Supporting Information

Determination and Source Apportionment of Five Classes of Steroid Hormones in Urban Rivers

Hong Chang, Yi Wan, Jianying Hu*

College of Urban and Environmental Sciences, Peking University, Beijing, 100871 China

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Materials. Thirty hormones as shown in Figure S1 were targeted in this study. 19-nor-4-androstene-3,17-diol (NAD), trenbolone (TBL), nandrolone (NDL), androstenedione (ADD), norethindrone (NTD), 17α-hydroxyprogesterone (17α-HPT), testosterone (TTR), 21α-hydroxyprogesterone (21α-HPT), norgestrel (NGT), 17α,20β-dihydroxy-4-progegnen-3-one (DPO), methyl testosterone (MTTR), epiandrosterone (EADR), stanozolol (SZL), 6α-methylhydroxyprogesterone (MHPT), megestrol acetate (MTA), medroxyprogesterone acetate (MPA), progesterone (PGT), androsterone (ADR), 17α-estradiol (αE2), cortisol (CRL), cortisone (CRN), prednisone (PRE), prednisolone (PREL), dexamethasone (DEX), 6α-methylprednisolone (MPREL), aldosterone (ADT), $^{13}C_2$-NTD, $^{13}C_2$-TTR, NGT-d_6 and PGT-d_9 were purchased from Sigma (St Louis, MO, USA). Ethinylestradiol (EE2), 17β-estradiol (E2), estrone (E1), diethylstilbestrol (DES), E2-d_3, E1-d_2, and EE2-d_4 were purchased as powders from Wako (Tokyo, Japan). CRL-d_2 was obtained from C/D/N Isotopes (Montreal, Canada). Formic and acetic acids were analytical grade (Beijing Chemicals, China). Methanol, acetonitrile, ethyl acetate, hexane, and dichloromethane were all HPLC grade purchased from Fisher Chemical Co. (Beijing, China). HPLC-grade water was prepared using a Milli-Q RC apparatus (Millipore, Bedford, MA, USA).

Sample Preparation. In our previous studies, we have developed separate analytical methods to detect estrogens (*1*), glucocorticoids (*2*), androgens, and progestogens (*3*) in various water matrices. In this study, based on these methods previously described, we further developed the present method to allow the measurement of all five classes of steroid hormones in one water sample. In this method, 200 mL of effluents spiked with 10 ng of E1-d_4 and 2 ng of other surrogate standards and 2 L of river water spiked with 50 ng of E1-d_4 and 10 ng of other surrogate standards were respectively extracted through an Oasis HLB cartridge (6 mL, 60 mg or 500 mg, Waters, USA), which was previously conditioned with 6 mL ethyl acetate, 6 mL acetonitrile and 12 mL distilled water at a flow rate of 5-10 mL/min. The cartridge was washed with 10 mL of distilled water, and then was dried under a flow of nitrogen. 15 mL of ethyl acetate and 6 mL of ethyl acetate/acetonitrile (1:1, v/v) were used to elute the analytes. For the effluent samples, daily 24-hour composites were extracted, and then the 7-day elutants were pooled as composite samples for a complete week. The extracts were dried and redissolved in 0.2 mL of ethyl acetate and 1.8 mL of hexane. The mixed solutions were applied to silica cartridges (3 mL, 500 mg, Waters), which had been preconditioned with 4 mL water-saturated ethyl acetate and 4 mL hexane/ethyl acetate (90:10, v/v). After the cartridges were rinsed with 3 mL of hexane/ethyl acetate (90:10, v/v), the fraction (F1) containing nine androgens, nine progestogens, and five estrogens were eluted with 3 mL of hexane/ethyl acetate (38:62, v/v), and the fraction (F2) containing six glucocorticoids and one mineralocorticoid were subsequently eluted with 3 mL of water-saturated ethyl acetate. For androgens, progestogens and adrenal steroids, F1 and F2 eluates were dried and reconstituted respectively with 0.5 mL of methanol for LC-ESI-MS/MS analysis. For estrogens, 0.2 mL of the methanol reconstituted solution of F1 was dried and redissolved with 1 mL hexane–methylene chloride (DCM) (1:1, v/v), and then passed through the preconditioned Florisil cartridges (6 mL, 1 g, Waters). 10 mL of a mixture of hexane–DCM (1:1, v/v) were discarded and the fraction containing all estrogens was eluted with 6 mL of acetone–DCM (1:9, v/v). The solution was evaporated to dryness under a gentle stream of nitrogen and reconstituted with 0.2 mL of acetonitrile for LC-ESI-MS/MS analysis.

LC-ESI-MS/MS Analysis. The LC apparatus was an Acquity Ultra Performance LC (Waters). All analytes were separated using a Waters Acquity UPLC® BEH C18 column (100 × 2.1 mm, 1.7 µm particle size) (USA). The column was maintained at 40°C at a flow rate of 0.3 mL/min and the injection volume was 5 µL. Methanol and water containing 0.1% formic acid were used for analyzing androgens and progestogens. Gradient conditions were initiated with 60% methanol followed by a linear increase to 65% methanol in 2.5 min. After increased to 70% in 3.5 min, methanol was increased sharply to 100% in 0.1 min and was held for 1 min. For separation of adrenal steroids, methanol and water containing 0.1% acetic acid were used as mobile phases. 35% methanol was increased linearly to 55% in 5 min, to 80% methanol in the next 0.5 min, and to 100% in the following 2.5 min (held for 1 min). For separation of estrogens, acetonitrile and 0.1% acetic acid in water were chosen as mobile phases. The gradient was increased linearly from an initial 20 to 80% acetonitrile in 4.5 min, and then to 100% acetonitrile in 0.1 min (held for 1 min). In our previous paper (*16*), we reported that methanol, combined with a phenyl column for good chromatographic separation, could enhance the signal intensities of E1, βE2 and EE2 compared with acetonitrile as organic modifier in LC-MS system. However, in 2006, only C18 columns with this small particle size could be commercially obtained for this improved LC system. Thus, for better separating target estrogens on the C 18 column, acetonitrile was used as organic modifier in this study, and this condition can separate all estrogens even for αE2 and βE2.

Mass spectrometry was performed using a Premier XE tandem quadrupole mass spectrometer (Waters) equipped with a Z-Spray ionization (ESI) source. ESI-MS/MS detections were performed in the negative ion mode for glucocorticoid and mineralocorticoid steroids and in the positive ion mode for the other steroids. In the analysis of androgens and progestogens, $[M+H]^+$ was selected as the precursor ion. For glucocorticoids and mineralocorticoids, $[M+acetate]^-$, the adducts with CH_3COOH were selected as the precursor ions. Concerning estrogens, the precursor ions for βE2, αE2 and EE2 were $[M+H-H_2O]^+$, and those for E1 and DES were their protonation ions ($[M+H]^+$). The two most abundant multi-selected reaction monitoring (MRM) transitions, cone voltages and collision energies were optimized for each steroid by infusing standard solutions in the mass spectrometer. Of the two MRM transitions, the first transition was selected for quantitation, and another was used for confirmation. For the surrogate standards, their most intense product ions were monitored, and their compensating target compounds following the corresponding surrogate standards were shown in Table S1 (Supporting Information). Common MS parameters were as follows: capillary voltage, 2.5 kV; source temperature, 120; desolvation temperature, 450; source gas flow, 50 L/h; and desolvation gas flow, 900 L/h.

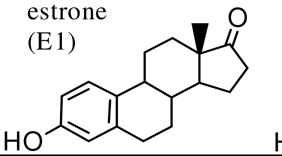
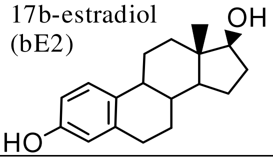
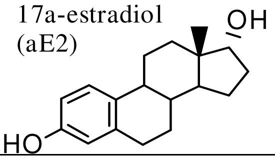
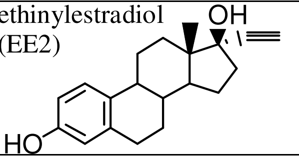
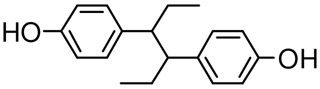
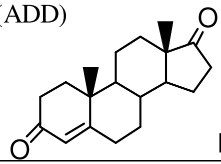
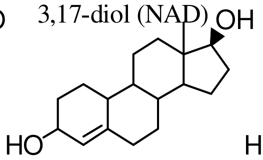
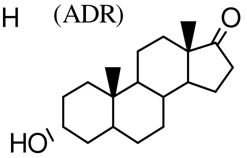
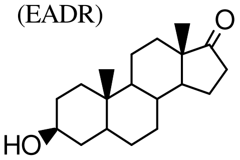
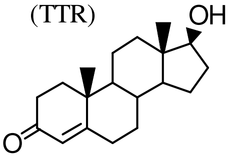
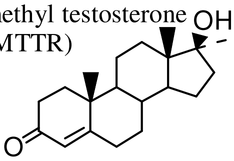
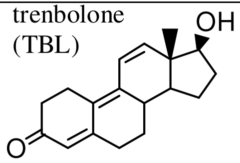
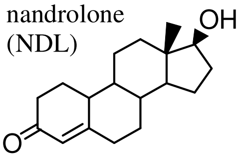
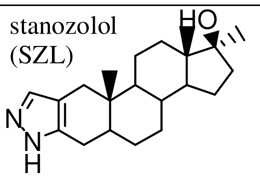
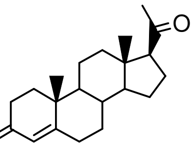
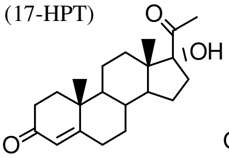
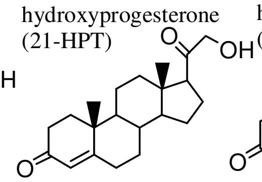
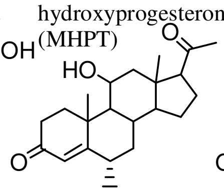
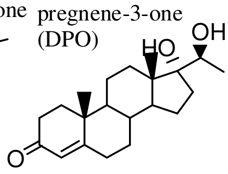
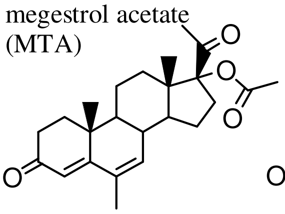
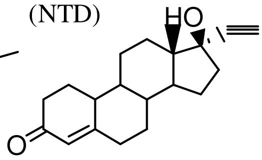
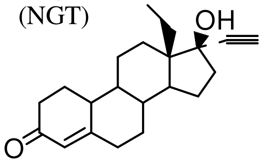
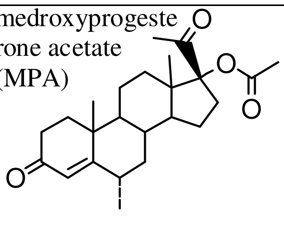
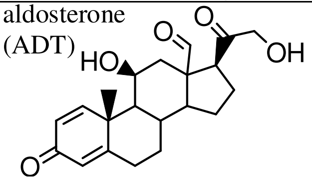
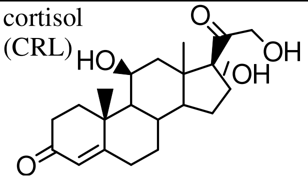
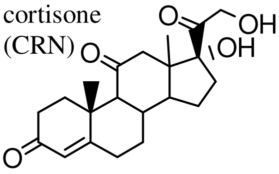
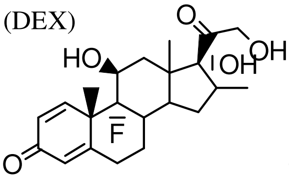
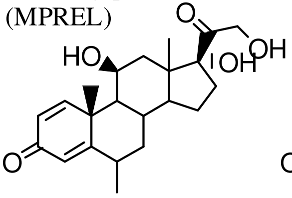
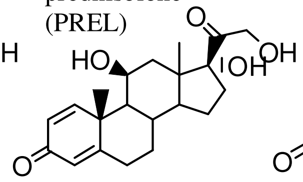
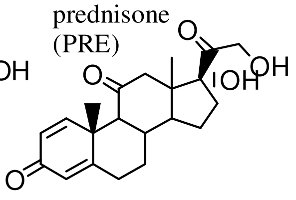
References

(1) Hu, J. Y.; Zhang, H. F.; Chang, H. Improved method for analyzing estrogens in water by liquid chromatography-electrospray mass spectrometry. *J. Chromatogr. A*. **2005**, *1070 (1-2)*, 221-224.

(2) Chang, H.; Hu, J. Y.; Shao, B. Occurrence of natural and synthetic glucocorticoids in sewage treatment plants and receiving river waters. *Environ. Sci. Technol.* **2007**, *41 (10)*, 3462-3468.

(3) Chang, H.; Wu, S. M.; Hu, J. Y.; Asami, M.; Kunikane, S. Trace analysis of androgens and progestogens in environmental waters by ultra-performance liquid chromatography-electrospray tandem mass. *J. Chromatogr. A* **2008**, *1195 (1-2)*, 44-51.

FIGURE S1. Structure of Target Five Classes of Steroid Hormones

Estrogens	
N	estrone (E1)  17b-estradiol (bE2)  17a-estradiol (aE2) 
S	ethinylestradiol (EE2)  diethylstilbestrol (DES) 
Androgens	
N	androstenedione (ADD)  19-nor-4-androstene-3,17-diol (NAD)  androsterone (ADR)  epiandrosterone (EADR)  testosterone (TTR) 
S	methyl testosterone (MTTR)  trenbolone (TBL)  nandrolone (NDL)  stanozolol (SZL) 
Progestogens	
N	progesterone (PGT)  17a-hydroxyprogesterone (17-HPT)  21a-hydroxyprogesterone (21-HPT)  6a-methyl-hydroxyprogesterone (MHPT)  17a,20b-dihydroxy-4-pregnene-3-one (DPO) 
S	megestrol acetate (MTA)  norethindrone (NTD)  norgestrel (NGT)  medroxyprogesterone acetate (MPA) 
Mineralocorticoid (aldosterone) and glucocorticoids	
N	aldosterone (ADT)  cortisol (CRL)  cortisone (CRN) 
S	dexamethasone (DEX)  6a-methylprednisolone (MPREL)  prednisolone (PREL)  prednisone (PRE) 

N: natural steroid; S: synthetic steroid

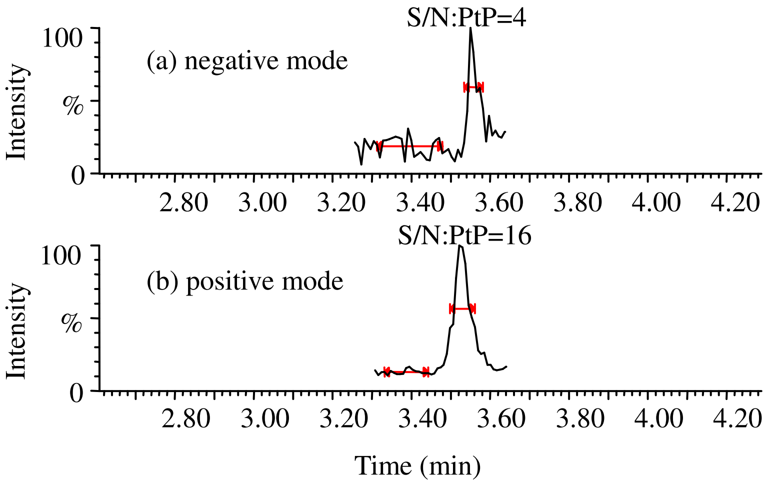


FIGURE S2. LC-ESI-MS/MS MRM chromatograms of βE2 analyzed in negative and positive mode for a sample collected at discharging site DBB1.

TABLE S1. Multi-selected Reaction Monitoring (MRM) Conditions of the Target Steroid Hormones

Steroid	MRM transition	Cone voltage (V)	Collision energy (eV)	Retain time (min)
Estrogen				
βE2-d$_3$	258 >159	33	20	3.52
βE2	255 > 159	33	20	3.53
	255 > 133		20	
αE2	255 >159	28	15	3.70
	255 >133		15	
EE2-d$_4$	283 > 135	30	21	3.77
EE2	279 > 133	30	21	3.78
	279 > 159		21	
E1-d$_2$	273 >159	31	19	3.92
E1	271 >159	31	19	3.89
	271 >197		17	
DES	269 > 135	20	14	4.05
	269 > 107		26	
Androgen and progestogen				
$^{13}C_2$-ethynyl-NTD	301 > 109	31	26	2.81
TBL	271 > 199	37	21	2.35
	271 > 253		19	
NAD	273 > 109	33	24	2.39
	273 > 197		18	
NDL	275 > 109	35	21	2.78
	275 > 257		15	
ADD	287 > 97	33	22	2.83
	287 > 109		24	
NTD	299 > 231	31	20	2.83
	299 > 109		26	
$^{13}C_2$-TTR	291 > 99	33	20	3.34
17-HPT	331 > 97	33	26	3.13
	331 > 109		24	
TTR	289 > 97	33	22	3.35
	289 > 109		22	
21-HPT	331 > 97	33	26	3.60
	331 > 109		24	
NGT-d$_6$	319 > 114	33	24	3.82
NGT	313 > 245	31	16	3.83
	313 > 109		26	
DPO	333 > 97	33	24	3.95
	333 > 109		30	
MTTR	303 > 97	33	23	3.98
	303 > 109		23	
EADR	291 > 255	25	12	4.32
	291 > 273		10	
PGT-d$_9$	324 > 100	33	22	5.47
SZL	329 > 81	47	40	4.76
	329 > 95		40	
MHPT	345 > 123	39	24	4.81
	345 > 97		24	
MTA	385 > 267	25	20	4.96
	385 > 325		14	
PGT	315 > 97	32	24	5.49
	315 > 109		24	
MPA	387 > 327	29	14	5.33
	387 > 285		18	
ADR	291 > 255	20	12	6.02
	291 > 273		10	
Mineralocorticoid (aldosterone) and glucocorticoid				
CRL-d$_2$	423 > 333	18	22	5.45
ADT	419 > 331	16	18	4.33

	419 > 359		12	
PRE	417 > 327	17	14	4.73
	417 > 357		10	
CRN	419 > 329	25	15	4.91
	419 > 359		10	
PREL	419 > 329	25	15	5.45
	419 > 359		11	
CRL	421 > 331	19	18	5.45
	421 > 361		12	
DEX	451 > 361	27	16	6.40
	451 > 391		11	
MPREL	433 > 343	23	12	6.47
	433 > 373		16	

TABLE S2. Recoveries (%) and Method Detection Limits (MDLs, ng/L) in Various Water Matrices

compound	Recovery[a] ±RSD		MDL	
	Sample from discharging site[b] 0.5L	River water[c] 2L	Sample from discharging site	River water
Estrogen				
βE2	78±6.3	80±7.9	0.25	0.10
αE2	82±5.7	81±5.3	0.04	0.02
EE2	85±7.3	83±8.5	0.30	0.10
E1	75±4.2	82±6.6	0.60	0.20
DES	81±7.9	86±3.6	0.60	0.25
Androgen and progestogen				
TBL	78±6.7	88±3.4	0.50	0.15
NAD	88±4.6	87±8.4	0.80	0.20
NDL	83±9.8	89±5.5	2.4	0.60
ADD	80±9.1	90±8.3	0.6	0.63
NTD	88±7.4	86±3.8	1.2	0.30
17-HPT	86±8.1	83±7.6	0.30	0.10
TTR	89±4.4	84±5.8	0.20	0.03
21- HPT	85±7.2	91±4.6	0.30	0.10
NGT	80±6.3	83±7.8	0.40	0.08
DPO	81±5.7	85±4.9	1.2	0.50
MTTR	83±7.3	81±8.6	0.80	0.20
EADR	84±5.2	86±7.5	20	12
SZL	89±9.9	91±7.9	0.24	0.06
MHPT	87±6.8	83±7.7	0.20	0.05
MTA	87±3.8	80±4.8	0.12	0.03
MPA	89±5.4	83±4.4	0.08	0.02
PGT	91±7.2	83±12	0.30	0.13
ADR	88±5.8	82±6.7	10	5.0
Mineralocorticoid and Glucocorticoid				
ADT	84±6.9	75±3.2	0.5	0.25
PRE	94±5.4	80±6.5	0.08	0.04
CRN	84±11	78±7.9	0. 04	0.02
PREL	86±2.2	81±6.5	0.05	0.02
CRL	83±5.7	84±4.8	0.10	0.04
DEX	87±4.3	84±9.4	0.02	0.008
MPREL	90±2.8	84±9.7	0.04	0.02

[a] Mean values from three determinations by external standard quantification procedures (n=3).

[b] Spiked concentration in the range of 5.0-25 ng/L for estrogens, 5.0-200 ng/L for most of androgens and progestogens (1.5 μg/L for ADD, ADR and EADR), 5.0-600 ng/L for mineralocorticoids and glucocorticoids.

[c] Spiked concentration at 1-5 ng/L.

TABLE S3. Signal Suppression (%) of Target Steroid Hormones in Various Water Matrices [a]

compound	Signal Suppression	
	Sample from discharging site [a]	River water [b]
Estrogen		
βE2	14	6.0
αE2	13	5.0
EE2	11	4.0
E1	8.0	7.0
DES	7.0	4.0
Androgen and progestogen		
TBL	6.0	6.0
NAD	4.0	6.0
NDL	8.0	4.0
ADD	10	8.0
NTD	5.0	5.0
17-HPT	6.0	6.0
TTR	10	6.0
21- HPT	11	4.0
NGT	8.0	3.0
DPO	7.0	4.0
MTTR	10	5.0
EADR	10	6.0
SZL	11	4.0
MHPT	8.0	8.0
MTA	7.0	5.0
MPA	11	4.0
PGT	8.0	3.0
ADR	7.0	4.0
Mineralocorticoid and Glucocorticoid		
ADT	8.0	6.0
PRE	6.0	3.0
CRN	5.0	3.0
PREL	8.0	4.0
CRL	7.0	4.0
DEX	9.0	5.0
MPREL	6.0	3.0

[a] Spiked concentration in the range of 5.0-25 ng/L for estrogens, 5.0-200 ng/L for most of androgens and progestogens (1.5 µg/L for ADD, ADR and EADR), 5.0-600 ng/L for mineralocorticoids and glucocorticoids.

[b] Spiked concentration at 1-5 ng/L.

Table S4. Concentrations (ng/L) of Estrogens and Androgens in All Water Samples Including River, Discharged and STP Composite Effluent Samples in July and August 2006[a]

Sampling site	E1	βE2	αE2	ADD	NAD	ADR	EADR	TTR	SZL
Q1	3.1	1.3	0.28	37	ND	330	110	8.6	ND
DQE	2.4	0.13	0.04	0.98	ND	ND	ND	ND	ND
Q2	6.9	0.93	0.87	43	ND	ND	ND	ND	ND
Q3	4.6	0.48	0.61	20	ND	ND	ND	ND	ND
Q4	3.3	0.82	0.56	34	ND	ND	ND	ND	ND
Q5	2.2	0.81	0.43	34	ND	ND	ND	ND	ND
Q6	1.6	1.6	0.53	18	ND	ND	ND	ND	ND
DQ1	1.5	0.36	0.34	6.0	ND	ND	ND	0.65	ND
DQ2	1.3	0.42	0.22	6.7	ND	30	ND	ND	ND
DQ3	0.80	0.16	0.05	1.9	ND	ND	ND	0.23	ND
Q7	1.0	0.69	0.06	8.3	ND	ND	ND	ND	ND
B1	6.5	0.31	0.20	25	ND	130	42	ND	ND
BL1	4.0	0.45	0.34	12	ND	16	13	1.0	ND
DJE	0.25	0.10	0.08	2.4	ND	ND	ND	0.53	ND
BL2	3.4	0.26	0.09	79	1.2	120	48	4.8	ND
B2	3.2	0.74	ND	16	ND	49	17	1.5	ND
DB1	22	2.1	0.51	240	ND	500	260	14	ND
B3	3.2	0.40	0.17	7.5	ND	ND	13	0.77	ND
BB1	1.4	0.83	0.08	17	ND	ND	ND	ND	ND
DBE	0.52	0.11	0.05	4.1	ND	ND	ND	0.39	ND
BB2	8.0	1.2	0.58	37	ND	80	ND	ND	ND
DBB1	7.0	1.1	0.36	370	ND	170	270	15	ND
DBB2	1.2	1.5	ND	7.9	ND	ND	20	ND	ND
DBB3	2.5	0.48	0.15	6.1	ND	ND	25	1.2	ND
DBB4	ND	0.28	ND	2.3	ND	ND	ND	ND	ND
DBB5	3.1	0.53	0.15	6.0	ND	ND	ND	0.57	ND
BB3	1.0	0.70	0.14	5.4	ND	ND	ND	ND	ND
B4	1.9	0.17	0.17	4.4	ND	ND	ND	ND	ND
B5	0.99	0.22	0.19	4.0	ND	ND	ND	ND	ND
T1	0.73	0.81	0.18	4.6	ND	ND	ND	ND	ND
T2	3.6	0.18	0.13	3.9	ND	5.2	ND	0.53	ND
T3	3.4	0.27	0.15	4.4	ND	6.7	ND	ND	ND
T4	2.0	0.19	0.04	2.4	ND	ND	ND	ND	ND
DTE	0.93	0.12	0.04	4.2	ND	ND	ND	0.61	ND
T5	5.1	0.50	0.17	8.3	ND	ND	ND	ND	ND
T6	1.8	0.27	0.06	4.7	ND	ND	ND	ND	ND
DT1	4.8	1.5	0.39	86	ND	680	240	30	ND
DT2	1.4	0.35	ND	4.2	ND	ND	ND	ND	ND
T7	4.3	0.53	0.24	17	ND	33	12	1.3	ND
DT3	14	2.3	0.30	160	ND	1300	410	17	0.29
DT4	6.2	1.5	0.45	88	ND	640	210	15	ND

T8	5.1	0.98	0.21	67	ND	53	59	2.6	ND
W1	4.1	1.6	0.58	99	ND	250	ND	ND	ND
W2	0.90	0.47	0.24	28	ND	390	ND	ND	ND
W3	0.69	0.17	0.04	2.9	ND	ND	ND	0.44	ND
W4	0.93	0.18	0.06	2.6	ND	5.2	ND	0.38	ND
W5	3.2	0.33	ND	9.3	ND	ND	ND	ND	ND
W6	0.98	0.33	0.11	27	ND	ND	ND	ND	ND
WT1	5.3	0.56	0.91	75	ND	ND	ND	ND	ND
W7	1.0	0.13	0.09	19	ND	ND	ND	ND	ND
W8	2.0	0.44	0.08	2.2	ND	ND	ND	0.23	ND
W9	1.4	0.37	0.11	1.7	ND	5.1	ND	0.22	ND
W10	1.8	0.27	0.11	3.0	ND	5.3	ND	0.19	ND
WT2	0.40	0.15	ND	5.6	ND	17	12	0.88	ND
W11	2.5	0.26	0.09	9.2	ND	17	ND	0.31	ND
W12	1.6	0.24	0.06	3.5	ND	5.5	ND	0.23	ND
W13	3.4	0.25	0.10	8.3	ND	6.1	ND	0.49	ND
W14	1.1	0.14	0.15	12	ND	ND	ND	ND	ND
WT3	0.87	ND	ND	3.2	ND	ND	ND	0.19	ND
W15	1.1	0.11	0.14	12	ND	ND	ND	ND	ND
W16	2.1	0.21	0.14	13	ND	ND	ND	0.33	ND
W17	6.3	0.28	0.63	40	ND	17	ND	ND	ND

[a]Average of duplicate injections. ND: under the method detection limit.

TABLE S5. Concentrations (ng/L) of Progestogens and Glucocorticoids in All Water Samples Including River Samples, Samplies from Discharged Sites and STP Composite Effluents in July and August 2006[a]

Sampling site	PGT	17-HPT	21-HPT	MHPT	DPO	MTA	NTD	NGT	MPA	CRL	CRN	DEX	MPREL	PREL	PRE
Q1	7.7	0.40	1.1	ND	0.72	0.84	ND	ND	1.9	18	22	1.2	ND	1.2	1.3
DQE	1.1	ND	ND	ND	ND	ND	ND	ND	ND	0.57	0.26	ND	ND	0.72	ND
Q2	6.8	ND	ND	ND	ND	25	ND	ND	18	9.4	7.8	8.0	ND	0.72	2.4
Q3	4.0	ND	ND	ND	ND	10	ND	ND	7.5	4.3	2.3	3.8	ND	0.46	0.82
Q4	4.8	ND	ND	ND	ND	15	ND	ND	13	3.1	0.71	2.4	ND	0.29	ND
Q5	4.8	ND	ND	ND	ND	11	ND	ND	16	2.8	1.7	3.2	ND	0.59	ND
Q6	3.5	ND	ND	ND	ND	7.3	ND	ND	8.5	3.7	1.0	2.3	ND	0.44	0.91
DQ1	0.59	ND	ND	0.53	ND	0.55	ND	ND	0.81	0.33	0.33	0.20	ND	0.31	ND
DQ2	2.7	ND	ND	ND	ND	ND	ND	ND	3.0	0.31	0.50	0.50	ND	0.41	ND
DQ3	ND	ND	ND	0.21	0.51	ND	ND	ND	ND	0.13	0.18	0.08	ND	0.31	ND
Q7	1.1	ND	ND	1.2	ND	0.87	ND	ND	2.0	2.0	2.2	0.07	ND	0.30	ND
B1	3.8	ND	ND	ND	1.2	2.9	ND	ND	1.2	1.4	1.6	1.6	0.20	0.54	ND
BL1	2.5	ND	1.7	ND	1.5	ND	ND	ND	ND	5.9	16	0.77	ND	0.71	0.71
DJE	0.48	ND	0.44	0.37	0.50	0.13	ND	ND	ND	0.21	0.26	ND	ND	0.60	ND
BL2	26	1.1	3.4	0.58	0.93	1.0	ND	ND	0.24	20	29	0.95	0.41	1.1	1.0
B2	2.5	ND	3.0	1.9	2.7	1.7	ND	ND	0.45	13	24	1.7	ND	0.88	0.83
DB1	ND	25	1.5	0.97	4.0	ND	ND	ND	ND	31	57	2.7	ND	3.8	2.4
B3	1.3	ND	2.1	1.4	0.94	0.44	ND	ND	0.42	8.1	16	1.1	ND	0.92	0.68
BB1	1.5	ND	ND	ND	ND	1.5	ND	ND	6.9	0.13	0.40	1.3	ND	0.53	ND
DBE	1.4	ND	0.53	1.3	0.52	0.28	ND	ND	0.60	0.24	0.88	0.05	ND	0.51	ND
BB2	6.5	ND	ND	ND	ND	9.5	ND	ND	7.9	3.8	1.8	5.1	ND	0.46	0.93
DBB1	35	1.4	170	ND	3.2	ND	ND	ND	ND	100	250	5.2	ND	17	18
DBB2	2.4	ND	ND	ND	ND	ND	16	ND	ND	8.8	12	0.37	ND	1.1	1.3
DBB3	2.0	0.43	4.9	ND	1.3	ND	4.7	ND	ND	26	69	1.6	ND	2.3	3.0
DBB4	0.70	ND	ND	ND	ND	ND	ND	ND	ND	0.35	0.18	ND	ND	0.40	ND

DBB5	0.45	ND	ND	ND	ND	ND	ND	ND	ND	0.69	1.3	0.13	ND	0.40	0.13
BB3	0.59	ND	ND	0.53	ND	0.83	ND	ND	0.91	0.26	0.13	0.19	ND	0.27	ND
B4	0.71	ND	ND	1.7	ND	0.82	ND	ND	0.71	0.82	1.3	0.48	ND	0.32	0.14
B5	0.37	ND	ND	0.57	ND	0.56	ND	ND	0.96	0.58	0.51	0.47	ND	0.46	ND
T1	ND	ND	ND	1.1	ND	4.9	ND	ND	3.0	0.28	ND	0.30	ND	0.49	ND
T2	0.25	ND	ND	ND	ND	0.37	ND	ND	0.45	0.11	0.21	0.12	ND	0.34	0.08
T3	0.40	ND	ND	ND	ND	0.45	ND	ND	0.60	0.15	0.32	0.14	ND	0.25	0.05
T4	ND	ND	ND	ND	ND	ND	ND	ND	0.36	0.11	0.20	0.09	ND	0.31	0.09
DTE	1.2	ND	0.33	0.41	0.50	ND	ND	ND	ND	0.19	0.31	ND	ND	0.47	ND
T5	0.54	ND	ND	ND	ND	1.9	ND	ND	1.2	0.16	0.54	0.15	ND	0.36	0.12
T6	0.58	ND	ND	ND	ND	ND	ND	ND	0.44	0.59	0.80	0.27	ND	0.36	0.24
DT1	25	1.6	4.0	ND	1.7	2.0	1.6	ND	5.6	46	50	4.0	ND	2.1	4.8
DT2	0.86	ND	0.80	ND	ND	0.12	ND	ND	0.16	2.4	4.1	0.28	ND	0.42	0.38
T7	1.6	ND	ND	ND	1.1	1.1	ND	ND	1.7	1.6	2.2	1.3	ND	0.37	0.44
DT3	38	2.2	4.0	ND	1.5	0.49	5.4	6.2	17	19	26	1.8	ND	0.80	2.1
DT4	18	1.4	3.6	ND	1.2	0.38	1.7	ND	2.3	48	66	3.0	ND	2.0	7.0
T8	8.9	1.1	3.8	ND	2.0	0.23	ND	ND	0.04	12	23	1.2	ND	0.99	1.4
W1	ND	ND	ND	ND	ND	ND	ND	ND	12	0.63	ND	3.4	ND	1.8	ND
W2	1.7	ND	ND	ND	ND	ND	ND	ND	ND	0.20	0.38	0.07	ND	0.37	ND
W3	0.40	ND	ND	ND	ND	ND	ND	ND	ND	0.35	0.78	0.06	ND	0.29	ND
W4	0.32	ND	ND	ND	ND	ND	ND	ND	0.15	0.29	0.14	0.31	ND	0.32	0.25
W5	1.2	ND	ND	ND	ND	ND	ND	ND	0.47	0.88	1.1	0.43	ND	0.45	ND
W6	1.4	ND	ND	1.6	ND	ND	ND	ND	0.85	0.30	0.37	0.42	ND	0.33	ND
WT1	5.9	ND	ND	ND	ND	ND	ND	ND	3.4	3.2	2.6	2.5	ND	0.50	0.63
W7	0.63	ND	ND	0.42	ND	ND	ND	ND	0.71	0.45	0.36	0.45	ND	0.40	ND
W8	0.19	ND	ND	ND	ND	ND	ND	ND	0.11	0.46	0.61	0.30	ND	0.34	0.08
W9	0.16	ND	ND	ND	ND	ND	ND	ND	0.08	0.30	0.05	0.51	ND	0.32	0.04
W10	0.18	ND	ND	0.09	ND	ND	ND	ND	0.17	0.26	0.46	0.64	ND	0.36	ND

WT2	0.80	ND	ND	ND	ND	ND	ND	ND	ND	0.62	0.50	0.10	ND	0.28	ND
W11	0.63	ND	ND	ND	ND	ND	ND	ND	0.19	0.61	0.70	0.22	ND	0.33	0.07
W12	0.26	ND	ND	0.06	ND	ND	ND	ND	0.15	0.28	0.63	0.25	ND	0.32	0.04
W13	0.97	ND	0.29	ND	0.50	ND	ND	ND	0.12	1.1	1.5	0.35	ND	0.37	0.10
W14	0.19	ND	ND	0.52	ND	ND	ND	ND	0.53	0.16	0.07	0.24	ND	0.35	ND
WT3	0.18	ND	ND	ND	ND	ND	ND	ND	0.26	0.15	0.24	0.05	ND	0.35	ND
W15	0.52	ND	ND	ND	ND	ND	ND	ND	1.2	0.77	0.39	0.41	ND	0.42	ND
W16	0.38	ND	ND	0.33	0.54	0.40	ND	ND	0.48	0.49	0.60	0.51	ND	0.30	ND
W17	1.9	ND	ND	2.1	ND	12	ND	ND	34	2.2	0.62	5.1	ND	0.62	ND

[a] Average of duplicate injections. ND: under the method detection limit.