	TP		19077-98-6		291.07	C13H13N3O3S	TRC
42	Sulfasalazine	SLZ	Sulfonamide	parent	Human/Vet	599-79-1		398.07	C18H14N4O5S	USP
43	Sulfathiazole	STZ	Sulfonamide	parent	Vet	72-14-0		255.01	C9H9N3O2S2	HPC
44	Acetyl sulfathiazole	aSTZ	Sulfonamide	TP		127-76-4		297.02	C11H11N3O3S2	TRC
45	Azithromycin	AZI	Macrolide	parent	Human	83905-01-5		748.51	C38H72N2O12	TRC
46	Desmethyl azithromycin	dmAZI	Macrolide	TP		172617-84-4		734.49	C37H70N2O12	TRC
47	Clarithromycin	CLA	Macrolide	parent	Human	81103-11-9		747.48	C38H69NO13	TRC
48	Desmethyl Clarithromycin	dmCLA	Macrolide	TP		101666-68-6		733.46	C37H67NO13	TRC
49	Erythromycin-H_2O	ERY-18	Macrolide	TP		23893-13-2		715.45	C37H65NO12	TRC
50	Erythromycin	ERY	Macrolide	parent	Human/Vet	114-07-8		733.46	C37H67NO13	TRC

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
51	Desmethyl erythromycin	dmERY	Macrolide	TP		992-62-1		719.45	C36H65NO13	TRC
52	Roxithromycin	ROX	Macrolide	parent	Human	80214-83-1		836.52	C41H76N2O15	TRC
53	Descladinose roxithromycin	dcROX	Macrolide	TP		214902-82-6		678.43	C33H62N2O12	TRC
54	Spiramycin I #	SP-I	Macrolide	parent	Human/Vet	24916-50-5		842.51	C43H74N2O14	TRC
55	Spiramycin III #	SP-III	Macrolide	parent	Human/Vet	24916-52-7		898.54	C46H78N2O15	TRC
56	Monoacetyl spiramycin II #	maSP-II	Macrolide	parent	Human/Vet	87111-42-0		926.54	C47H78N2O16	TRC
57	Diacetyl spiramycin II #	daSP-II	Macrolide	parent	Human/Vet	110101-92-3		968.55	C49H80N2O17	TRC
58	Monoacetyl spiramycin III #	maSP-III	Macrolide	parent	Human/Vet	112501-15-2		940.55	C48H80N2O16	TRC
59	Diacetyl spiramycin III #	daSP-III	Macrolide	parent	Human/Vet	99664-83-2		982.56	C50H82N2O17	TRC
60	Spiramycin II #	SP-II	Macrolide	parent	Human/Vet	24916-51-6		884.52	C45H76N2O15	TRC
61	Tilmicosin	TIL	Macrolide	parent	Vet	108050-54-0		868.57	C46H80N2O13	TRC
62	Tulathromycin	TUL	Macrolide	parent	Vet	217500-96-4		805.57	C41H79N3O12	TRC
63	Tylosin	TYL	Macrolide	parent	Vet	1401-69-0		915.52	C46H77NO17	HPC
64	Virginiamycin M1	VIR-M	Macrolide	parent	Vet	21411-53-0		525.25	C28H35N3O7	TRC
65	Virginiamycin S1	VIR-S	Macrolide	parent	Vet	23152-29-6		823.35	C43H49N7O10	TRC
66	Chlortetracycline	CTC	Tetracycline	parent	Vet	57-62-5	Hydrochloride	478.11	C22H23ClN2O8	TRC
67	Demeclocycline	DMC	Tetracycline	parent	Human[a]	127-33-3	Hydrochloride	464.10	C21H21ClN2O8	LGC

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
68	Doxycycline	DOX	Tetracycline	parent	Human/Vet	564-25-0	Hyclate	444.15	C22H24N2O8	TRC
69	Minocycline	MIN	Tetracycline	parent	Human	10118-90-8	Hydrochloride dihydrate	457.18	C23H27N3O7	LGC
70	Oxytetracycline	OTC	Tetracycline	parent	Vet	79-57-2	Hydrochloride	460.15	C22H24N2O9	TRC
71	Tetracycline	TET	Tetracycline	parent	Human/Vet	60-54-8		444.15	C22H24N2O8	TRC
72	Fluconazole	FCZ	Azole	parent	Human/Vet	86386-73-4		306.10	C13H12F2N6O	TRC
73	Fluconazole N-oxide	FNO	Azole	TP		1997296-62-4		322.10	C13H12F2N6O2	TRC
74	Metronidazole	MTZ	Azole	parent	Human/Vet	443-48-1		171.06	C6H9N3O3	HPC
75	Hydroxy metronidazole	hMTZ	Azole	TP		4812-40-2		187.06	C6H9N3O4	TRC
76	Clindamycin	CLI	Lincosamide	parent	Human/Vet	18323-44-9	Hydrochloride	424.18	C18H33ClN2O5S	TRC
77	Clindamycin sulfoxide	CSO	Lincosamide	TP		22431-46-5		440.17	C18H33ClN2O6S	TRC
78	Lincomycin	LIN	Lincosamide	parent	Human/Vet	154-21-2	Hydrochloride monohydrate	406.21	C18H34N2O6S	LGC
79	Rifampicin	RFP	Rifamycin	parent	Human	13292-46-1		822.41	C43H58N4O12	HPC
80	Rifaximin	RFX	Rifamycin	parent	Human	80621-81-4		785.35	C43H51N3O11	LGC
81	Desacetyl rifaximin	daRFX	Rifamycin	TP		80621-88-1		743.34	C41H49N3O10	TRC
82	Chloramphenicol	CHL	Amphenicol	parent	Human/Vet	56-75-7		322.01	C11H12Cl2N2O5	Sigma-Aldrich
83	2-amino-1-(4-nitrophenyl)-1,3-	ANP	Amphenicol	TP		716-61-0		212.08	C9H12N2O4	Biosynth

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
	propanediol									
84	Florfenicol	FF	Amphenicol	parent	Vet	73231-34-2		357.00	C12H14Cl2FNO4S	TRC
85	Florfenicol amine	FFA	Amphenicol	TP		76639-93-5		247.07	C10H14FNO3S	HPC
86	Tazobactam	TAZ	β-lactamase inhibitor	parent	Human	89786-04-9		300.05	C10H12N4O5S	TRC
87	Tazobactam M1 metabolite	TAZ-M	β-lactamase inhibitor	TP		118175-11-4		248.06	C7H12N4O4S	Biosynth
88	Trimethoprim	TMP	Diaminopyrimidine	parent	Human/Vet	738-70-5		290.14	C14H18N4O3	HPC
89	Hydroxy trimethoprim	hTMP	Diaminopyrimidine	TP		112678-48-5		306.13	C14H18N4O4	TRC
90	Linezolid	LZD	Oxazolidinone	parent	Human	165800-03-3		337.14	C16H20FN3O4	TRC
91	PNU 142586	PNU	Oxazolidinone	TP		189038-36-6	Sodium salt	353.14	C16H20FN3O5	TRC
92	Gentamicin C1 #	GEN1	Aminoglycoside	parent	Human/Vet	25876-10-2	Sulfate	477.32	C21H43N5O7	HPC
93	Gentamicin C1a #	GEN1a	Aminoglycoside	parent	Human/Vet	26098-04-4	Sulfate	449.28	C19H39N5O7	HPC
94	Gentamicin C2,C2a,C2b #	GEN2	Aminoglycoside	parent	Human/Vet	25876-11-3, 59751-72-3, 52093-21-7	Sulfate	463.30	C20H41N5O7	HPC
95	Neomycin	NEO	Aminoglycoside	parent	Human/Vet	119-04-0	Acetate	614.31	C23H46N6O13	TRC
96	Colistin A #	COL-A	Cyclic polypeptide	parent/TP	Human	7722-44-3	Sulfate	1168.77	C53H100N16O13	LGC
97	Colistin B #	COL-B	Cyclic polypeptide	parent/TP	Human	7239-48-7	Sulfate	1154.75	C52H98N16O13	LGC
98	Fusidic acid	FA	Fusidane	parent	Human/V	6990-06-3		516.35	C31H48O6	TRC

	Name	Abbr.	Class A	Class B	Class C	CAS No.	Salt form	M.I. mass	Formula	Supplier
99	Meropenem	MER	Carbapenem	parent	et Human	96036-03-2	Trihydrate	383.15	C17H25N3O5S	HPC
100	Nitrofurantoin	NIT	Nitrofuran	parent	Human	67-20-9		238.03	C8H6N4O5	HPC
101	Salinomycin	SAL	Ionophore	parent	Vet	53003-10-4		750.49	C42H70O11	TRC
102	Vancomycin	VAN	Glycopeptide	parent	Human	1404-90-6		1447.43	C66H75Cl2N9O24	TRC

M.I., Mass, monoisotopic mass; TP, antimicrobial transformation product; Human, human antimicrobial; Vet, veterinary antimicrobial.

Compounds containing isomers were not separated in this method.

#, One standard used for all forms within the drug complex.

&, Salt corrections considered in all calculations, i.e., analysis of the free base.

[a], Antimicrobial not registered in Australia for any purpose of use

Li J, Shimko KM, He C, Patterson B, Bade R, Shiels R, et al. Direct injection liquid chromatography-tandem mass spectrometry as a sensitive and high-throughput method for the quantitative surveillance of antimicrobials in wastewater. Science of the Total Environment 2023; 900: 165825.

Quality Assurance/Quality Control Results

Table S2: Calibration and quality control results.

Name	Abbr.	Cali_Lower_Range	Cali_Upper_Range	LOQ_ppb	R^2	QAQC_1ppb	QAQC_10ppb
Ampicillin	AMP	0.05	20	0.05	0.998	1.20	9.95
Cefalexin	CFX	0.01	20	0.01	0.996	1.20	10.73
Ciprofloxacin	CIP	0.1	20	0.1	0.993	0.81	9.45
Norfloxacin	NOR	0.05	20	0.05	0.994	0.78	10.09
Ofloxacin	OFL	0.05	10	0.05	0.990	/	0.74
Sulfadiazine	SDZ	0.01	10	0.01	0.995	1.24	11.01
Sulfamethoxazole	SMX	0.005	20	0.005	0.993	/	12.23
Sulfapyridine	SPY	0.01	10	0.01	0.994	1.09	11.47
Erythromycin-H2O	ERY-18	0.01	10	0.01	0.995	1.13	11.96
Tilmicosin	TIL	0.01	20	0.01	0.990	0.46	8.64
Doxycycline	DOX	0.05	20	0.05	0.991	0.79	10.01
Tetracycline	TET	0.05	5	0.05	0.964	0.96	9.84
Fluconazole	FCZ	0.01	10	0.01	0.993	0.90	9.66
Metronidazole	MTZ	0.01	10	0.01	0.995	0.70	9.46
Clindamycin	CLI	0.01	10	0.01	0.997	0.97	8.52
Lincomycin	LIN	0.01	5	0.01	0.993	0.94	8.78
Rifaximin	RFX	0.01	20	0.01	0.996	1.08	12.25
Trimethoprim	TMP	0.01	20	0.01	0.996	/	8.17
Linezolid	LZD	0.05	10	0.05	0.997	0.88	9.54

/; background concentration was too high to get the concentration of QC spiking at 1ppb.

Table S3: Raw antibiotics data from influent analysis.

Sample Name	Ampicillin	Cephalexin	Ciprofloxacin	Clindamycin	Norfloxacin	Sulfadiazine	Sulfapyridine	Erythromycin-18	Tilmicosin	Doxycycline	Tetracycline
16/11/2023			0.373	0.018	0.114	<LOQ	0.865	0.029		0.338	
23/11/2023			0.792	0.031	0.133	<LOQ	0.886	0.017		0.990	
30/11/2023			0.324	0.015	0.097	<LOQ	0.727	0.024		0.468	
7/12/2023			0.429	0.015	0.084	<LOQ	0.531	0.017		3.212	
8/12/2023		0.011	1.665	0.017			0.267	0.017		1.602	
11/12/2023		0.031	2.393	0.024	0.071	<LOQ	0.225	0.024	0.057	1.063	
14/12/2023			0.130	0.020	0.069	<LOQ	0.500	0.025	0.707	2.136	
14/12/2023			1.747	0.026	0.065		0.516	0.014	7.998	2.787	
21/12/2023			0.419	0.021	0.080		0.608	0.027	3.385	12.600	0.161
28/12/2023	0.183	0.185	0.300	0.027	0.092	<LOQ	0.515	0.025	0.804	3.646	0.410
28/12/2023 duplicate	0.183	0.181	0.290	0.029	0.085	<LOQ	0.537	0.024	0.842	3.585	0.462

Concentration is at μg/L.
Cells without value indicate <LODs (limits of detection).
LOQ, limit of quantification.

Results for **10** times diluted samples.

Sample Name	Fluconazole	Metronidazole	Lincomycin	Rifaximin	Linezolid	Ofloxacin	Sulfamethoxazole	Trimethoprim
16/11/2023	0.288	0.016				12.800	2.782	0.746
23/11/2023	0.332					24.440	3.717	2.013
30/11/2023	0.231					10.710	33.780	7.554
7/12/2023	0.282	0.089	0.085	0.088		10.590	59.010	18.622
8/12/2023	0.148	1.035	0.014			9.725	6.749	0.993
11/12/2023	0.122	0.021	0.013			4.211	3.513	0.359
14/12/2023	1.116	2.206	<LOQ		<LOQ	4.356	22.630	3.905
14/12/2023	0.192	3.827	0.021			6.280	20.060	6.844

21/12/2023	0.237		5.262	20.765	3.294
28/12/2023	0.370	0.664	4.936	0.724	0.280
28/12/2023 duplicate	0.403	0.696	4.908	0.786	0.305

Concentration is at μg/L.
Cells without value indicate <LODs (limits of detection).
LOQ, limit of quantification.

Results for **10** times diluted samples.

Table S4: Raw data for antibiotics and removal efficiencies between 16th November 2023 and 8th February 2024 for Pearson correlation *r* analyses and two-sample t-tests.

	Doxycycline (ug/L)	Ampicillin (ug/L)	Tilmicosin (ug/L)	Metronidazole (ug/L)	Ciprofloxacin (ug/L)	Ofloxacin (ug/L)	Sulfadiazine (ug/L)	Sulfamethoxazole (ug/L)	Trimethoprim (ug/L)	Denitrification %	Nitrification %	COD Removal %
16/11/2023	0.337971	0.025	0.005	0.015923	0.373077	12.8	0.005	2.7821616	0.746418	99.63%	96.13%	98.66%
23/11/2023	0.989622	0.025	0.005	0.005	0.792087	24.44	0.005	3.7172888	2.013364	100.00%	96.59%	94.89%
30/11/2023	0.467562	0.025	0.005	0.005	0.323798	10.71	0.005	33.78	7.55385	99.28%	62.77%	95.97%
7/12/2023	3.212085	0.025	0.005	0.088821	0.428733	10.59	0.005	59.01	18.62186	96.96%	32.53%	87.62%
14/12/2023	2.787038	0.025	7.99795	3.827446	1.747001	6.28	0.005	20.06	6.84366	99.44%	63.10%	94.36%
21/12/2023	12.59984	0.025	3.385212	0.005	0.418595	5.262037	0.005	20.764557	3.29415	62.17%	85.11%	96.67%
28/12/2023	3.64586	0.183	0.803995	0.664175	0.300211	4.935887	0.005	0.7242257	0.279916	97.89%	93.32%	95.35%
4/01/2024	1.891275	0.025	0.155504	0.036532	0.204872	2.386727	0.007296	2.6809516	0.729858	92.09%	88.86%	71.99%
11/01/2024	1.212188	0.025	0.52719	0.072019	0.213865	5.967581	0.014695	1.3109851	0.889058	99.32%	79.56%	95.60%
18/01/2024	1.040776	0.025	0.099148	0.247276	0.198331	9.423206	0.004872	0.3626898	0.394299	98.50%	96.18%	97.23%
25/01/2024	0.025	0.025	0.005	0.005	0.05	0.025	0.005	0.0025	0.0025	88.38%	91.22%	90.67%
1/02/2024	2.175689	0.247054	0.005	0.150351	0.351878	5.424428	0.584138	5.9427682	2.536537	86.24%	83.87%	95.19%
8/02/2024	1.173476	0.376172	0.005	0.111279	0.271631	8.337782	0.777856	0.6904332	0.359945	92.23%	83.56%	95.56%

Table S5: Pearson correlation *r* calculations for antibiotics and removal efficiencies.

Pearson Correlation r	*Doxycycline (ug/L)*	*Ampicillin (ug/L)*	*Tilmicosin (ug/L)*	*Metronidazole (ug/L)*	*Ciprofloxacin (ug/L)*	*Ofloxacin (ug/L)*	*Sulfadiazine (ug/L)*	*Sulfamethoxazole (ug/L)*	*Trimethoprim (ug/L)*	*Denitrification %*	*Nitrification %*	*COD Removal %*
Doxycycline (ug/L)	1											
Ampicillin (ug/L)	-0.06353	1										

Tilmicosin (ug/L)	0.416911	-0.19126	1									
Metronidazole (ug/L)	0.039279	-0.07856	**0.901927**	1								
Ciprofloxacin (ug/L)	0.116181	-0.16142	**0.849723**	**0.886414**	1							
Ofloxacin (ug/L)	-0.19551	-0.13664	-0.17961	-0.12825	0.27101	1						
Sulfadiazine (ug/L)	-0.11398	**0.910054**	-0.19338	-0.1175	-0.13271	-0.08472	1					
Sulfamethoxazole (ug/L)	0.264425	-0.28611	0.166033	0.103297	0.235167	0.127698	-0.22245	1				
Trimethoprim (ug/L)	0.141153	-0.24908	0.157568	0.167406	0.29487	0.168636	-0.18337	**0.9723641**	1			
Denitrification %	**-0.83404**	-0.08078	-0.16799	0.21277	0.182043	0.380115	-0.14792	-0.039166	0.097338	1		
Nitrification %	-0.08097	0.143511	-0.23095	-0.2587	-0.29672	0.021047	0.066769	**-0.921657**	**-0.94606**	-0.12194	1	
COD Removal %	0.074489	0.182131	0.120065	0.082436	0.141552	0.329718	0.144205	-0.091516	-0.12755	0.009255	0.147492	1

Table S6: Two-sample t-tests (assuming unequal variances) for selected antibiotics and removal efficiencies.

	DOXY	*DENIT*		*TRIM*	*NITR*		*SULF*	*NITR*
Mean	2.425645	0.932402	Mean	3.40484	0.809844	Mean	11.67893	0.809844
Variance	10.59975	0.010849	Variance	26.96205	0.033809	Variance	312.549	0.033809
Observations	13	13	Observations	13	13	Observations	13	13
Pearson Correlation	**-0.83404**		**Pearson Correlation**	**-0.94606**		**Pearson Correlation**	**-0.92166**	
Hypothesized Mean Difference	0		Hypothesized Mean Difference	0		Hypothesized Mean Difference	0	
df	12		df	12		df	12	
t Stat	1.610474		t Stat	1.743388		t Stat	2.195629	
P(T<=t) one-tail	0.066635		P(T<=t) one-tail	0.053402		P(T<=t) one-tail	0.024258	
t Critical one-tail	1.782288		t Critical one-tail	1.782288		t Critical one-tail	1.782288	
P(T<=t) two-tail	**0.133269**		**P(T<=t) two-tail**	**0.106805**		**P(T<=t) two-tail**	**0.048516**	
t Critical two-tail	2.178813		t Critical two-tail	2.178813		t Critical two-tail	2.178813	

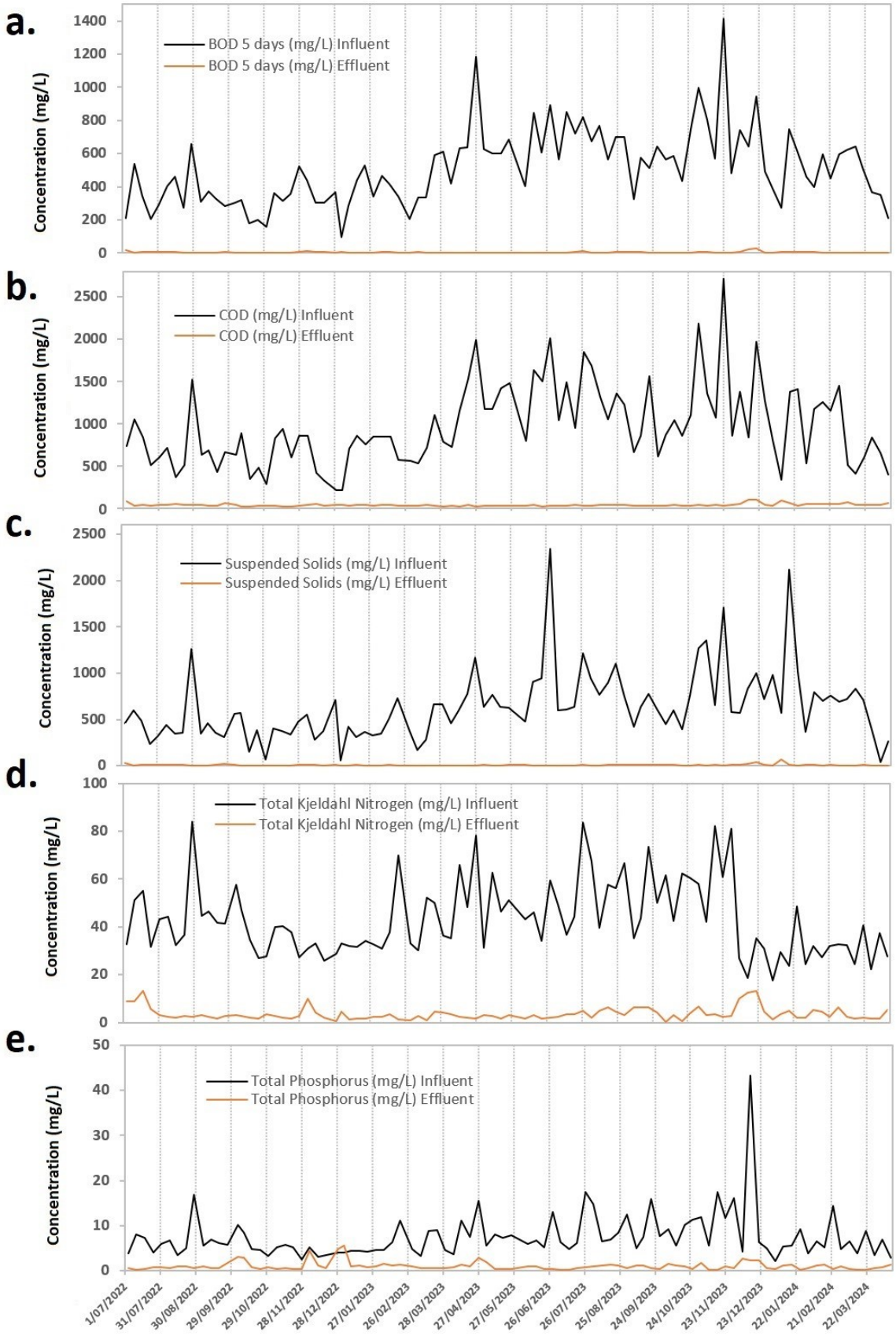

Figure S2 Stacked scatter plots depicting temporal influent and effluent concentrations between 1st July 2022 and 22nd April 2024 for biochemical oxygen demand (BOD) (a), chemical oxygen demand (COD), (b). suspended solids (c), total Kjeldahl nitrogen (TKN) (d) and total phosphorous (TP) (e).

WWTP Influent and Effluent Characteristics and Removal Efficiencies

Approaching the predicted toxic shock period, there is a notable spike in influent concentrations for BOD and COD on 23rd November 2023, at 1400 mg/L and 2700 mg/L, respectively. BOD and COD are measures of the amount of oxygen required to biologically or chemically oxidise any organic material present in solution, therefore elevated concentrations can indicate the presence of possibly harmful chemicals such as hydrocarbons that lead to toxic shocks. However, there was no immediate effect on WWTP biological treatment processes observed. Following this, effluent BOD and COD increased to 23 mg/L and 105 mg/L on 14th December 2023, corresponding to the removal efficiencies of 96% for BOD and 88% for COD during this period. Although the removal efficiencies remained high, the BOD concentration in the effluent exceeded the compliance limit of 20 mg/L. COD removal efficiency experienced a decline to 72% on 11th January 2024. Concurrently, the effluent concentration for SS increased to 38 mg/L on 21st December 2023 (96% removal) and 66 mg/L on 11th January 2024 (88% removal), exceeding the compliance limit of 30 mg/L.

There was a notable increase in influent TP concentration on 14th December 2023 to 43 mg/L but no change in TKN. As with BOD, COD and SS, there were increases in effluent concentrations for both TP and TKN on 7th and 14th December 2023, to 2.7 mg/L (35% removal) and 13 mg/L (33% removal) respectively. Although the outliers were noted as a reflection of worsening treatment performance, no effluent compliance limits were breached. The decline in effluent quality was indicative of a toxic shock event occurring, first noticed in samples taken on 7th December 2023.

WWTP Influent Chromium

Chromium was detected at higher concentration levels (averaging 0.13+0.07 mg/L) in the WWTP influent between 1st July 2022 and 22nd April 2024, compared to other local WWTPs (average <0.03 mg/L), as seen in Figure 2 (d). The high chromium concentrations at this WWTP are attributed to one of nine major industrial trade waste sites, a cement product manufacturer, which can contribute up to 20% of daily total influent flows. Chromium is considered in this study as a potential cause of the toxic shock due to the high concentrations observed, and literature indicates chromium can have individual or synergistic effects on biological treatment processes, especially in hexavalent form.(53-54) However, the chromium concentrations during the period of toxic shock event were 0.05–0.27 mg/L, which was consistent with normal concentrations in the wastewater influent to this WWTP. There are no observable events across the period in this study that could indicate a correlation between chromium and the decreased nitrogen and COD removal efficiencies.

WWTP Rainfall and Temperature Impacts

Daily maximum air temperature and total rainfall data were also monitored (see Figure 2 (e)) to exclude possible seasonal effects on the biological treatment processes. The mean maximum daily air temperature for the WWTP was 27+4 ^{o}C from the period 1st July 2022 to 22nd April 2024, with this rising to 31+3 ^{o}C during the toxic shock period. This rise is common during the summer season in Southeast Queensland. The average daily rainfall measurement for the same period was 3+9 mm, rising to 8+17 mm during the toxic shock period, which is also common during summer. The decreases in nitrogen and COD removal efficiencies are not observed to correspond to any ongoing air temperatures or rainfall events. Conversely, some of the rainfall events during the toxic shock period may have diluted the chemical spill or toxic shock event, with a noticeable but brief recovery of contaminant removal efficiencies. This is difficult to correlate since the removal efficiencies are calculated weekly, compared to the daily rainfall measurements.

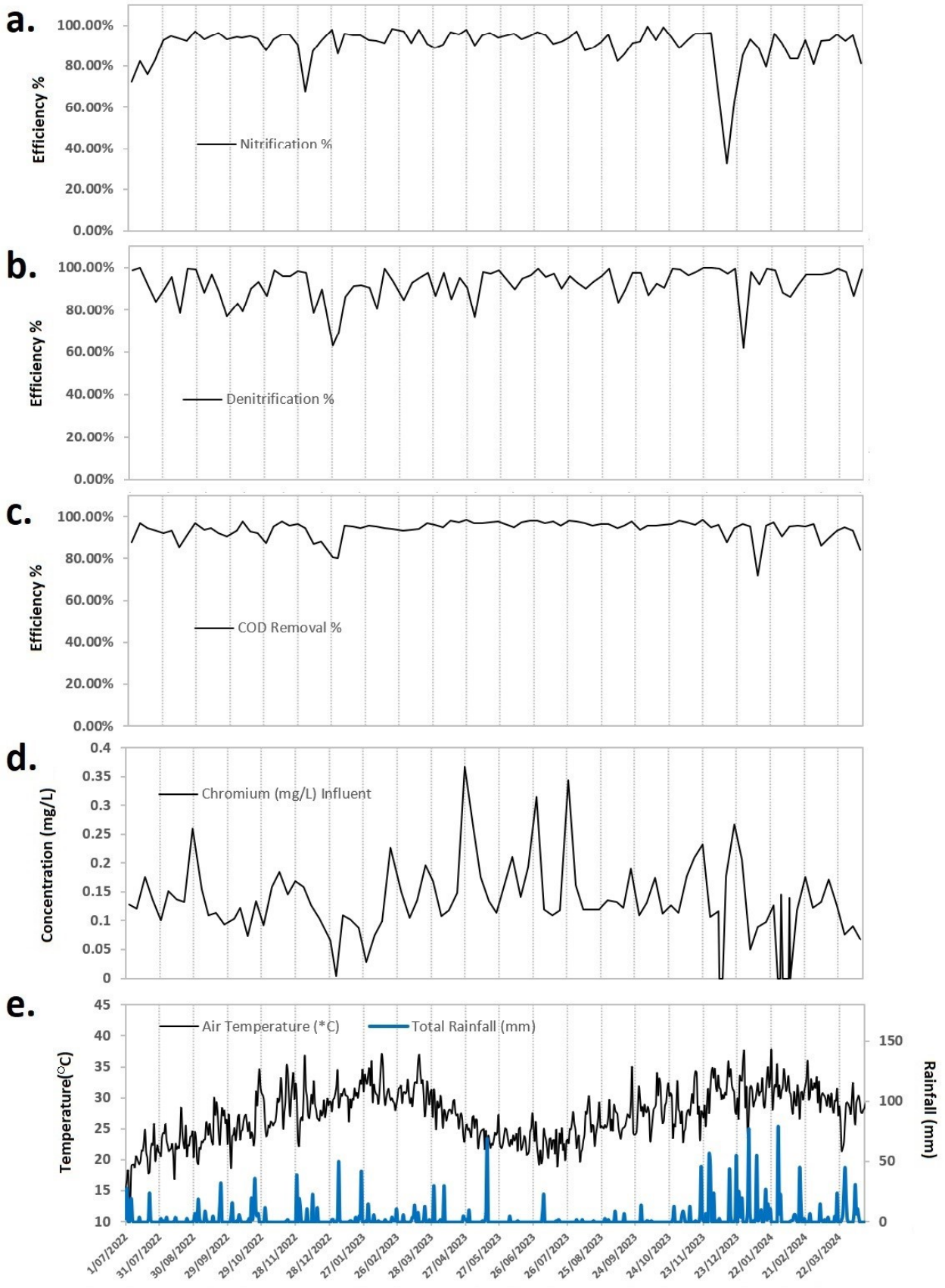

Figure S3 Stacked scatter plots depicting temporal nitrification efficiency (a), denitrification efficiency (b), COD removal efficiency (c), chromium concentrations (d) and daily rainfall totals and maximum air temperatures (e) between 1st July 2022 and 22nd April 2024.

Table S7 Trade waste PFAS, PPCPs (excluding antibiotics), drugs and pesticides data from the WWTP catchment (shaded grey) compared to other trade waste sites in nearby catchments. The first column, the pharmaceutical manufacturer, was identified as a CECs hotspot with elevated pharmaceuticals.

	TW01 61	TW00 61	TW00 63	TW00 69	TW01 43	TW01 45	TW02 23	TW02 57	TW02 59	TW0 261	TW 000	TW0 003	TW00 05	TW0 007	TW00 09	TW00 13	TW00 15	TW00 17	TW00 19	TW0 021	TW00 23	TW 002	TW 002	TW0 029	TW00 31	TW 003	TW00 35	TW00 37	TW00 39	TW0 041	TW00 43	TW00 45	TW00 47	TW00 49	TW00 51	TW00 53
ppb (ug/L)	**18 - Basic Chemical and Chemical Produc**	**15 - Pulp, Paper, and Converted Paper**	**15 - Pulp, Paper, and Converted Paper**	**20 - Non-Metallic Mineral Produc**	**23 - Transport Equipment Manufacturin**	**24 - Machinery and Equipment Manuf**	**11 - Food Product Manufacturing**	**12 - Beverage and Tobacco Product**	**11 - Food Product Manufacturing**	**29 - Waste Collection, Trea**	**95 - Personal and Other**	**29 - Waste Collection, Trea**	**11 - Food Product Manufacturing**	**29 - Waste Collection, Trea**	**11 - Food Product Manufacturing**	**19 - Polymer Product and Rubber**	**11 - Food Product Manufacturing**	**13 - Textile, Leather, Clothing and**	**11 - Food Product Manufacturing**	**29 - Waste Collection, Trea**	**11 - Food Product Manufacturing**	**95 - Personal and Other**	**95 - Personal and Other**	**29 - Waste Collection, Trea**	**18 - Basic Chemical and Chemical Produc**	**95 - Personal and Other**	**22 - Fabricated Metal Product Manuf**	**19 - Polymer Product and Rubber**	**20 - Non-Metallic Mineral Produc**	**29 - Waste Collection, Trea**	**18 - Basic Chemical and Chemical Produc**	**11 - Food Product Manufacturing**	**25 - Furniture and Other Manufacturin**	**12 - Beverage and Tobacco Product**	**11 - Food Product Manufacturing**	**11 - Food Product Manufacturing**
Acesulfame	21.75528	n.d.	0.06708	n.d.	8.25192	n.d.	n.d.	n.d.	0.15312	29.52072	0.4433	2.82178	n.d.	2.84986	0.13754	n.d.	n.d.	n.d.	n.d.	n.d.	0.37296	2.39616	0.6006	n.d.	n.d.	0.5837	n.d.	n.d.	n.d.	0.70044	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Amphetamine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.16874	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Atenolol	6.68928	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.13338	n.d.	n.d.	n.d.	0.0221	n.d.	n.d.	n.d.	n.d.	n.d.	0.09888	0.10374	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.21606	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Benzoylecgonine	n.d.	n.d.	n.d.	n.d.	0.1332	n.d.	n.d.	n.d.	0.00336	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.23738	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Caffeine	55.96848	n.d.	n.d.	n.d.	9.65496	n.d.	0.2256	2.0616	2.51304	32.61312	3.58202	26.8086	0.12402	6.06268	3.1304	n.d.	0.15106	n.d.	0.60164	n.d.	0.21576	62.7970	7.1812	n.d.	0.80522	45.6591	n.d.	0.06942	n.d.	0.46488	6.07568	0.0936	n.d.	10.392	0.48672	0.14534
Carbamazepine	20.93592	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.01056	0.00168	0.17056	n.d.	n.d.	0.09776	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.34216	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.42458	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cocaine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Codeine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Cotinine	0.2076	n.d.	n.d.	n.d.	0.46416	n.d.	n.d.	n.d.	0.08112	n.d.	n.d.	0.06422	n.d.	0.49088	0.00364	n.d.	n.d.	n.d.	n.d.	n.d.	0.006	0.36764	0.07384	n.d.	n.d.	0.04264	n.d.	n.d.	n.d.	0.03796	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ephedrine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.0924	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ketamine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.18226	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.20722	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.03458	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lidocaine	n.d.	n.d.	0.06214	n.d.	0.10152	n.d.	n.d.	n.d.	0.01272	n.d.	3.4424	n.d.	n.d.	0.77454	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	13.8616	0.22204	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
MDA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.45552	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.3172	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
MDMA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	3.3612	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Mephedrone	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Methamphetamin	n.d.	0.043056	0.01807	n.d.	0.45432	n.d.	n.d.	n.d.	0.0096	1.42944	0.01708	n.d.	0.022724	0.085722	0.01144	n.d.	0.01456	n.d.	n.d.	0.0325	0.22608	0.48328	0.14024	n.d.	n.d.	0.02748	n.d.	0.01118	n.d.	0.18434	0.013208	n.d.	n.d.	n.d.	0.094562	n.d.
Nicotine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.5888	n.d.	n.d.	n.d.	6.04188	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6.95942	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Oxycodone	19.65408	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Paracetamol	38.05272	n.d.	n.d.	n.d.	1.86792	n.d.	n.d.	n.d.	0.00288	0.0024	n.d.	0.25246	n.d.	0.10894	0.0299	n.d.	n.d.	n.d.	n.d.	n.d.	0.27888	8.69674	n.d.	n.d.	n.d.	n.d.	n.d.	0.0156	n.d.	n.d.	0.03978	n.d.	n.d.	n.d.	n.d.	n.d.
Pseudoephedrine	0.0732	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.03648	0.07152	n.d.	n.d.	n.d.	0.13312	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Salicylic acid	2.6088	0.693116	0.56498	n.d.	3.00336	n.d.	1.21872	20.05688	0.72072	2.03856	5.9982	32.87934	1.33484	1.19938	3.869066	0.461766	1.77034	n.d.	1.39984	0.60216	1.739552	21.25089	5.74262	n.d.	1.00516	1.0387	n.d.	0.50804	n.d.	0.4576	3.014444	1.99992	n.d.	135.3576	2.994688	1.059716
Tapentadol	7.6428	n.d.	n.d.	n.d.	0.05136	n.d.	n.d.	n.d.	0.03456	n.d.	n.d.	n.d.	n.d.	0.03926	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.33436	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Temazepam	99.72816	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.089288	0.04344	n.d.	n.d.	n.d.	0.05772	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.20506	n.d.	n.d.	n.d.	n.d.	0.41704	0.040004	n.d.	n.d.	3.2592	n.d.	n.d.
Tramadol	0.182888	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.020166	0.0108	0.09152	n.d.	n.d.	0.07332	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.25194	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.2626	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Venlafaxine	0.054724	n.d.	n.d.	n.d.	0.06864	n.d.	n.d.	n.d.	0.21744	n.d.	0.21866	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.27196	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Atorvastatin	0.034088	n.d.	n.d.	n.d.	0.01872	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.07462	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Citalopram	18.68644	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.065766	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.02938	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Diclofenac	0.082088	n.d.	0.037178	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.04632	2.92552	n.d.	n.d.	0.13936	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.68074	0.57694	n.d.	n.d.	0.09178	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.028344	n.d.

Hydrochlorthiazid	1.06752	n.d.	n.d.	n.d.	0.13704	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.0403	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Naproxen	310.0037	n.d.	n.d.	n.d.	0.37512	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Paraxanthine	151.457	n.d.	n.d.	n.d.	14.53632	n.d.	n.d.	n.d.	2.1672	2.50248	1.6055	n.d.	n.d.	n.d.	0.51688	n.d.	n.d.	n.d.	n.d.	n.d.	1.00152	10.4358	1.18274	n.d.	n.d.	1.68636	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
24 D	0.43104	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.72246	485.5755	0.70642	0.40664	0.7787	n.d.	0.16848	n.d.	n.d.	2.87846	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.24258	n.d.	0.34398	n.d.	n.d.	1.65906	n.d.
Ametryn	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09568	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Atrazine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.6107	n.d.	n.d.	0.04186	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Carbaryl	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Carbendazim	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
DEET	0.26688	33.79168	0.48438	n.d.	0.29064	n.d.	0.48144	1.6848	0.56016	0.3	1.29116	5.32974	1.5184	11.0656	0.83512	0.25792	0.81978	n.d.	0.3926	0.22074	0.47294	98.8418	18.4649	n.d.	0.37414	10.3643	n.d.	0.3887	n.d.	0.33566	0.32006	0.34554	n.d.	19.6392	0.38012	0.24934
Diazinon	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Diuron	n.d.	n.d.	n.d.	n.d.	0.11328	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.69282	n.d.	0.36062	0.31564	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.08918	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fipronil	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.2298	0.01092	0.36634	n.d.	n.d.	0.19266	n.d.	n.d.	0.01118	0.08008	n.d.	n.d.	n.d.	n.d.	0.07306	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.20384
Fluazifop	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.27092	35.48948	0.06916	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Haloxyfop	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	100.1218	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Hexazinone	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.21086	n.d.	n.d.	0.04394	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Imidaclorprid	n.d.	n.d.	n.d.	n.d.	0.02856	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	115.8011	0.0572	0.09802	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09256	0.05252	n.d.	n.d.	0.11622	n.d.	n.d.	n.d.	n.d.	0.07696	n.d.	n.d.	n.d.	0.07904	n.d.
MCPA	0.28128	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	66.3572	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Methomyl	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Metolachlor	0.07032	0.01638	0.03484	n.d.	n.d.	n.d.	n.d.	2.28	1.63032	n.d.	0.20358	7.9105	0.19136	0.21944	n.d.	n.d.	0.19058	n.d.	0.17654	13.5967	0.16224	0.04576	0.08736	n.d.	n.d.	0.11856	n.d.	0.03354	n.d.	n.d.	0.05746	0.05356	n.d.	n.d.	0.038774	0.0299
Metribuzin	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Metsulfuron	n.d.	n.d.	n.d.	n.d.	0.0552	n.d.	n.d.	n.d.	0.05112	0.06096	n.d.	3.20164	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Prometryn	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.03302	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.01004	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Propazine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Propiconazole	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.14904	n.d.	n.d.	n.d.	n.d.	2.30958	n.d.	0.12896	0.3809	n.d.	0.08866	n.d.	n.d.	n.d.	n.d.	0.05564	0.13234	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Propoxur	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Simazine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.43676	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.08008	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Tebuconazole	n.d.	n.d.	n.d.	n.d.	0.10368	n.d.	n.d.	n.d.	n.d.	n.d.	0.04264	2.59454	n.d.	0.14742	0.03354	0.0091	0.07878	n.d.	n.d.	0.16822	0.03328	0.03198	n.d.	n.d.	n.d.	0.12272	n.d.	n.d.	n.d.	1.15232	n.d.	n.d.	n.d.	n.d.	n.d.
Terbuthylazine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.07696	n.d.	n.d.	0.036114	n.d.	n.d.	n.d.	n.d.	0.03952	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
6:2 FTS	n.d.	n.d.	n.d.	n.d.	0.1476	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.85 74	n.d.	0.29 9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.6188	n.d.	n.d.	n.d.	n.d.	n.d.
8:2 FTS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.1378	n.d.	n.d.	n.d.	n.d.	n.d.
N-EtFOSE	0.3624	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.1768	0.3848	n.d.	n.d.	0.0598	0.104	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
N-MeFOSE	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFBA	0.0312	0.0338	0.0702	n.d.	0.05976	n.d.	0.02784	0.06	n.d.	0.01824	0.10014	0.4966	n.d.	0.1794	0.039	0.0494	0.0182	n.d.	0.3666	0.0754	0.0754	0.0728	0.1248	n.d.	0.1456	0.0884	n.d.	0.0754	n.d.	0.0494	0.0754	0.0234	n.d.	0.6984	0.9386	0.026
PFBS	0.02928	0.0208	0.0104	n.d.	0.0247 2	n.d.	0.02856	n.d.	0.03	0.02928	0.0286	0.9776	0.0286	0.143	0.0338	0.0442	0.0312	n.d.	0.0338	0.0338	0.0442	0.0364	0.0338	n.d.	0.0312	0.0338	n.d.	0.0338	n.d.	0.0364	0.0338	0.0338	n.d.	n.d.	0.0312	0.0312

PFDA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.1368	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.01 04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.013	n.d.
PFDoDA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFDS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFECHS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09 62	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.01 82	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFHpA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.27 04	n.d.	0.03 9	n.d.	n.d.	n.d.	n.d.	0.039	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFHpS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFHxA	n.d.	n.d.	n.d.	n.d.	0.0381 6	n.d.	n.d.	0.0312	n.d.	n.d.	n.d.	1.17 52	n.d.	0.07 8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.391	n.d.	n.d.	n.d.	n.d.	n.d.	0.0494	n.d.	n.d.	0.2016	n.d.	n.d.
PFHxS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.18 56	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFHxS_total	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.30 52	n.d.	0.04 94	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFNA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFOA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.00 696	n.d.	n.d.	0.05 2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFOS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.08 84	n.d.	0.04 68	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFOS_total	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09 36	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.0182	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFPeA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.00 288	n.d.	0.46 28	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PFPeS	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.43 68	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.