

Metropolitan Water Reclamation District of Greater Chicago

## MONITORING AND RESEARCH DEPARTMENT

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CONCENTRATIONS OF PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE AND BIOSOLIDS IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

July 2014

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### CONCENTRATIONS OF PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE AND BIOSOLIDS IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

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#### SUMMARY

Pharmaceuticals and personal care products (PPCPs) comprise a large and diverse group of chemical substances, including prescription and over-the-counter drugs, veterinary drugs, fragrances, cosmetics, and cleaning agents. Although PPCPs in wastewater effluent and biosolids are not currently regulated, continued media attention to trace concentrations of PPCPs in wastewater effluent lead the Metropolitan Water Reclamation District of Greater Chicago (District) to monitor for 11 targeted PPCPs in the District's seven water reclamation plants' (WRPs') influent, effluent, sludge, and biosolids. Semi-annual or annual monitoring was conducted from January 2010 through February 2014. Sample extractions were conducted using a Solid Phase Extractor, and analyses were performed using the High Performance Liquid Chromatograph with a Triple Quad Mass Spectrometer, using modified EPA Method 1694.

The Hanover Park Water Reclamation Plant (WRP) had the highest influent concentrations for 6 of the 11 target PPCPs, and the Lemont WRP had the other five highest influent concentrations. These two WRPs receive almost exclusively domestic wastewater, indicating the dominance of households as the source of these PPCPs in the District's wastewater treatment systems. There is no trend in influent, effluent, or biosolids concentrations over the five-year sampling period for any of the 11 PPCPs. Concentrations for all 11 PPCPs reported in the District's biosolids or waste-activated sludge samples were lower than concentrations reported in the United States Environmental Protection Agency's (USEPA's) Targeted National Sewage Sludge Survey (TNSSS) (2009). Removal efficiencies from wastewater influent were greatest for ibuprofen, naproxen, and triclosan, which were greater than 89 percent for all seven WRPs. Triclocarban and Gemfibrozil also had high removal efficiencies of greater than 80 percent and 50 percent, respectively. The lowest removal efficiencies were for codeine, diphenhydramine, and fluoxetine, which were less than 50 percent for most WRPs.

When removed from the wastewater stream, PPCPs volatilize, biodegrade, or adsorb to the solid particulates in sludge and move with sludge for the duration of the treatment process. The Hanover Park WRP had the highest concentrations for 9 of the 11 PPCPs in biosolids. Although these 11 targeted PPCPs were detected in biosolids samples, there is still much uncertainty regarding their migration from biosolids to the environment. Research suggests that the majority of the PPCPs detected in biosolids likely exert no acute effects on aquatic organisms when applied as required by the USEPA's Part 503 biosolids regulation.

Concentrations for 9 of the 11 PPCPs in the District's effluent were compared to concentrations from other wastewater treatment plants published in scientific papers. The highest concentrations of carbamazepine, ciprofloxacin, codeine, fluoxetine, gemfibrozil, ibuprofen, naproxen, and triclosan in the District's effluent were lower than the values reported in effluent concentrations from other wastewater treatment plants reported in published scientific papers. Although PPCPs have been detected in wastewater effluent, there is currently no known risk to human health, and understanding the potential risks to aquatic organisms continues to be an active area of research. Further research is warranted to monitor concentrations of PPCPs in influent, effluent, and biosolids to asses their fate and transport in surface waters and biosolids-amended soils.

#### **INTRODUCTION**

Pharmaceuticals and personal-care products generally refer to any product used by individuals for health or cosmetic reasons or used by agribusiness to enhance the growth or health of livestock. These compounds comprise a large and diverse group of chemical substances, including prescription and over-the-counter drugs, veterinary drugs, fragrances, cosmetics, and cleaning agents. Although evidence suggests that certain endocrine-disrupting drugs may have adverse effects on aquatic organisms, there is no evidence of adverse human health effects from PPCPs in the environment. There has been increasing attention given to PPCPs concerning the fate and transport of these compounds in the environment from wastewater effluent and biosolids application to land (Boxall et al., 2012; Kinney et al., 2006); however, these chemicals have been present in wastewater effluent and surface waters for as long as they have been produced and used by consumers. Recent advances in analytical technology now allow for improved quantification of these chemicals in trace concentrations. Pharmaceutical and personal-care products are continuously released into wastewater streams and ultimately reach wastewater treatment plants (WWTPs) where they partition to sludge solids, are biodegraded, volatilize, or remain soluble in the WWTP's effluent. Although PPCPs are not currently regulated in wastewater effluent and biosolids, continued media attention to trace concentrations of PPCPs in wastewater effluent lead the District to monitor for PPCPs in the District's seven WRPs' influent, effluent, sludge, and biosolids.

The USEPA conducted four sewage sludge surveys since 1982, the most recent survey being the TNSSS in 2009. The 2009 TNSSS collected sewage sludge from 74 randomly selected, publicly owned treatment works in 35 states in 2006 and 2007 and provided results for 145 analytes, including 72 pharmaceuticals and 25steroids and hormones. Some PPCPs were found in all samples, while others were found in none or only a few of the sewage sludge samples (USEPA, 2009). Additionally, there have been a number of studies conducted investigating the presence of PPCPs in wastewater effluent (Andreozzi et al., 2003; Rosal et al., 2010; Barber et al., 2011; Deblonde et al., 2011; Waiser et al., 2011; Loos et al., 2013).

This report presents concentrations for 11 targeted PPCPs in the District's seven WRPs from semi-annual or annual monitoring conducted from January 2010 through February 2014. These 11 targeted PPCPs were monitored in influent, effluent, and waste-activated sludge or biosolids samples in all seven WRPs. The targeted PPCPs, their use in consumer products, select physical properties, and range in concentration in biosolids from two published studies are listed in <u>Table 1</u> (Kinney et al., 2006; USEPA, 2009). To better understand how PPCP concentrations found in the District's WRPs compare to other treatment plants, monitoring data are compared to other wastewater effluent data published in scientific papers.

### TABLE 1: ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS MONITORED IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS' INFLUENT, EFFLUENT, SLUDGE, AND BIOSOLIDS COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014, THEIR USE IN CONSUMER PRODUCTS, AND RANGE IN CONCENTRATIONS IN BIOSOLIDS FROM TWO PUBLISHED STUDIES<sup>1,2</sup>

РРСР	Use in Consumer Product	Octanol-Water Partitioning Coefficient <sup>3</sup>	Solubility in Water at 25°C (mg/L) <sup>3</sup>	Molecular Weight <sup>3</sup>	Range in Concentration (µg kg <sup>-1</sup> )
Carbamazepine	Anticonvulsant	2.45	17.7	236.2	ND - 6,030 <sup>1,2</sup>
Ciprofloxacin	Antibiotic	0.28	30,000 (20°C)	331.3	75 - 40,800 <sup>1,2</sup>
Codeine	Analgesic	1.19	9,000 (20°C)	299.4	ND - 328 <sup>1,2</sup>
Diphenhydramine	Antihistamine	3.27	3,060 (37°C)	255.4	12 - 7,018 <sup>1,2</sup>
Fluoxetine	Antidepressant	4.05	60.3	309.3	ND - $3,140^{1,2}$
Gemfibrozil	Antihyperlipidemic	4.77	19	250.3	ND - 2,650 <sup>1,2</sup>
Ibuprofen	Analgesic	3.97	21	206.2	ND - 11,900 <sup>1</sup>
Naproxen	Analgesic	3.18	15.9	230.3	ND - $1,020^2$
Thiabendazole	Anthelminitic	2.47	50	201.3	$1 - 5,000^{1,2}$
Triclocarban	Antimicrobial	4.9	11 (20°C)	315.6	$187 - 441,000^{1}$
Triclosan	Antimicrobial	4.76	10 (20°C)	289.5	ND - 133,000 <sup>1,2</sup>

<sup>1</sup>USEPA, 2009.

 $^{2}$ Kinney et al., 2006.

<sup>3</sup>USNLM, 2014.

#### **METHODS AND MATERIALS**

Samples were collected from the influent, effluent, and sludge or biosolids (i.e. digester draw biosolids for Egan, Hanover Park, Stickney, and Calumet WRPs; waste-activated sludge for O'Brien and Lemont WRPs; return sludge for Kirie WRP) biannually from all seven WRPs for 2010 - 2011. Only one sampling event per year was conducted for 2012 - 2014.

Samples were collected in glass bottles (i.e., a gallon for influent and effluent, a quart for sludge) at 4:00 a.m. and continually every four hours for a total of six collections. Sample vials were placed on ice in coolers for transport to the laboratory.

Sample extractions were conducted by the Organic Compounds Analytical Laboratory located at the John E. Egan WRP using the Oasis Disk (47 mm HLB) on the Automated Solid Phase Extractor (Horizon SPE-DEX 4790), and analyses were performed using the High Performance Liquid Chromatograph with a Triple Quad Mass Spectrometer (Agilent 1200 HPLC and 6410B Triple Quad MS), using modified EPA Method 1694 (USEPA, 2007).

#### **RESULTS AND DISCUSSION**

The mean concentrations of 11 PPCPs in influent and effluent samples and their removal efficiencies for the District's seven WRPs from January 2010 to February 2014 are summarized in <u>Table 2</u>. Concentrations for individual sampling dates for influent, effluent, and removal efficiency for each WRP for the duration of the sampling period are available in <u>Appendix A</u>. Mean concentrations for analytes in biosolids and waste-activated sludge samples are summarized in <u>Table 3</u>. Concentrations in biosolids and waste-activated sludge samples for individual sampling dates for each WRP for the duration of the sampling period are available in <u>Appendix B</u>.

The Hanover Park WRP had the highest influent concentrations for 6 of the 11 target PPCPs, and the Lemont WRP had the other 5 highest influent concentrations (Table 2). These two WRPs receive almost exclusively domestic wastewater, indicating the dominance of households as the source of these PPCPs in the District's wastewater treatment systems. These compounds exhibit low solubility due to their structural properties and are expected to be removed from wastewater by partitioning to solids. The ring-structure and molecular weight of these compounds contribute to their low solubility in water as indicated by their relatively high octanol-water partitioning coefficients, except for ciprofloxacin, which is the most soluble of all the compounds (Table 1). Removal efficiency, defined here as the difference between influent and effluent concentrations (Table 2), was greatest for ibuprofen, naproxen, and triclosan and ranged from 89 to 100 percent for all seven WRPs. Triclocarban and gemfibrozil also had high removal efficiencies, which were greater than 80 percent and 50 percent, respectively, for all seven WRPs. Although ciprofloxacin is the most water soluble of the compounds, its removal efficiency was as high as 80 percent in the Kirie WRP. The lowest removal efficiencies were for codeine, diphenhydramine, and fluoxetine, which were less than 50 percent for most WRPs. Removal efficiency results for carbamazepine and thiabendazole are inconclusive due to the high variability in data and concentrations being near the level of quantification.

When removed from the wastewater stream, PPCPs will volatilize, biodegrade, or adsorb to solid particulates in sludge and move with sludge for the duration of the treatment process. The Hanover Park WRP had the highest concentrations for 9 of the 11 PPCPs in biosolids (Table\_3). Carbamazepine, ciprofloxacin, diphenhydramine, fluoxetine, gemfibrozil, ibuprofen, thiabendazole, triclocarban, and triclosan were all greatest at Hanover Park; however, the concentrations of these compounds were all less than the values reported in the two published studies (Table 1). Triclocarban and triclosan were present in District biosolids at  $35.3\pm7.2$  and 30.5±5.5 mg kg<sup>-1</sup>, respectively. The 2009 TNSSS reported values for triclocarban and triclosan up to 441 and 133 mg kg<sup>-1</sup>, respectively. Although these 11 targeted PPCPs were detected in biosolids samples, the USEPA has previously evaluated organic pollutants of similar chemical structure and properties in the initial 40 CFR Part 503 Standards for the Use or Disposal of Sewage Sludge (Part 503). The USEPA conducted comprehensive risk assessments on 12 organic pollutants, including known carcinogens such as DDT, benzo(a)pyrene, hexachlorobenzene, and polychlorinated biphenyls (PCBs). The risk assessment used 14 exposure pathways, including direct ingestion of biosolids by a child, and concluded that the risk to human health from exposure to these 12 organic pollutants in biosolids-amended soil was negligible.

## TABLE 2: MEAN<sup>1</sup> CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

WRP <sup>2</sup>	<u>Carbamazepine<sup>3</sup></u> Influent Effluent		<u>Ciprofloxacin</u> Influent Effluent Removal			Influent	<u>Codeine</u> Effluent	Removal	Diphenhydramine Influent Effluent Removal			
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Stickney <sup>4</sup>	0.14±0.03	0.14±0.04	1.9±1.5	0.33±0.1	77±13	0.10±0.02	0.07±0.04	26±46	0.39±0.14	0.27±0.07	27±19	
Calumet	0.17±0.05	0.17±0.05	1.6±0.99	0.67±0.32	47±26	0.17±0.06	0.13±0.07	23±31	0.50±0.26	0.29±0.13	37±18	
Hanover Park	0.27±0.05	0.28±0.05	4.1±3.1	0.76±0.64	77±16	0.14±0.05	0.09±0.07	36±50	0.99±0.32	0.25±0.14	75±16	
Lemont	0.33±0.08	0.36±0.14	4.3±3.6	1.5±0.93	54±27	0.28±0.21	0.20±0.18	30±32	1.1±0.60	0.50±0.27	44±29	
Egan	0.19±0.03	0.21±0.04	3.5±1.8	0.73±0.63	79±14	0.14±0.04	0.15±0.10	13±64	0.72±0.33	0.18±0.12	74±17	
Kirie	0.23±0.04	0.25±0.07	2.5±1.3	0.58±0.44	80±11	0.10±0.03	0.05±0.03	48±25	0.65±0.30	0.35±0.11	41±20	
O'Brien	0.32±0.39	0.18±0.06	2.1±0.89	0.78±0.43	63±10	0.09±0.04	0.08±0.04	-3.8±59 <sup>5</sup>	0.43±0.19	0.36±0.09	8.8±23	

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## TABLE 2 (Continued): MEAN<sup>1</sup> CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PER-SONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE MET-ROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

WRP <sup>2</sup>		Fluoxet	ine		Gemfibr	ozil		Ibupro	fen	Naproxen		
WKr	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
Stickney <sup>4</sup>	0.03±0.01	0.01±0.0	61±12	0.58±0.17	0.28±0.18	54±24	6.1±2.3	0.33±0.18	94±3.4	5.6±2.3	0.23±0.19	96±2.8
Calumet	0.02±0.02	0.01±0.01	31±42	0.84±0.15	0.34±0.27	60±31	6.9±3.0	0.70±1.6	91±21	7.8±3.0	0.95±2.0	89±22
Hanover Park	0.07±0.03	0.04±0.01	44±17	1.8±0.45	0.44±0.43	76±23	11.4±3.5	0.06±0.04	100±0.44	13.0±5.1	0.17±0.18	99±1.2
Lemont	0.06±0.03	0.03±0.01	37±22	1.0±0.39	0.41±0.35	60±29	14.5±8.0	0.83±1.3	95±5.8	12.3±6.4	0.50±0.63	96±3.6
Egan	0.06±0.02	0.04±0.01	36±10	1.2±0.45	0.22±0.18	83±14	9.9±3.6	0.01±0.0	100±0.09	9.8±4.4	0.04±0.04	100±0.40
Kirie	0.04±0.01	0.03±0.01	26±13	1.4±0.48	0.26±0.16	80±16	10.1±4.2	0.02±0.01	100±0.20	9.8±4.9	0.06±0.04	99±1.0
O'Brien	0.03±0.02	0.02±0.01	27±20	1.1±0.31	0.42±0.31	61±29	8.5±2.5	0.05±0.07	99±0.83	7.4±2.9	0.16±0.20	97±3.7

## TABLE 2 (Continued): MEAN<sup>1</sup> CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

	Thiaben	dazole <sup>3</sup>		Triclocarba	n		Triclosan			
WRP <sup>2</sup>	Influent	Effluent	Influent	Effluent	Removal	Influent	Effluent	Removal		
Stickney <sup>4</sup>	0.02±0.01	0.01±0.01	2.1±1.4	0.09±0.03	95±1.9	3.2±1.2	0.08±0.03	97±1.6		
Calumet	0.01±0.01	0.01±0.01	2.4±1.5	0.21±0.06	83±21	6.8±5.9	0.23±0.24	89±22		
Hanover Park	0.19±0.20	0.25±0.28	4.9±4.6	0.25±0.05	92±5.2	<b>8.8</b> ±5.1	$0.09 \pm 0.07$	98±1.5		
Lemont	0.02±0.01	0.03±0.01	3.1±1.8	0.23±0.11	87±12	6.9±2.5	0.13±0.07	98±1.5		
Egan	0.04±0.02	0.03±0.01	3.5±1.4	0.12±0.05	96±1.8	7.5±2.0	0.07±0.06	99±0.82		
Kirie	0.04±0.01	0.03±0.01	2.4±1.1	0.10±0.04	95±1.5	7.6±3.5	0.07±0.03	99±0.64		
O'Brien	$0.02 \pm 0.01$	$0.03 {\pm} 0.01$	2.0±0.54	0.20±0.09	90±4.0	4.6±1.2	0.17±0.09	96±2.9		

<sup>1</sup>Mean of six samples for carbamazepine, ciprofloxacin, codeine, diphenhydramine, fluoxetine, and thiabendazole. Mean of seven samples for gemfibrozil, ibuprofen, naproxen, triclocarban, and triclosan.

<sup>2</sup>Water Reclamation Plant.

<sup>3</sup>Removal efficiencies for carbamazepine and thiabendazole are inconclusive due to the high variability in data and concentrations being near the level of quantification.

<sup>4</sup>Mean of ten influent samples for carbamazepine, ciprofloxacin, diphenhydramine, and fluoxetine. Mean of 12 influent samples for codeine, gemfibrozil, ibuprofen, and naproxen. Separate samples were taken from the west side and southwest wastewater streams at the Stickney Water Reclamation Plant.

<sup>5</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value.

### TABLE 3: MEAN<sup>1</sup> CONCENTRATIONS OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS AND WASTE-ACTIVATED SLUDGE SAMPLES<sup>2</sup> IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014 AND COMPARISON TO CONCENTRATIONS FROM TWO PUBLISHED STUDIES

WRP <sup>3</sup>	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine	Fluoxetine	Gemfibrozil
			ug kg <sup>-1</sup> dry-we	ight basis		
Published Studies⁴	ND - 6,030	75-40,800	ND - 328	12 - 7,018	ND - 3,140	ND – 2,650
Stickney	31.2±16.9	7,684±4,663	15.4±17.9	665±401	72.7±39.1	68.8±46.7
Calumet	53.5±24.2	10,362±6,373	27.0±15.2	713±378	79.4±33.0	186±119
Hanover Park	127±16.9	28,160±12,561	20.7 ±15.9	1042±351	256±130	447±138
Lemont	96.6±46.1	14,057±6,471	117±82.7	484±193	146±28.6	200±84.3
Egan	69.8±32.0	16,829±8,884	23.7±16.5	790±368	189±67.2	160±64.8
Kirie	48.2±19.1	10,423±5,536	92.5±62.8	403±169	115±24.7	95.2±98.0
O'Brien	68.3±59.1	11,001±5,933	46.2±47.7	346±101	77.3±15.0	148±68.7

## TABLE 3 (Continued): MEAN<sup>1</sup> CONCENTRATIONS OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS AND WASTE-ACTIVATED SLUDGE SAMPLES<sup>2</sup> IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014 AND COMPARISON TO CONCENTRATIONS FROM TWO PUBLISHED STUDIES

WRP <sup>3</sup>	Ibuprofen	Naproxen	Thiabendazole	Triclocarban	Triclosan
		μg kg	<sup>-1</sup> dry-weight basis -		
Published <sup>4</sup> Studies	ND – 11,900	ND – 1,020	1 – 5,000	187 - 441,000	ND – 133,000
Stickney	854±851	44.3±37.3	21.9±8.7	7,488±3,343	4,094±2,042
Calumet	995±510	109±201	16.5±4.5	17,292±5,017	6,682±3,454
Hanover Park	1,719±831	27.3±10.8	408±319	35,277±7,238	30,506±5,539
Lemont	429±261	243±229	55.7±43.1	9,605±3,874	2,668±941
Egan	1,340 ±653	34.7±25.5	39.0±16.7	14,902±7,517	12,096±5,613
Kirie	272±176	69.1±54.8	34.3±21.6	8,478±3,167	2,487±818
O'Brien	540±208	331±250	28.0±19.5	7,638±4,255	6,603±3,625

Mean of six samples for carbamazepine, ciprofloxacin, codeine, diphenhydramine, fluoxetine, and thiabendazole. Mean of seven samples for gemfibrozil, ibuprofen, naproxen, triclocarban, and triclosan. <sup>2</sup>Samples from Lemont, Kirie, and O'Brien are waste-activated sludge.

<sup>3</sup>Water Reclamation Plant.

<sup>4</sup>Range in concentration from two published studies (Kinney et al., 2006; USEPA, 2009).

McClellan and Halden (2010) analyzed for 72 PPCPs in 110 biosolids samples collected by the USEPA in the 2001 National Sewage Sludge Survey. Of the 72 PPCPs analyzed for, 38 were detected. The two most abundant PPCPs were triclocarban (48 percent of total detected PPCP mass) and triclosan (17 percent). Their mean concentrations were  $36.0\pm8.0$  and  $12.6\pm3.8$ mg kg<sup>-1</sup> on a dry-weight basis, respectively. In a similar study, Higgens et al. (2011) measured concentrations of triclocarban and triclosan in municipal biosolids at  $9.2\pm1.6$  and  $6.4\pm0.3$  mg kg<sup>-1</sup> on a dry-weight basis, respectively. The second most abundant class of PPCPs found by McClellan and Halden (2010) was antibiotics; ciprofloxacin was found in the highest concentration at  $6.8\pm2.3$  mg kg<sup>-1</sup> on a dry-weight basis.

McClellan and Halden (2010) also estimated porewater concentrations for PPCPs and concluded that the leaching of dissolved PPCPs into surface waters is not an important pathway for exposure of aquatic organisms for the majority of analytes detected. Concentrations calculated for soil porewater were several orders of magnitude below the lowest effect concentration<sup>1</sup> reported for aquatic organisms tested. There were several notable exceptions, including ciprofloxacin, triclosan, and triclocarban. They concluded that the majority of the PPCPs detected in biosolids likely exert no acute effects on aquatic organisms, assuming that biosolids are applied as required by the USEPA's Part 503 biosolids regulation (McClellan and Halden, 2010). Similarly, Topp et al. (2008) targeted PPCPs, including carbamazepine, gemfibrozil, naproxen, ibuprofen, and triclosan, in agricultural runoff from the application of biosolids and found that in injected plots, concentrations of PPCPs were generally below the level of detection. Although broadcast application of biosolids to agricultural fields resulted in detectable concentrations in runoff, overall the study demonstrated that the injection of biosolids below the soil surface could effectively eliminate surface runoff of PPCPs. Biodegradation data on PPCPs as a class of compounds is sparse, but data on triclocarban and triclosan suggest that although PPCPs may not be mobile in biosolids-amended soils, they may biodegrade slowly and persist for years, depending on soil depth and type of biosolids applied (Al-Rajab et al., 2009; Snyder et al., 2010; Xia et al., 2010).

Concentrations for nine PPCPs in the District's effluent and corresponding values reported in published scientific papers for other wastewater treatment plants are presented in <u>Table 4</u>. Overall, the highest concentrations of carbamazepine, ciprofloxacin, codeine, fluoxetine, gemfibrozil, ibuprofen, naproxen, and triclosan in the District's effluent were lower than values reported in effluent concentrations from other wastewater treatment plants. Waiser et al. (2011) found that concentrations of PPCPs were highest within the first 10 km of a treatment plant with a flow of 1.6 million gallons per day (MGD); however, codeine and carbamazepine were detected 105 km and 60 km, respectively, downstream from the same treatment plant. Based on observations that concentrations of PPCPs were higher in spring sampling periods than in summer periods, Waiser et al. (2011) also concluded that temperature plays an important role in the degradation of PPCPs in surface water.

Although PPCPs have been detected in wastewater effluent, there is currently no known risk to human health, and understanding the potential risks to aquatic organisms continues to be

<sup>&</sup>lt;sup>1</sup>The lowest level of a chemical stressor evaluated in a toxicity test that shows harmful effects on a plant or animal.

### TABLE 4: RANGE IN CONCENTRATIONS FOR PHARMACEUTICALS AND PERSONAL CARE PRODUCTS IN EFFLUENT SAMPLES FROM THE METROPOLITAN WATER RECLAMATIONDISTRICT OF GREATER CHICA-GO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014 AND THEIR COMPARISON TO PUBLISHED EFFLUENT CONCENTRATIONS

РРСР	Present Study	Waiser et al., 2011	Andreozzi et al., 2003	Barber et al., 2011 <sup>1</sup>	Deblonde et al., 2011	Rosal et al., 2010	Loos et al., 2013
				μg L <sup>-1</sup>			
Carbamazepine	0.14 - 0.36	0.35	0.30 - 1.2	. 3.2	0.04 - 2.1	0.07 - 0.17	4.6
Ciprofloxacin	0.33 - 1.5	0.03	$NA^2$	0.13 <sup>3</sup>	0.01 - 2.4	$ND^{4} - 5.7$	0.26
Codeine	0.05 - 0.20	NA	NA	NA	1.9	ND - 0.16	0.26
Diphenhydra- mine	0.18 - 0.50	NA	NA	0.02			0.14
Fluoxetine	0.01 - 0.04	NA	NA	0.02 <sup>3</sup>	0.11	0.03 - 0.22	0.02
Gemfibrozil	0.22 - 0.44	4.2	0.06 - 2.1	0.64 <sup>3</sup>	0.01 - 2.9	ND - 0.85	3.6
Ibuprofen	0.01 - 0.83	1.6	0.02 - 7.1	$1.4^{3}$	0.03 - 12.6	ND - 0.14	2.1
Naproxen	0.04 - 0.95	2.7	0.29 - 5.2	NA	0.02 - 2.6	0.36 - 0.92	0.96
Triclosan	0.07 - 0.23	0.11	NA	0.46	0.01 - 0.22	ND - 0.22	4.3

<sup>1</sup>Values are composite sample (mean of 3-10 sampling events) except where noted. Samples taken from the District's North Shore Channel.

 $^{2}$ NA = no data available.

<sup>3</sup>Value is composite sample (mean of 21-48 sampling events) calculated from supplemental data.

 $^{4}$ ND = non-detect.

an active area of research. Further research is warranted to monitor concentrations of PPCPs in influent, effluent, and biosolids to asses their fate and transport in surface waters and biosolids-amended soils.

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#### APPENDIX A

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CONCENTRATIONS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCIES: INDIVIDUAL SAMPLING DATES FROM THE METROPOLITAN RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

## TABLE A-1: CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Carbamazepine Influent <sup>1</sup> Effluent Removal			Ciprofloxacin Influent Effluent Removal			Codeine Influent Effluent Removal			Diphenhydramine Influent Effluent Removal		
7/22/2010	0.13	0.16	-16 <sup>2</sup>	0.40	0.16	59	0.13	0.02	81	0.25	0.22	10
1/27/2011	0.15	0.15	-1.9	1.5	0.34	78	0.08	0.13	-48	0.39	0.38	1.6
7/21/2011	0.17	0.17	-2.0	4.7	0.42	91	0.10	0.03	69	0.62	0.29	53
1/26/2012	0.10	0.09	15	1.8	0.64	65	0.06	0.05	23	0.26	0.20	23
1/15/2013	0.18	0.17	1.3	1.9	0.23	88	0.12	0.10	18	0.47	0.33	29
1/14/2014	0.14	0.10	31	1.1	0.20	82	0.10	0.09	13	0.35	0.20	42

## TABLE A-1 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

	Fluoxetine			(	Gemfibrozil			Ibuprofe	n		Naproxen		
Sampling Date -	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	
1/28/2010	NA <sup>3</sup>	NA	NA	0.58	0.30	47	3.6	0.36	90	3.2	0.17	95	
7/22/2010	0.02	0.01	75	0.38	0.06	85	3.0	0.09	97	2.3	0.09	96	
1/27/2011	0.03	0.02	45	0.91	0.58	36	9.5	0.37	96	9.1	0.25	97	
7/21/2011	0.04	0.01	75	0.62	0.08	88	6.1	0.16	97	5.6	0.18	97	
1/26/2012	0.02	0.01	52	0.51	0.35	31	5.9	0.59	90	5.3	0.25	95	
1/15/2013	0.03	0.01	63	0.60	0.23	62	7.9	0.26	97	7.0	0.05	99	
1/14/2014	0.02	0.01	56	0.49	0.33	32	6.7	0.48	93	6.5	0.64	90	

## TABLE A-1 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT ANDEFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Т	hiabendazo	ole	-	Friclocarba	ın	Triclosan				
Date –	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal		
1/28/2010	NA <sup>3</sup>	NA	NA	2.2	0.11	95	4.0	0.14	97		
7/22/2010	0.00	0.00	0.0	0.86	0.07	92	4.7	0.08	98		
1/27/2011	0.02	0.02	10	3.1	0.14	96	4.3	0.09	98		
7/21/2011	0.02	0.02	6.7	4.6	0.11	98	3.3	0.04	99		
1/26/2012	0.01	0.01	18	1.5	0.08	94	2.1	0.04	98		
1/15/2013	0.03	0.02	21	1.7	0.05	97	2.4	0.07	97		
1/14/2014	0.02	0.02	18	0.86	0.04	95	1.6	0.09	94		

<sup>1</sup>All influent values are the mean of two samples collected from the west side and southwest wastewater streams. <sup>2</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value.

<sup>2</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value. <sup>3</sup>No established method.

## TABLE A-2: CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Carbamazepine Influent Effluent Removal			Ciprofloxacin Influent Effluent Removal				Codein		Diphenhydramine Influent Effluent Removal		
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
7/22/2010	0.16	0.17	-7.6 <sup>1</sup>	0.25	0.21	18	0.19	0.07	63	0.23	0.16	30
1/27/2011	0.15	0.16	-6.0	1.4	0.49	64	0.13	0.14	-10	0.48	0.29	39
7/21/2011	0.19	0.20	-3.3	2.8	1.1	60	0.14	0.07	53	0.55	0.26	53
1/26/2012	0.09	0.08	3.3	2.5	0.98	60	0.08	0.07	12	0.21	0.16	23
1/8/2013	0.23	0.23	-2.2	2.0	0.61	69	0.25	0.18	29	0.93	0.35	63
1/7/2014	1.9	1.8	14	0.70	0.64	9.2	0.21	0.23	-8.0	0.58	0.49	15

## TABLE A-2 (continued): CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Fluoxetine				Gemfibro	zil		Ibuprofen	l	Naproxen		
Date	Influent	Effluent	Removal	Influent	Effluent	Removal			Removal	Influent		Removal
1/28/2010	NA <sup>2</sup>	NA	NA	0.80	0.37	54	3.7	0.0	100	5.4	0.21	96
7/22/2010	0.02	0.01	75	0.58	0.02	97	3.5	0.06	98	3.9	0.03	99
1/27/2011	0.02	0.01	29	1.0	0.55	45	9.4	0.19	98	10.4	0.22	98
7/21/2011	0.02	0.01	34	1.0	0.06	94	8.0	0.03	100	8.9	0.14	98
1/26/2012	0.01	0.01	33	0.76	0.46	39	5.1	0.19	96	5.4	0.39	93
1/8/2013	0.05	0.02	63	0.91	0.20	78	11.3	0.17	98	12.1	0.26	98
1/7/2014	0.01	0.02	-47	0.84	0.75	11	7.5	4.2	44	8.6	5.4	38

## TABLE A-2 (continued): CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	]	Thiabendazo	le		Triclocart	oan	Triclosan			
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	
1/28/2010	NA <sup>2</sup>	NA	NA	2.5	0.24	90	6.3	0.31	95	
7/22/2010	0.0	0.0	0.0	0.58	0.11	82	11.0	0.12	99	
1/27/2011	0.02	0.01	13	3.2	0.20	94	18.1	0.15	99	
7/21/2011	0.02	0.01	33	4.8	0.22	95	4.9	0.07	99	
1/26/2012	0.01	0.01	13	2.3	0.30	87	2.1	0.12	95	
1/8/2013	0.02	0.02	18	2.8	0.17	94	4.1	0.11	97	
1/7/2014	0.02	0.01	13	0.42	0.27	37	1.2	0.75	38	

<sup>1</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value. <sup>2</sup>No established method.

## TABLE A-3: CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

	С	Carbamazepine			Ciprofloxa	ncin		Codein	e	Dij	ohenhydrai	mine
Sampling - Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
7/15/2010	0.31	0.28	8.1	1.5	0.11	92	0.20	0.02	90	0.56	0.04	93
1/20/2011	0.20	0.24	-211	2.3	0.48	79	0.17	0.09	51	0.99	0.18	82
7/14/2011	0.33	0.35	-6.3	10.2	0.30	97	0.10	0.01	90	1.5	0.21	86
1/19/2012	0.25	0.22	11	5.2	1.8	65	0.08	0.08	5.4	1.0	0.25	76
1/29/2013	0.30	0.33	-8.1	4.0	1.2	71	0.13	0.17	-35	0.78	0.39	50
1/28/2014	0.23	0.24	-6.5	1.5	0.65	57	0.18	0.15	15	1.0	0.41	61

# TABLE A-3 (continued): CONCENTRATIONS ( $\mu$ g L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling _		Fluoxetine	e		Gemfibroz	il		Ibuprofer	ł		Naproxer	L
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
1/21/2010	NA <sup>2</sup>	NA	NA	1.8	0.87	52	8.2	0.11	99	9.2	0.25	97
7/15/2010	0.04	0.03	35	1.1	0.01	99	5.9	0.02	100	5.2	0.01	100
1/20/2011	0.06	0.03	48	2.3	0.22	90	16.5	0.02 <sup>3</sup>	100	19.2	0.07	100
7/14/2011	0.12	0.03	76	2.2	0.01	100	12.5	0.01	100	16.1	0.03	100
1/19/2012	0.05	0.03	40	1.3	0.23	83	13.5	0.02 <sup>3</sup>	100	11.4	0.06	99
1/29/2013	0.08	0.06	31	1.6	0.67	59	10.6	0.07	99	12.0	0.25	98
1/28/2014	0.07	0.04	34	2.0	1.1	47	12.5	0.07	99	18.1	0.51	97

## TABLE A-3 (continued): CONCENTRATIONS ( $\mu$ g L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	-	Thiabendaz	ole	~	Friclocarba	m	Triclosan				
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal		
1/21/2010	NA <sup>2</sup>	NA	NA	4.8	0.30	94	10.3	0.18	98		
7/15/2010	0.07	0.12	-68	2.5	0.31	87	8.7	0.00	100		
1/20/2011	0.46	0.74	-61	5.1	0.25	95	8.8	0.07	99		
7/14/2011	0.44	0.44	0.04	14.8	0.23	98	19.0	0.01	100		
1/19/2012	0.04	0.04	3.4	3.5	0.27	92	6.5	0.09	99		
1/29/2013	0.08	0.12	-51	2.9	0.25	92	4.8	0.14	97		
1/28/2014	0.04	0.04	18	0.98	0.17	83	3.6	0.14	96		

<sup>1</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value. <sup>2</sup>No established method.

<sup>3</sup>Sample was non-detect but reported using 1/2 level of quantification.

## TABLE A-4: CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Carbamazepine			(	Ciprofloxad	in		Codeine		Diphenhydramine		
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
7/22/2010	0.29	0.29	1.5	0.85	0.81	4.8	0.13	0.05	63	0.49	0.48	2.2
1/26/2011	0.24	0.25	-2.7 <sup>1</sup>	2.7	0.66	76	0.20	0.16	24	1.0	0.35	66
7/21/2011	0.39	0.52	-33	7.6	3.1	59	0.25	0.07	72	1.4	0.31	79
1/26/2012	0.25	0.16	35	3.0	1.4	51	0.08	0.08	-6.1	0.32	0.27	15
1/8/2013	0.41	0.50	-20	9.9	1.9	80	0.64	0.47	26	1.9	0.97	49
1/7/2014	0.39	0.42	-8.8	1.9	0.86	56	0.40	0.39	1.0	1.3	0.64	50

## TABLE A-4 (continued): CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND THEIR REMOVAL EFFICIENCY (%) IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Fluoxetine			Gemfibrozil				Ibuprofer	1	Naproxen		
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
1/27/2010	NA <sup>2</sup>	NA	NA	0.69	0.33	52	5.3	0.26	95	4.3	0.12	97
7/22/2010	0.02	0.02	0.0	0.82	0.17	79	6.5	0.07	99	5.3	0.17	97
1/26/2011	0.06	0.03	50	1.3	0.56	57	19.5	0.24	99	17.8	0.09	99
7/21/2011	0.09	0.03	63	1.3	0.02	99	15.3	0.01	100	15.2	0.19	99
1/26/2012	0.04	0.03	30	0.56	0.38	32	7.8	0.74	91	7.4	0.59	92
1/8/2013	0.08	0.04	45	1.5	1.0	33	22.6	0.89	96	16.2	0.50	97
1/7/2014	0.07	0.05	34	0.12	0.78	-552	24.4	3.6	85	19.9	1.9	91

## TABLE A-4 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Т	hiabendaz	ole		Friclocarba	an	Triclosan				
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal		
1/27/2010	NA <sup>2</sup>	NA	NA	4.6	0.15	97	8.3	0.18	98		
7/22/2010	0.00	0.02	0.0	1.2	0.38	67	9.2	0.14	98		
1/26/2011	0.02	0.03	-24	5.0	0.14	97	7.8	0.09	99		
7/21/2011	0.03	0.04	-62	4.9	0.24	95	9.5	0.06	99		
1/26/2012	0.02	0.01	22	1.5	0.09	94	3.7	0.07	98		
1/8/2013	0.04	0.05	-28	3.4	0.36	90	6.3	0.26	96		
1/7/2014	0.04	0.03	20	1.1	0.23	79	3.3	0.14	96		

 $^{1}$ Variability in monitoring data can result in false values, such as a negative removal efficiency value.  $^{2}$ No established method.

## TABLE A-5: CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE JOHN E. EGAN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling	Carbamazepine			(	Ciprofloxad	ein		Codeine		Diphenhydramine		
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
7/15/2010	0.19	0.24	-26 <sup>1</sup>	1.8	0.05	97	0.09	0.01 <sup>2</sup>	89	0.31	0.03	91
1/20/2011	0.20	0.21	-5.3	2.3	0.56	76	0.15	0.20	-32	1.0	0.21	79
7/14/2011	0.23	0.26	-15	6.3	0.32	95	0.12	0.01	96	1.2	0.08	93
1/19/2012	0.16	0.17	-4.8	5.2	1.8	64	0.10	0.14	-46	0.59	0.27	55
2/26/2013	0.20	0.22	-7.4	3.3	0.98	70	0.20	0.27	-32	0.80	0.35	57
2/25/2014	0.14	0.15	-4.3	2.1	0.62	71	0.15	0.15	1.5	0.47	0.16	66

## TABLE A-5 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE JOHN E. EGAN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling	Fluoxetine			Gemfibrozil				Ibuprofen		Naproxen			
Date -	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	The second s	Influent	Effluent	Removal	
1/21/2010	NA <sup>3</sup>	NA	NA	0.99	0.14	85	6.9	0.02 <sup>2</sup>	100	5.5	0.03	99	
7/15/2010	0.04	0.03	27	0.49	0.01	98	3.7	0.01	100	2.5	0.0	100	
1/20/2011	0.08	0.04	54	1.8	0.24	87	14.0	0.02 <sup>2</sup>	100	14.8	0.03	100	
7/14/2011	0.08	0.05	35	1.7	0.01	100	12.5	0.01	100	13.1	0.00	100	
1/19/2012	0.05	0.03	34	1.2	0.25	79	9.6	0.02 <sup>2</sup>	100	9.9	0.12	99	
2/26/2013	0.06	0.04	38	1.4	0.48	66	12.5	0.02 <sup>2</sup>	100	11.6	0.04	100	
2/25/2014	0.05	0.04	28	1.0	0.38	63	9.9	0.01	100	11.2	0.05	100	

# TABLE A-5 (continued): CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE JOHN E. EGAN WATER **RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH** FEBRUARY 2014

Sampling	Т	`hiabendaz	ole	•	Triclocarba	an		Triclosan	
Date –	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
1/21/2010	NA <sup>3</sup>	NA	NA	4.4	0.07	98	8.5	0.08	99
7/15/2010	0.0	0.02	0.0	3.9	0.08	98	9.1	0.0	100
1/20/2011	0.04	0.03	22	3.8	0.15	96	9.2	0.06	99
7/14/2011	0.04	0.03	19	5.5	0.16	97	8.7	0.02	100
1/19/2012	0.03	0.03	8.0	3.4	0.19	95	7.3	0.18	98
2/26/2013	0.05	0.04	10	1.7	0.10	94	4.7	0.07	99
2/25/2014	0.05	0.03	41	1.7	0.09	94	4.7	0.06	99

<sup>1</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value. <sup>2</sup>Sample was non-detect and reported value is 1/2 level of quantification limit.

<sup>3</sup>No established method.

## TABLE A-6: CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE JAMES C. KIRIE WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling	С	arbamazep	ine	(	Ciprofloxad	cin		Codeine		Dij	ohenhydrai	nine
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
7/15/2010	0.24	0.20	-23 <sup>1</sup>	0.93	0.15 <sup>2</sup>	84	0.13	0.03	81	0.40	0.30	24
1/20/2011	0.27	0.26	-4.0	2.0	0.32	84	0.13	0.06	51	1.0	0.32	69
7/14/2011	0.36	0.29	-24	4.3	0.42	90	0.10	0.03	72	0.98	0.44	55
1/19/2012	0.21	0.19	-12	3.3	1.3	59	0.05	0.04	22	0.41	0.33	18
2/26/2013	0.25	0.25	0.91	2.9	0.55	81	0.13	0.09	27	0.69	0.50	28
2/25/2014	0.21	0.14	34	1.3	0.26	81	0.07	0.05	32	0.37	0.19	48

## TABLE A-6 (continued): CONCENTRATIONS (µg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE JAMES C. KIRIE WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling		Fluoxetin	e		Gemfibroz	zil		Ibuprofer	1		Naproxer	l
Date -	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent		Influent	Effluent	Removal
1/21/2010	NA <sup>3</sup>	NA	NA	1.5	0.28	81	6.6	0.02 <sup>2</sup>	100	5.4	0.04	99
7/15/2010	0.02 <sup>2</sup>	0.02	0.0	0.84	0.07	92	4.7	0.03	99	3.3	0.10	97
1/20/2011	0.05	0.03	32	2.2	0.16	93	16.2	0.02 <sup>2</sup>	100	17.7	0.05	100
7/14/2011	0.04	0.03	33	1.8	0.14	92	12.2	0.03	100	12.4	0.13	99
1/19/2012	0.03	0.02	24	1.1	0.21	80	7.1	0.02 <sup>2</sup>	100	7.4	0.07	99
2/26/2013	0.05	0.04	27	1.5	0.50	65	13.8	0.02 <sup>2</sup>	100	12.6	0.04	100
2/25/2014	0.04	0.02	38	1.0	0.45	56	10.2	0.02 <sup>2</sup>	100	10.1	0.04	100

## TABLE A-6 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE JAMES C. KIRIE WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling	Thiabendazole			,	Triclocarba	m	Triclosan			
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	
1/21/2010	NA <sup>3</sup>	NA	NA	3.8	0.09	98	10.0	0.12	99	
7/15/2010	0.00	0.02	0.0	2.2	0.07	97	13.1	0.05	100	
1/20/2011	0.03	0.03	10	3.6	0.10	97	9.4	0.06	99	
7/14/2011	0.03	0.02	13	2.9	0.18	94	7.5	0.03	100	
1/19/2012	0.03	0.03	0.16	1.8	0.09	95	5.4	0.10	98	
2/26/2013	0.04	0.03	28	1.5	0.09	94	4.0	0.06	98	
2/25/2014	0.02	0.02	37	0.89	0.06	93	3.8	0.04	99	

<sup>T</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value.

<sup>2</sup>Sample was non-detect and reported value is 1/2 level of quantification limit.

<sup>3</sup>No established method.

## TABLE A-7: CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	С	arbamazep	ine	(	Ciprofloxac	cin		Codeine		Dij	ohenhydrai	nine
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
7/15/2010	0.14	0.16	-13 <sup>1</sup>	0.67	0.27	59	0.16	0.03	79	0.33	0.27	18
1/20/2011	0.15	0.15	4.3	1.9	0.52	73	0.09	0.13	-47	0.60	0.44	26
7/14/2011	0.24	0.20	18	3.3	1.1	67	0.10	0.04	65	0.68	0.41	40
1/19/2012	0.15	0.12	18	2.7	1.4	47	0.05	0.07	-55	0.24	0.29	-22
1/29/2013	0.13	0.16	-23	1.9	0.80	57	0.07	0.08	-28	0.25	0.28	-12
1/28/2014	1.1	0.30	73	2.1	.54	74	0.09	0.12	-36	0.48	0.47	2.9

## TABLE A-7 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling		Fluoxetin	e		Gemfibroz	zil		Ibuprofer	1		Naproxer	1
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent		Influent	Effluent	
1/21/2010	NA <sup>2</sup>	NA	NA	1.1	0.43	61	8.1	0.02 <sup>3</sup>	100	5.1	0.08	98
7/15/2010	0.0	0.02	0.0	0.71	0.02	98	5.1	0.02	100	3.4	0.04	99
1/20/2011	0.04	0.02	43	1.5	0.76	49	11.6	0.01	100	10.5	0.10	99
7/14/2011	0.03	0.02	27	1.4	0.07	95	9.4	0.02 <sup>3</sup>	100	9.3	0.07	99
1/19/2012	0.04	0.02	42	0.88	0.35	60	7.0	0.02 <sup>3</sup>	100	6.9	0.06	99
1/29/2013	0.04	0.03	6.0	0.79	0.54	32	6.5	0.15	98	5.8	0.59	90
1/28/2014	0.05	0.03	44	1.2	0.81	33	11.6	0.01	100	10.9	0.23	98

## TABLE A-7 (continued): CONCENTRATIONS (μg L<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN INFLUENT AND EFFLUENT SAMPLES AND REMOVAL EFFICIENCY (%) IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling	Т	hiabendaz	ole	,	Triclocarba	m		Triclosan	
Date	Influent	Effluent	Removal	Influent	Effluent	Removal	Influent	Effluent	Removal
1/21/2010	NA <sup>2</sup>	NA	NA	2.7	0.20	92	6.2	0.25	96
7/15/2010	0.0	0.04	0.0	1.3	0.09	93	4.6	0.07	99
1/20/2011	0.03	0.02	21	2.4	0.36	85	5.5	0.31	94
7/14/2011	0.03	0.03	7.7	2.1	0.10	95	5.0	0.08	98
1/19/2012	0.02	0.02	5.5	1.9	0.24	87	4.1	0.13	97
1/29/2013	0.03	0.02	17	1.2	0.17	86	2.5	0.22	91
1/28/2014	0.03	0.02	10	2.2	0.22	90	4.6	0.11	98

<sup>1</sup>Variability in monitoring data can result in false values, such as a negative removal efficiency value.

<sup>2</sup>No established method.

<sup>3</sup>Sample was non-detect but reported using 1/2 level of quantification.

#### APPENDIX B

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CONCENTRATIONS IN BIOSOLIDS OR WASTE-ACTIVATED SLUDGE SAMPLES: INDIVIDUAL SAMPLING DATES FROM THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

## TABLE B-1: CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
7/22/2010	15.4 <sup>1</sup>	1,221	30.9 <sup>1</sup>	90.6
1/27/2011	28.0	6,624	44.6 <sup>1</sup>	819
7/21/2011	26.0	15,435	3.0	1,060
1/26/2012	16.9	6,889	5.0	251
1/15/2013	60.4	9,537	7.0	998
1/14/2014	40.4	6,396	2.0	773

#### TABLE B-1 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/28/2010	NA <sup>2</sup>	20.0	43.0 <sup>1</sup>	30.0
7/22/2010	15.4 <sup>1</sup>	17.3	214	3.0
1/27/2011	80.6	137	1,678	16.0
7/21/2011	93.9	52.7	849	20.0
1/26/2012	36.9	45.2	50.0	63.6
1/15/2013	121	105	2,251	108
1/14/2014	88.6	105	892	69.6

## TABLE B-1 (continued): CONCENTRATIONS (μg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/28/2010	NA <sup>2</sup>	7,800	5,400
7/22/2010	15.4 <sup>1</sup>	949	426
1/27/2011	21.0	11,847	4,794
7/21/2011	18.0	8,532	4,271
1/26/2012	12.0	6,312	2,568
1/15/2013	33.0	9,087	6,713
1/14/2014	32.0	7,891	4,484

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.

## TABLE B-2: CONCENTRATIONS (μg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
7/22/2010	16.2 <sup>1</sup>	682	32.4 <sup>1</sup>	124
1/27/2011	57.4	8,656	43.7 <sup>1</sup>	891
7/21/2011	63.6	17,739	2.0	1,076
1/26/2012	84.1	15,922	33.8 <sup>1</sup>	821
1/8/2013	64.9	12,745	16.0	990
1/7/2014	34.7	6,428	34.0	375

## TABLE B-2 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/28/2010	NA <sup>2</sup>	40.0	56.2 <sup>1</sup>	11.21
7/22/2010	16.2 <sup>1</sup>	272	1,418	11.0
1/27/2011	89.9	272	1,418	11.0
7/21/2011	97.2	94.3	611	10.0
1/26/2012	80.2	221	1,158	12.0
1/8/2013	111	342	1,376	162
1/7/2014	82.1	63.0	926	547

### TABLE B-2 (continued): CONCENTRATIONS (μg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/28/2010	NA <sup>2</sup>	15,200	13,300
7/22/2010	16.0	22,309	6,200
1/27/2011	12.0	22,309	6,200
7/21/2011	14.0	17,672	8,125
1/26/2012	25.0	15,772	1,984
1/8/2013	$16.2^{1}$	19,837	5,867
1/7/2014	16.0	7,946	5,096

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.

## TABLE B-3: CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
7/15/2010	129	9,147	46.3 <sup>1</sup>	431
1/20/2011	96.9	20,062	17.0	1,005
7/14/2011	145	34,062	6.0	1,211
1/19/2012	123	38,527	7.0	909
1/29/2013	131	42,547	33.0	1,419
1/28/2014	140	24,613	15.0	1,273

#### TABLE B-3 (continued): CONCENTRATIONS (μg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/21/2010	NA <sup>2</sup>	420	780	27.6 <sup>1</sup>
7/15/2010	46.0	202	580	13.3
1/20/2011	240	585	2,199	31.4
7/14/2011	252	406	1,735	16.0
1/19/2012	224	424	1,529	46.2
1/29/2013	434	626	2,854	28.9
1/28/2014	339	467	2,360	28.0

#### TABLE B-3 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/21/2010	NA <sup>2</sup>	32,600	36,300
7/15/2010	666	44,470	39,766
1/20/2011	818	45,752	28,393
7/14/2011	132	36,530	25,400
1/19/2012	158	29,108	29,839
1/29/2013	586	29,651	24,913
1/28/2014	86.3	28,825	28,930

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.

## TABLE B-4: CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
7/22/2010	128 <sup>1</sup>	5,491	256 <sup>1</sup>	369
1/26/2011	29.0	11,476	110	488
7/21/2011	142	19,272	26.0	246
1/26/2012	54.0	22,344	40.0	482
1/8/2013	131	16,795	128	824
1/7/2014	95.3	8,963	139	495

## TABLE B-4 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/27/2011	NA <sup>2</sup>	200	431 <sup>1</sup>	86.2 <sup>1</sup>
7/22/2010	128 <sup>1</sup>	69.7	65.6	53.3
1/26/2011	175	170	179 <sup>1</sup>	37.0
7/21/2011	97.0	238 <sup>1</sup>	405	79.0
1/26/2012	162	132	455 <sup>1</sup>	411
1/8/2013	158	285	851	566
1/7/2014	157	307	613	469

#### TABLE B-4 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/27/2011	NA <sup>2</sup>	5,600	3,600
7/22/2010	128 <sup>1</sup>	6,786	3,053
1/26/2011	34.0	12,344	2,345
7/21/2011	33.0	8,043	1,411
1/26/2012	21.0	7,020	1,864
1/8/2013	89.0	16,509	4,038
1/7/2014	29.0	10,931	2,362

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.

## TABLE B-5: CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE EGAN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
7/15/2010	12.8 <sup>1</sup>	4,086	25.6 <sup>1</sup>	182
1/20/2011	51.0	9,329	44.4 <sup>1</sup>	673
7/14/2011	87.8	23,125	12.0	1,216
1/19/2012	83.4	28,218	3.0	809
2/26/2013	88.9	19,413	41.0	1,122
2/25/2014	95.2	16,807	$16.0^{1}$	741

## TABLE B-5 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE EGAN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/21/2010	NA <sup>2</sup>	120	750	22.0 <sup>1</sup>
7/15/2010	71.0	44.8	170	80.4
1/20/2011	148	248	1,915	24.0
7/14/2011	213	173	1,476	59.3
1/19/2012	228	152	1,495	21.0
2/26/2013	244	206	1,972	8.3
2/25/2014	232	176	1,600	$28.0^{1}$

## TABLE B-5 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN BIOSOLIDS SAMPLES IN THE EGAN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/21/2010	NA <sup>2</sup>	13,800	18,600
7/15/2010	12.8 <sup>1</sup>	731	1,586
1/20/2011	32.0	15,175	10,757
7/14/2011	36.0	26,027	17,786
1/19/2012	40.0	15,892	10,912
2/26/2013	61.0	18,353	12,965
2/25/2014	52.0	14,340	12,064

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.

## TABLE B-6: CONCENTRATIONS (μg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE KIRIE WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
		2 007	154]	265
7/15/2010	$76.9^{1}$	2,997	154 <sup>1</sup>	265
1/20/2011	24.0	6,778	120 <sup>1</sup>	411
7/14/2011	64.0	15,212	143 <sup>1</sup>	730
1/19/2012	39.0	18,070	12.0	366
2/26/2013	45.0	10,848	110	355
2/25/2014	40.0	8,633	16.0	291

## TABLE B-6 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE KIRIE WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/21/2010	NA <sup>2</sup>	280	641 <sup>1</sup>	$22.0^{1}$
7/15/2010	$76.9^{1}$	29.5	126	102
1/20/2011	127	62.3	120 <sup>1</sup>	35.0
7/14/2011	125	33.9	237	104
1/19/2012	108	26.0	$282^{1}$	28.2 <sup>1</sup>
2/26/2013	102	183	220	164
2/25/2014	149	51.5	278 <sup>1</sup>	28.0

## TABLE B-6 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE KIRIE WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH FEBRUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/21/2010	NA <sup>2</sup>	9,100	3,900
7/15/2010	$76.9^{1}$	1,932	2,014
1/20/2011	28.0	9,705	2,743
7/14/2011	19.0	12,263	1,280
1/19/2012	26.0	8,298	2,857
2/26/2013	35.0	9,511	2,127
2/25/2014	21.0	8,535	2,490

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.

## TABLE B-7: CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Carbamazepine	Ciprofloxacin	Codeine	Diphenhydramine
7/15/2010	61.0 <sup>1</sup>	2,960	122 <sup>1</sup>	262
1/20/2011	9.0	16,382	86.2 <sup>1</sup>	236
7/14/2011	83.0	13,729	6.0	472
1/19/2012	33.0	15,871	14.0	310
1/29/2013	46.0	12,950	39.0	467
1/28/2014	178	4,114	10.0	326

### TABLE B-7 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Fluoxetine	Gemfibrozil	Ibuprofen	Naproxen
1/21/2010	NA <sup>2</sup>	240	460	320
7/15/2010	61.0 <sup>1</sup>	112	698	457
1/20/2011	64.0	62.3	558	35.0
7/14/2011	98.0	145	927	790
1/19/2012	78.0	129	395	148
1/29/2013	92.0	244	401	174
1/28/2014	70.9	107	344	391

## TABLE B-7 (continued): CONCENTRATIONS (µg kg<sup>-1</sup>) OF ELEVEN TARGETED PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN WASTE-ACTIVATED SLUDGE SAMPLES IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED FROM JANUARY 2010 THROUGH JANUARY 2014

Sampling Date	Thiabendazole	Triclocarban	Triclosan
1/21/2010	NA <sup>2</sup>	13,100	13,100
7/15/2010	61.0 <sup>1</sup>	621	8,430
1/20/2011	17.0	9,705	2,743
7/14/2011	16.0	8,415	7,713
1/19/2012	17.0	11,137	6,806
1/29/2013	43.0	5,948	3,247
1/28/2014	14.0	4,542	4,184

<sup>1</sup>Sample was non-detect and reported value is 1/2 level of quantification limit. <sup>2</sup>No established method.