

**60 Organic Wastewater Compounds in Drinking Water, Wastewater Effluent, and the Big Sioux River, 2001–2004**

**Table 9.** Statistical summaries of analytical results for laboratory reagent-spike samples.

[Bold text indicates suspected endocrine-disrupting compound (EDC). Analytical method number: 1, Cahill and others (2004); 2, U.S. Geological Survey Organic Geochemistry Research Laboratory; 3, Lee and others (2004); 4, Zaugg and others (2002). RSD, relative standard deviation; --, not applicable]

Compound	Analytical method number	Footnote	Number of laboratory spikes	Minimum percent recovery	Median percent recovery	Maximum percent recovery	Percent recovery RSD
Human pharmaceutical compounds (HPCs)							
1,7-Dimethylxanthine, dissolved	1	1	16	62	81	119	19
Acetaminophen, dissolved	1	1	16	50	67	106	23
Caffeine, dissolved	1	1	16	68	75	101	10
Caffeine, dissolved	4	1	3	95	115	120	12
Carbamazepine, dissolved	1	1	16	56	69	90	13
Cimetidine, dissolved	1	2	16	6	27	41	41
Codeine, dissolved	1	1	16	61	71	167	34
Cotinine, dissolved	1	1	16	56	74	92	15
Cotinine, dissolved	4	2	3	43	44	45	3
Cotinine, whole water	3	2	4	48	62	113	42
Dehydronifedipine, dissolved	1	1	16	57	69	97	15
Diltiazem, dissolved	1	2	16	21	36	60	25
Diphenhydramine, dissolved	1	2	16	41	45	64	14
<b>Fluoxetine</b> , dissolved	1	2	16	20	29	55	33
Furosemide, dissolved	1	2	16	0	15	35	253
Gemfibrozil, dissolved	1	2	16	9	26	160	102
Ibuprofen, dissolved	1	2	16	32	44	100	38
Metformin, dissolved	1	2	16	0	0	4	118
Miconazole, dissolved	1	2	16	2	5	43	105
Ranitidine, dissolved	1	2	16	24	34	59	25
Salbutamol, dissolved	1	1	16	64	73	97	11
Thiabendazole, dissolved	1	1	16	67	70	90	11
Warfarin, dissolved	1	1	16	51	58	111	27
Human and veterinary antibiotic compounds (HVACs)							
Azithromycin, dissolved	1	2	16	0	7	21	75
Erythromycin, dissolved	1	2	16	0	10	23	69
Sulfamethoxazole, dissolved	1	2	16	34	49	81	25
Trimethoprim, dissolved	1	1	16	56	65	78	10
Major agricultural herbicides (MAHs)							
<b>Atrazine</b> , whole water	3	1	1	60	60	60	--
Metolachlor, dissolved	4	1	3	90	95	100	5
Metolachlor, whole water	3	1	4	34	78	92	36
Prometon, dissolved	4	1	3	90	110	115	13
Prometon, whole water	3	1	4	49	73	89	23
Household, industrial, and minor agricultural use compounds (HIACs)							
1,4-Dichlorobenzene, dissolved	4	1	3	60	65	85	19
1,4-Dichlorobenzene, whole water	3	2	4	20	24	89	86
<b>2,2',4,4'-Tetrabromodiphenyl ether</b> , whole water	3	1	1	47	47	47	--
3,4-Dichlorophenyl isocyanate, whole water	3	2	1	175	175	175	--
3-Methyl-1H-indole (skatol), dissolved	4	1	3	90	90	100	6
3-Methyl-1H-indole (skatol), whole water	3	1	4	38	69	107	40

**Table 9.** Statistical summaries of analytical results for laboratory reagent-spike samples.—Continued

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Compound	Analytical method number	Footnote	Number of laboratory spikes	Minimum percent recovery	Median percent recovery	Maximum percent recovery	Percent recovery RSD
Household, industrial, and minor agricultural use compounds (HIACs)—Continued							
<b>3-tert-Butyl-4-hydroxy anisole (BHA)</b> , dissolved	4	1	3	70	75	75	4
<b>3-tert-Butyl-4-hydroxy anisole (BHA)</b> , whole water	3	2	4	2	35	75	82
<b>4-Cumylphenol</b> , dissolved	4	1	3	85	105	115	15
<b>4-Cumylphenol</b> , whole water	3	1	4	50	76	116	34
<b>4-normal-Octylphenol</b> , dissolved	4	1	3	80	85	85	3
<b>4-normal-Octylphenol</b> , whole water	3	1	4	60	72	97	21
<b>4-tert-Octylphenol</b> , dissolved	4	1	3	80	85	90	6
<b>4-tert-Octylphenol</b> , whole water	3	1	4	58	68	94	23
5-Methyl-1H-benzotriazole, dissolved	4	1	3	57	81	94	24
5-Methyl-1H-benzotriazole, whole water	3	1	4	63	85	101	23
<b>7-Acetyl-1,1,3,4,4,6-hexamethyl tetrahydronaphthalene (AHTN)</b> , dissolved	4	1	3	85	90	100	8
<b>7-Acetyl-1,1,3,4,4,6-hexamethyl tetrahydronaphthalene (AHTN)</b> , whole water	3	1	4	50	71	114	35
Acetophenone, dissolved	4	1	3	85	100	110	13
Acetophenone, whole water	3	3	4	41	71	116	43
Anthraquinone, dissolved	4	1	3	75	85	90	9
Anthraquinone, whole water	3	1	4	39	72	89	31
<b>Benzophenone</b> , dissolved	4	1	3	80	95	100	11
<b>Benzophenone</b> , whole water	3	2	4	41	74	119	42
Bis(2-ethylhexyl) phthalate, whole water	3	1	1	70	70	70	--
<b>Bisphenol-A</b> , dissolved	4	1	3	80	85	90	6
<b>Bisphenol-A</b> , whole water	3	1	4	51	65	108	36
Bromacil, dissolved	4	1	3	88	95	113	13
Bromacil, whole water	3	1	4	51	72	92	23
Camphor, dissolved	4	1	3	90	95	95	3
Camphor, whole water	3	1	4	40	69	109	40
<b>Carbaryl</b> , dissolved	4	1	3	50	55	55	5
<b>Carbaryl</b> , whole water	3	2	4	14	65	121	66
<b>Chlorpyrifos</b> , dissolved	4	1	3	80	80	85	4
<b>Chlorpyrifos</b> , whole water	3	1	4	48	74	112	35
N,N-Diethyl- <i>meta</i> -toluamide (DEET), dissolved	4	1	3	75	95	100	15
N,N-Diethyl- <i>meta</i> -toluamide (DEET), whole water	3	1	4	47	79	100	29
<b>Diazinon</b> , dissolved	4	1	3	85	95	100	8
<b>Diazinon</b> , whole water	3	1	4	46	71	108	35
Dichlorvos, dissolved	4	2	3	4	6	7	19
Dichlorvos, whole water	3	2	4	33	74	105	44
<b>Diethyl phthalate</b> , whole water	3	2	1	44	44	44	--
D-Limonene, dissolved	4	2	3	37	47	65	29

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Compound	Analytical method number	Footnote	Number of laboratory spikes	Minimum percent recovery	Median percent recovery	Maximum percent recovery	Percent recovery RSD
Household, industrial, and minor agricultural use compounds (HIACs)—Continued							
D-Limonene, whole water	3	2	4	10	11	60	107
<b>1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethyl cyclopenta-g-2-benzopyran (HHCB)</b> , dissolved	4	1	3	75	90	95	12
<b>1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethyl cyclopenta-g-2-benzopyran (HHCB)</b> , whole water	3	1	4	50	76	99	27
Indole, dissolved	4	1	3	85	90	95	6
Indole, whole water	3	3	4	36	64	100	42
Isoborneol, dissolved	4	1	3	80	90	90	7
Isoborneol, whole water	3	1	4	40	76	95	34
Isophorone, dissolved	4	1	3	80	90	90	7
Isophorone, whole water	3	2	4	41	73	114	41
Isopropylbenzene (cumene), dissolved	4	2	3	41	49	70	28
Isopropylbenzene (cumene), whole water	3	2	4	9	13	67	110
Isoquinoline, dissolved	4	1	3	75	80	90	9
Isoquinoline, whole water	3	2	4	30	67	108	48
Menthol, dissolved	4	1	3	90	90	95	3
Menthol, whole water	3	2	4	41	72	120	44
Metalaxyl, dissolved	4	1	3	90	100	100	6
Metalaxyl, whole water	3	1	4	47	80	85	25
Methyl salicylate, dissolved	4	1	3	80	90	90	7
Methyl salicylate, whole water	3	1	4	37	69	104	40
<b>Nonylphenol diethoxylate (NP2EO)</b> , dissolved	4	1	3	97	97	109	7
<b>Nonylphenol diethoxylate (NP2EO)</b> , whole water	3	1	4	59	72	78	11
<b>Nonylphenol monoethoxylate (NP1EO)</b> , whole water	3	1	1	57	57	57	--
<b>Octylphenol diethoxylate (OP2EO)</b> , dissolved	4	1	3	79	86	86	5
<b>Octylphenol diethoxylate (OP2EO)</b> , whole water	3	1	4	46	75	109	34
<b>Octylphenol monoethoxylate (OP1EO)</b> , dissolved	4	1	3	69	79	79	7
<b>Octylphenol monoethoxylate (OP1EO)</b> , whole water	3	1	4	39	77	94	32
<i>para</i> -Cresol, dissolved	4	1	3	85	95	95	6
<i>para</i> -Cresol, whole water	3	1	4	43	69	110	39
<b><i>para</i>-Nonylphenol (NP)</b> , dissolved	4	1	3	86	89	92	3
<b><i>para</i>-Nonylphenol (NP)</b> , whole water	3	1	4	45	64	74	22
<b>Pentachlorophenol</b> , dissolved	4	1	3	35	53	64	29
<b>Pentachlorophenol</b> , whole water	3	2	4	44	62	257	95
Phenol, dissolved	4	1	3	95	100	120	13
Phenol, whole water	3	3	4	50	64	121	43

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Compound	Analytical method number	Footnote	Number of laboratory spikes	Minimum percent recovery	Median percent recovery	Maximum percent recovery	Percent recovery RSD
Household, industrial, and minor agricultural use compounds (HIACs)—Continued							
Tetrachloroethylene, dissolved	4	2	3	29	34	60	41
Tetrachloroethylene, whole water	3	2	4	5	8	38	104
Tributyl phosphate, dissolved	4	1	3	75	90	95	12
Tributyl phosphate, whole water	3	1	4	43	79	87	29
<b>Triclosan</b> , dissolved	4	1	3	75	85	85	7
<b>Triclosan</b> , whole water	3	1	4	60	73	90	17
Triethyl citrate (ethyl citrate), dissolved	4	1	3	90	105	110	10
Triethyl citrate (ethyl citrate), whole water	3	1	4	46	69	75	20
Triphenyl phosphate, dissolved	4	1	3	90	105	110	10
Triphenyl phosphate, whole water	3	1	4	65	75	111	25
Tri(2-butoxyethyl)phosphate, dissolved	4	4	3	115	130	140	10
Tri(2-butoxyethyl)phosphate, whole water	3	1	4	48	65	75	20
Tri(2-chloroethyl)phosphate, dissolved	4	1	3	90	110	115	13
Tri(2-chloroethyl)phosphate, whole water	3	1	4	42	71	93	30
Tri(dichloroisopropyl)phosphate, dissolved	4	1	3	90	115	130	18
Tri(dichloroisopropyl)phosphate, whole water	3	1	4	50	75	108	31
Polyaromatic hydrocarbons (PAHs)							
1-Methylnaphthalene, dissolved	4	1	3	80	80	90	7
1-Methylnaphthalene, whole water	3	2	4	35	46	103	55
2,6-Dimethylnaphthalene, dissolved	4	1	3	80	80	90	7
2,6-Dimethylnaphthalene, whole water	3	2	4	34	46	99	54
2-Methylnaphthalene, dissolved	4	1	3	80	80	90	7
2-Methylnaphthalene, whole water	3	2	4	33	51	114	60
<b>Anthracene</b> , dissolved	4	1	3	85	100	105	11
<b>Anthracene</b> , whole water	3	1	4	50	68	115	37
<b>Benzo[a]pyrene</b> , dissolved	4	1	3	75	80	85	6
<b>Benzo[a]pyrene</b> , whole water	3	1	4	46	68	110	37
Carbazole, dissolved	4	1	3	100	115	120	9
Carbazole, whole water	3	1	4	55	80	117	32
Fluoranthene, dissolved	4	1	3	90	100	110	10
Fluoranthene, whole water	3	1	4	60	72	112	29
Naphthalene, dissolved	4	1	3	70	75	80	7
Naphthalene, whole water	3	2	4	27	50	102	55
<b>Phenanthrene</b> , dissolved	4	1	3	90	90	100	6
<b>Phenanthrene</b> , whole water	3	1	4	45	67	111	38
<b>Pyrene</b> , dissolved	4	1	3	80	85	90	6
<b>Pyrene</b> , whole water	3	1	4	50	72	113	34
Sterol compounds (SCs)							
3- <i>beta</i> -Coprostanol, dissolved	4	1	3	79	80	81	2
3- <i>beta</i> -Coprostanol, whole water	3	1	4	64	71	76	8
<i>beta</i> -Sitosterol, dissolved	4	1	3	51	54	83	28
<i>beta</i> -Sitosterol, whole water	3	1	4	65	76	86	12
<i>beta</i> -Stigmastanol, dissolved	4	1	3	53	59	91	31

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<b>Compound</b>	<b>Analytical method number</b>	<b>Footnote</b>	<b>Number of laboratory spikes</b>	<b>Minimum percent recovery</b>	<b>Median percent recovery</b>	<b>Maximum percent recovery</b>	<b>Percent recovery RSD</b>
Sterol compounds (SCs)—Continued							
<i>beta</i> -Stigmastanol, whole water	3	1	4	60	74	87	18
Cholesterol, dissolved	4	1	3	68	83	88	13
Cholesterol, whole water	3	1	4	64	71	76	7

<sup>1</sup>Median percent recovery for reagent-spike samples near or within acceptable range (50–120 percent), and percent recovery RSD acceptable (less than 40 percent); laboratory-reagent spike results judged to be acceptable.

<sup>2</sup>Median percent recovery for reagent-spike samples outside of acceptable range (50–120 percent), or percent recovery RSD unacceptable (greater than 40 percent); compound excluded from analyses and discussion related to occurrence of organic wastewater compounds in drinking water, wastewater effluents, and the Big Sioux River.

<sup>3</sup>Median percent recovery for reagent-spike samples within acceptable range (50–120 percent), but percent recovery RSD slightly exceeded acceptable range (less than 40 percent); all other quality-assurance/quality-control results for compound were acceptable; laboratory-reagent spike results judged to be acceptable.

<sup>4</sup>Median percent recovery for reagent-spike samples exceeded acceptable range (50–120 percent), but percent recovery RSD within acceptable range (less than 40 percent); all other quality-assurance/quality-control results for compound were acceptable; laboratory-reagent spike results judged to be acceptable.