Electronic Supplementary Material (ESI) for Analytical Methods.

Table S1 Validation parameters (n = 6) obtained for 58 analytes at three concentration levels in blank river samples

Analytes	Internal standard	Linear equation (RSD, %)	Average recovery [c], % (RSD, %)			LOQ (ng/L)	LOD (ng/L)	R^2
			Low level [d]	Medium level	High level			
Sulfaguanidine	Sulfathiazole-D4	Y=0.42X –0.087 (1.6[a], 3.2[b])	80.6 (2.6[e], 3.6[f])	92.0 (3.5, 6.2)	86.2 (4.0, 6.2)	0.24	0.073	0.9991
Sulfacetamide	Sulfathiazole-D4	Y=0.27X – 0.037 (2.1, 2.6)	82.5 (2.5, 4.8)	90.2 (2.0, 5.8)	82.0 (3.1, 7.2)	0.28	0.085	0.9989
Sulfisomidine	Sulfathiazole-D4	Y=0.35X + 0.031 (2.2, 1.6)	80.8 (5.2, 6.4)	101 (2.8, 5.0)	91.2 (6.4, 8.0)	0.26	0.080	0.9989
Sulfadiazine	Sulfathiazole-D4	Y=0.64X – 0.015 (1.2, 1.7)	82.0 (3.3, 6.6)	100 (3.0, 7.2)	92.1 (3.5, 7.3)	0.17	0.052	0.9987
Sulfathiazole	Sulfathiazole-D4	Y=0.39X – 0.012 (0.9, 1.1)	90.3 (2.9, 6.0)	92.9 (2.2, 5.6)	89.8 (2.7, 6.9)	0.25	0.076	0.9986
Sulfapyridine	Sulfamerazine-D4	Y=0.47X + 0.012 (1.1, 1.3)	82.0 (2.7, 7.2)	90.8 (3.0, 7.3)	90.6 (3.2, 7.6)	0.22	0.067	0.9989
Sulfamerazine	Sulfamerazine-D4	Y=0.43X – 0.051 (1.8, 1.7)	88.2 (3.0, 6.7)	96.2 (4.7, 5.6)	99.3 (5.2, 5.2)	0.24	0.074	0.9992
Sulfamethazine	Sulfamethazine-D4	Y=0.87X + 0.020 (1.4, 1.2)	83.0 (2.1, 6.8)	92.3 (2.2, 6.2)	89.2 (3.2, 6.8)	0.10	0.030	0.9986
Sulfamethizole	Sulfamethazine-D4	Y=0.42X + 0.012 (1.6, 1.3)	91.2 (3.4, 8.8)	92.0 (5.0, 6.8)	101 (2.6, 8.0)	0.24	0.073	0.9991
sulfisozole sodium	Sulfamethazine-D4	Y=0.17X – 0.012 (1.2, 0.9)	83.2 (2.5, 7.9)	88.2 (2.9, 5.7)	82.9 (3.9, 6.0)	0.30	0.091	0.9982
Sulfamethoxypyridazine	Sulfamethazine-D4	Y=0.77X – 0.022 (1.8, 1.7)	86.0 (2.7, 6.2)	89.3 (5.0, 7.3)	102 (3.2, 7.6)	0.14	0.042	0.9992
Sulfamethoxydiazine	Sulfamethazine-D4	Y=0.64X – 0.017 (2.1, 2.0)	82.2 (3.0, 7.7)	93.9 (4.2, 3.6)	87.3 (2.2, 6.2)	0.17	0.052	0.0089
Sulfamonomethoxine	Sulfamethazine-D4	Y=0.44X – 0.023 (2.1, 1.8)	83.0 (2.1, 7.8)	92.3 (3.1, 6.2)	90.2 (4.0, 6.8)	0.23	0.070	0.9983
Sulfachloropyridazine	Sulfamethazine-D4	Y=0.34X + 0.032 (1.1, 1.3)	91.2 (3.4, 6.1)	94.2 (3.0, 6.8)	82.6 (2.6, 7.0)	0.27	0.082	0.9989
Sulfaclozine	Sulfamethazine-D4	Y=0.12 X + 0.011 (1.3, 1.2)	82.1 (2.5, 5.3)	89.6 (2.7, 6.5)	90.3 (3.0, 5.3)	0.36	0.11	0.9986
Sulfadoxine	Sulfadoxine-D6	Y=1.14X – 0.014 (1.1, 0.8)	78.0 (1.7, 6.7)	80.6 (3.0, 5.6)	90.2 (2.7, 5.8)	0.052	0.017	0.9986
Sulfamethoxazole	Sulfadoxine-D6	Y=0.16X + 0.042 (1.5, 1.8)	84.0 (3.6, 6.7)	90.9 (6.8, 6.9)	80.3 (3.9, 6.2)	0.30	0.091	0.9987
Sulfisoxazole	Sulfadoxine-D6	Y=0.35X + 0.032 (0.9, 0.8)	82.6 (2.8, 6.9)	92.5 (3.9, 7.8)	89.8 (3.0, 6.8)	0.26	0.079	0.9991
Sulfabenzamide	Sulfadoxine-D6	Y=0.58X – 0.022 (1.1, 1.4)	83.2 (3.4, 5.7)	89.2 (4.8, 6.8)	79.9 (3.2, 5.5)	0.20	0.061	0.9986
Sulfadimethoxine	Sulfadoxine-D6	Y=0.10X – 0.011 (2.3, 2.1)	80.8 (3.5, 7.6)	90.9 (4.2, 4.8)	81.2 (3.0, 5.8)	0.39	0.12	0.9982
Sulfaquinoxaline	Sulfadoxine-D6	Y=0.53X + 0.033 (2.0, 1.8)	88.1 (2.8, 6.0)	96.2 (3.3, 5.9)	83.9 (2.2, 6.3)	0.22	0.067	0.9986
Sulfaphenazole	Sulfadoxine-D6	Y=0.43X + 0.022 (1.6, 1.8)	82.1 (3.8, 7.0)	92.2 (2.3, 6.9)	81.9 (2.6, 7.8)	0.24	0.074	0.9988

[a] RSD of the slope, n = 7 replicates; [a] RSD of the intercept, n = 7 replicates; [c] The mean value was determined in one day (n = 6 replicates); [d] Spiked level: low, medium and high level mean 2.0, 20.0 and 80.0 ng/L for sulfonamides, 4.0, 40.0 and 160 ng/L for quinolones, 8.0, 80.0 and 320 ng/L for steroid hormones, respectively. [e] Intra-day, n = 6 replicates; [f] Inter-day, n = 3 replicates × 6 days within a 2-week period.

Table S1 continued

Analytes	Internal standards	Linear equation (RSD, %)	Average recovery ,% (RSD, %)			LOQ (ng/L)	LOD (ng/L)	R^2
			Low level	Medium level	High level			
Trenbolone	17-Methyltestosterone-D3	Y=0.43X -0.032(1.2, 1.6)	99.2 (3.2, 5.6)	88.0 (3.5, 6.2)	106 (2.0, 6.2)	1.26	0.38	0.9983
Boldenone	17-Methyltestosterone-D3	Y=0.56X – 0.052(2.2, 2.3)	105 (2.5, 6.8)	90.2 (4.0, 5.8)	96.0 (4.1, 7.2)	0.99	0.34	0.9989
Nandrolone	17-Methyltestosterone-D3	Y=0.10X + 0.021(1.2, 1.4)	96.8 (5.3, 5.4)	92.7 (2.8, 5.0)	103 (5.4, 8.0)	1.98	0.60	0.9989
Metandienone	17-Methyltestosterone-D3	Y=1.1 X – 0.032(1.6, 2.0)	103 (2.3, 6.6)	106 (5.0, 6.3)	99.1 (3.5, 6.3)	0.75	0.23	0.9987
Testosterone	17-Methyltestosterone-D3	Y=0.38X – 0.017(1.4, 1.6)	92.2 (3.9, 5.0)	92.9 (3.6, 5.6)	109 (5.3, 6.9)	1.39	0.42	0.9986
Norethindrone	17-Methyltestosterone-D3	Y=0.19X + 0.023(1.7, 1.1)	110 (3.6, 6.2)	102 (5.0, 7.3)	92.9 (4.9, 6.6)	1.72	0.52	0.9989
17-a-hydroxyprogesterone	17-Methyltestosterone-D3	Y=0.28X – 0.031 (1.2, 1.3)	98.6 (2.0, 6.7)	90.2 (4.2, 3.6)	89.5 (2.2, 6.2)	1.49	0.45	0.9984
Stanozolol	17-Methyltestosterone-D3	Y=0.43X + 0.051 (1.7, 1.6)	107 (2.1, 5.8)	92.3 (2.9, 6.2)	102 (4.2, 5.8)	1.25	0.38	0.9986
17-Methyltestosterone	17-Methyltestosterone-D3	Y=0.25X + 0.021 (2.2, 1.9)	102 (3.4, 6.8)	99.7 (3.0, 7.8)	102 (2.6, 5.0)	1.56	0.47	0.9983
Ethisterone	17-Methyltestosterone-D3	Y=0.91X +0.092 (1.8, 2.0)	96.2 (3.2, 6.4)	82.8 (3.9, 5.7)	98.9 (3.9, 5.0)	0.83	0.25	0.9983
21-a-hydroxyprogesterone	Levonorgestrel-D6	Y=0.44X – 0.041 (2.2, 1.7)	99.0 (2.1, 5.2)	89.1 (2.0, 6.3)	102 (3.2, 5.6)	1.26	0.38	0.9985
Norgestrel	Levonorgestrel-D6	Y=0.50X – 0.036 (1.7, 1.6)	96.2 (3.0, 6.7)	83.2 (4.2, 3.6)	97.8 (2.2, 5.2)	1.20	0.36	0.9983
Levonorgestrel	Levonorgestrel-D6	Y=0.61X – 0.042 (1.2, 1.8)	98.0 (5.8, 7.8)	92.3 (3.1, 6.2)	99.2 (3.0, 6.8)	1.02	0.31	0.9983
Medroxyprogesterone	Levonorgestrel-D6	Y=0.41X – 0.052 (2.1, 1.9)	103 (5.4, 6.1)	91.2 (6.0, 5.8)	101 (3.6, 8.0)	1.32	0.40	0.9989
17-a-hydroxyprogesterone acetate	Levonorgestrel-D6	Y=0.34X +0.023 (2.2, 2.0)	101 (3.5, 6.3)	82.9 (3.8, 5.5)	106 (3.0, 5.3)	1.45	0.44	0.9986
Dydrogesterone	Levonorgestrel-D6	Y=0.67X + 0.022 (1.8, 1.6)	98.8 (3.7, 5.7)	91.6 (2.0, 5.6)	98.9 (2.7, 5.8)	1.01	0.31	0.9986
Megestrol acetate	Levonorgestrel-D6	Y=0.49X + 0.032 (1.3, 1.3)	97.0 (3.6, 6.5)	82.9 (3.8, 6.9)	96.3 (3.9, 6.1)	1.19	0.36	0.9987
Chlormadinone acetate	Levonorgestrel-D6	Y=0.21X + 0.026 (1.2, 1.6)	91.6 (2.8, 6.9)	90.2 (4.9, 5.8)	96.8 (3.0, 7.8)	1.65	0.50	0.9984
Medroxyprogesterone acetate	Levonorgestrel-D6	Y=0.19X + 0.012 (2.2, 1.9)	101 (4.4, 6.7)	89.2 (2.8, 6.8)	99.9 (3.2, 5.5)	1.69	0.51	0.9986
Progesterone	Levonorgestrel-D6	Y=0.10X – 0.032 (2.3, 2.6)	96.8 (2.5, 7.6)	81.9 (3.2, 4.8)	103 (4.0, 4.8)	2.09	0.63	0.9985
Melengestrol acetate	Levonorgestrel-D6	Y=0.79X +0.024 (1.5, 1.3)	92.1 (2.8, 7.0)	86.6 (3.3, 6.9)	99.0 (2.2, 6.3)	0.90	0.27	0.9986
Nandrolone propionate	Levonorgestrel-D6	Y=0.23X + 0.017 (1.4, 1.6)	102 (5.0, 8.3)	98.0 (4.1, 7.8)	102 (3.6, 7.0)	1.58	0.48	0.9984
Testosterone propionate	Levonorgestrel-D6	Y=0.16X – 0.026 (1.2, 1.7)	109 (5.0, 8.3)	89.0 (3.1, 7.8)	101 (5.0, 8.3)	1.89	0.57	0.9985
Hydroxyprogesterone caproate	Levonorgestrel-D6	Y=0.19X + 0.023 (2.0, 1.9)	101 (6.4, 9.0)	86.0 (3.3, 6.6)	102 (2.4, 8.8)	1.68	0.51	0.9988

Table S1 continued

Analytes	Internal standards	Linear equation (RSD, %)	Average recovery ,% (RSD, %)			LOQ (ng/L)	LOD (ng/L)	R^2
			Low level	Medium level	High level			
Enoxacin	Enoxacin-D8	Y=0.27X -0.021 (0.9, 1.3)	93.1 (2.9, 6.3)	102 (6.4, 8.0)	100 (2.8, 6.6)	0.34	0.10	0.9991
Norfloxacin	Norfloxacin-D5	Y=0.17X – 0.014 (1.3, 1.1)	91.8 (5.2, 5.8)	103 (3.8, 5.2)	96.0 (5.2, 7.2)	0.40	0.12	0.9988
Ofloxacin	Ofloxacin-D3	Y=0.90X + 0.051 (1.9, 2.3)	90.2 (3.6, 3.6)	96.8 (3.0, 6.8)	109 (5.1, 7.3)	0.14	0.043	0.9982
Pefloxacin	Pefloxacin-D5	Y=0.26X – 0.030 (1.5, 1.2)	89.9 (3.1, 5.2)	91.9 (3.2, 4.6)	99.6 (5.2, 6.8)	0.36	0.11	0.9987
Ciprofloxacin	Ciprofloxacin-D8	Y=0.42X – 0.039 (1.4, 1.3)	82.0 (3.6, 7.9)	98.3 (5.5, 6.1)	91.2 (3.0, 5.8)	0.23	0.07	0.9986
Lomefloxacin	Ciprofloxacin-D8	Y=0.20X + 0.023 (2.0, 2.3)	90.5 (2.5, 4.8)	102 (3.4, 7.8)	90.2 (2.9, 6.0)	0.48	0.15	0.9982
Danofloxacin	Danofloxacin-D3	Y=1.1X – 0.031 (1.8, 1.7)	92.2 (3.9, 6.2)	98.7 (2.2, 6.2)	98.3 (3.2, 6.2)	0.12	0.036	0.9982
Enrofloxacin	Enrofloxacin-D5	Y=0.49X + 0.036 (1.1, 1.3)	92.2 (3.9, 6.9)	104 (4.2, 6.9)	98.0 (3.7, 5.2)	0.18	0.055	0.9986
Sarafloxacin	Sarafloxacin-D8	Y=0.28X + 0.021 (0.9, 1.3)	86.0 (5.2, 5.9)	91.2 (3.2, 3.6)	94.2 (2.0, 5.7)	0.32	0.095	0.9981
Sparfloxacin	Sarafloxacin-D8	Y=0.85X – 0.033 (1.6, 1.3)	90.2 (4.2, 7.6)	103 (3.0, 6.3)	96.2 (2.0, 5.7)	0.16	0.048	0.9982
Difloxacin	Difloxacin-D3	Y=0.25X – 0.14 (1.2, 1.1)	80.0 (3.0, 7.8)	82 (3.6, 7.0)	88.9 (4.9, 6.0)	0.36	0.11	0.9988
Flumequine	Difloxacin-D3	Y=0.42X – 0.029 (1.7, 2.1)	109 (5.0, 7.3)	89.8 (2.2, 4.7)	102 (4.2, 6.6)	0.26	0.08	0.9982