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CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT, EFFLUENT, AND BIOSOLIDS AND SLUDGE IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

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SUMMARY

4-Nonylphenol (4-NP) and 4-tert-octylphenol (4-t-OP) are degradation products of alkylphenol ethoxylates, including nonylphenol diethoxylates (NP2EO), nonylphenol monoethoxylates (NP1EO), and octylphenol ethoxylates, which are widely used surfactants in household and industrial cleaners. Bisphenol A (BPA) is a precursor in the manufacturing of polycarbonate plastics and epoxy resins from which a wide variety of other products are generated, such as food and beverage containers. 4-Nonylphenol, NP2EO, NP1EO, 4-t-OP, and BPA have been widely detected in wastewater effluent, which has raised public attention due to potential endocrine disrupting effects of 4-NP, 4-t-OP, and BPA on aquatic life. These compounds are not currently regulated in wastewater effluent or biosolids, and trace concentrations present no known risk to human health. Increased public attention to trace concentrations of these compounds in wastewater effluent and biosolids lead the Metropolitan Water Reclamation District of Greater Chicago (District) to implement a program to monitor influent, effluent, sludge, and biosolids semi-annually (winter and summer) for NP2EO, NP1EO, 4-NP, 4-t-OP, and BPA in the District's seven water reclamation plants (WRPs). This report summarizes monitoring results from February 2003 through July 2006. The results are also compared to values published in the peer-reviewed literature.

Mean influent concentrations for NP2EO were highest at the Calumet and Stickney WRPs, and removal efficiencies for all seven WRPs ranged from 59 to 82 percent. Mean influent concentrations for NP1EO were highest at the Lemont and Stickney WRPs, and removal efficiencies for all seven WRPs ranged from 87 to 96 percent. High removal efficiencies for these compounds are expected due to their degradation to 4-NP and partitioning to sludge during the treatment process. Removal efficiencies were also high for 4-NP, 4-t-OP, and BPA, with 4-NP having the highest removal efficiency of 93 to 98 percent. Influent concentrations for 4-NP and NP1EO were consistent across all four sampling years. Mean influent concentrations for summer 4-NP and NP1EO samples were higher than their respective winter samples; however, there were no seasonal differences for 4-NP and NP1EO, NP2EO, and BPA in the effluent of all seven WRPs were in the parts per trillion (ppt) to parts per billion (ppb) range, and were mostly within range or lower than effluent concentrations reported in the peer-reviewed literature.

Mean biosolids and sludge concentrations for NP2EO and NP1EO were highest at the Terrence J. O'Brien (O'Brien) WRP, but concentrations of these compounds were lower than 4-NP, most likely due to their degradation to 4-NP during the treatment process. The high 4-NP concentrations could be attributed to the degradation of the nonylphenol ethoxylates as well as the direct release of 4-NP from its associated usage and disposal. The highest 4-NP concentration in biosolids and sludge was for the Hanover Park WRP. Mean 4-NP concentrations were consistent across all four sampling years, and there were no seasonal differences between summer and winter samples. Similar to effluent concentrations, mean biosolids and sludge concentrations for NP2EO, NP1EO, 4-NP, 4-t-OP, and BPA from the District's WRPs were mostly comparable or lower than concentrations reported in the peer-reviewed literature. These compounds are expected to degrade to non-toxic end products under aerobic soil conditions. Similarly, studies have demonstrated that 4-NP in biosolids-amended soils is not mobile and will readily degrade to non-toxic end products in days to months.

INTRODUCTION

4-Nonylphenol and 4-t-OP are degradation products of alkylphenol ethoxylates, including NP2EO and NP1EO, which are widely used surfactants in household and industrial cleaners (Table 1). Bisphenol A is a precursor in the manufacturing of polycarbonate plastics and epoxy resins from which a wide variety of other products are generated, such as food and beverage containers (Table 1). Nonylphenol diethoxylate, NP1EO, 4-NP, 4-t-OP, and BPA have been widely detected in wastewater effluent (Hohne and Puttmann, 2008, Stasinakis et al., 2008, Phillips and Chalmers, 2009; Chen et al., 2011; Vajda et al., 2011, Vidal-Dorsch et al., 2012), which has raised public attention due to their potential endocrine disrupting effects in a variety of aquatic species (Soto et al. 1991, Krishnan et al., 1993; Brotons, et al., 1995; Soto et al., 1995). Although NP2EO and NP1EO are considered less toxic than 4-NP, they degrade to 4-NP in the natural environment and during wastewater treatment processes. It has been shown that 4-NP, 4t-OP and BPA preferentially partition to solids and are removed from wastewater during treatment, and as a result have been detected in biosolids and sludge (La Guardia et al., 2001; Gehring et al., 2004; Kinney et al., 2006; Stasinakis et al., 2008; Venkatesan and Halden, 2013). The ring-structure and molecular weight of these three compounds contribute to their low solubility in water as indicated by their relatively high octanol-water partitioning coefficients (Table 1).

Nonylphenol diethoxylate, NP1EO, 4-NP, 4-t-OP, and BPA are not currently regulated in wastewater effluent or biosolids, and trace concentrations of these compounds present no known risk to human health (Touraud et al., 2011); however, increased public attention to trace concentrations of these compounds in wastewater effluent and biosolids lead the District to implelment a program to monitor influent, effluent, sludge, and biosolids from the District's seven WRPs semi-annually (winter and summer) for NP2EO, NP1EO, 4-NP (including ring and chain isomers), 4-t-OP, and BPA. This report summarizes semi-annual monitoring data for these targeted compounds in the District's seven WRPs from February 2003 through July 2006. The results are also compared to values published in the peer-reviewed literature to better understand how concentrations of these compounds in the District's WRPs compare to the other WRPs.

Compound	Major Use in Consumer Products	Octanol-Water Partitioning Coefficient ¹	Solubility in Water at 25°C (mg L ⁻¹) ¹	Molecular Weight ¹
4-Nonylphenol Diethoxylate	Surfactant	5.3 ²	1.05 ²	308.4
4-Nonylphenol Monoethoxylate	Surfactant	NA ³	NA	264.4
4-Nonylphenol	Surfactant, bactericide and fungicide, antioxidant for plastics and rubber, adhesive, heat stabilizer for polymers, and plasticizer	5.76	7.0	220.4
4-tert-Octylphenol	Intermediate in manufacture of surfactants	5.28	5.0	206.3
Bisphenol A	Intermediate in manufacture of surfactants, epoxy, polycarbonate, flame retardants, and rubber chemicals	3.32	120	228.3

TABLE 1: PHYSICAL PROPERTIES AND CONSUMER USES FOR NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A

¹U.S. National Library of Medicine, 2014. ²European Chemicals Agency, 2013. ³NA = Data not available.

METHODS AND MATERIALS

Samples were collected from the influent, effluent, and biosolids and sludge (i.e., digester draw sludge for the John E. Egan (Egan), Hanover Park, Stickney, and Calumet WRPs; waste activated sludge for the O'Brien and Lemont WRPs; return activated sludge for the James C. Kirie (Kirie) WRP) semi-annually (winter and summer) from all seven WRPs from February 2003 through July 2006. Effluent and influent samples were collected in glass gallon bottles for the 24-hour composite samples at each four-hour interval for a total of six collections. Biosolids and sludge samples were one-time grabs in quart bottles. Samples were placed on ice in coolers for transport to the laboratory.

Effluent samples were picked up by the United States Environmental Protection Agency (USEPA) Region 5 Central Laboratory personnel and extracted and analyzed by their laboratory. They analyzed the samples using the Large Volume Injector and Selected Ion Monitoring (SIM) GC/MS. Influent, biosolids and sludge samples were extracted and analyzed by the Organic Compounds Analytical Laboratory (OCAL) located at the Egan WRP. The OCAL extracted the samples using separatory funnels and performed analysis following the USEPA Region 5 Central Laboratory method, using the SIM GC/MS (without Large Volume Injector).

RESULTS AND DISCUSSION

Mean concentrations for NP2EO, NP1EO, 4-NP, 4-t-OP, and BPA in influent and effluent samples for the District's seven WRPs from February 2003 to July 2006 are summarized in <u>Table 2</u>. Removal efficiencies, defined here as the percent difference between influent and effluent concentrations, are summarized in <u>Table 3</u>, and concentrations for individual sampling dates for the duration of the sampling period are available in <u>Appendix A</u>.

Mean influent concentrations for NP2EO from all seven WRPs ranged from nondetectable (<9.5) to 16.9 μ g L⁻¹, and mean effluent concentrations ranged from 1.7 to 5.0 μ g L⁻¹. The highest influent concentrations for NP2EO were at the Calumet and Stickney WRPs, and the highest effluent concentration was at the O'Brien WRP. Removal efficiencies for NP2EO from all seven WRPs ranged from 59 to 82 percent (<u>Table 3</u>). Mean influent concentrations for NP1EO ranged from 21.7 to 47.9 μ g L⁻¹, and mean effluent concentrations ranged from 0.96 to 5.1 μ g L⁻¹ (<u>Table 2</u>). The highest influent and effluent concentrations for NP1EO were at the Lemont and O'Brien WRPs, respectively. High removal efficiencies for NP2EO and NP1EO are expected due to their degradation to 4-NP and partitioning to sludge during the treatment process, and ranged from 59 to 96 percent (<u>Table 3</u>).

Mean influent concentrations for 4-NP, 4-t-OP, and BPA ranged from 24.9 to 61.5, 0.69 to 1.8, and 0.94 to 4.4 μ g L⁻¹, respectively, and mean effluent concentrations ranged from 0.50 to 1.3, 0.10 to 0.13, and 0.12 to 0.22 μ g L⁻¹, respectively (<u>Table 2</u>). Removal efficiencies were also high for these compounds and ranged from 93 to 98 percent for 4-NP (<u>Table 3</u>). Mean concentrations for NP2EO, NP1EO, NP, 4-t-OP, and BPA in the effluent of all seven WRPs were mostly within range or lower than effluent concentrations reported in the peer-reviewed literature (<u>Table 4</u>). Hohne and Puttmann (2008) reported effluent concentrations for 4-NP, 4-t-OP, and BPA from two different treatment plants up to 14.4, 0.39, and 7.6 μ g L⁻¹, respectively. The highest concentrations for 4-NP, 4-t-OP and BPA in the District's WRPs were 1.3, 0.13, and 0.22 μ g L⁻¹, respectively. Mean influent concentrations for summer NP1EO and 4-NP samples across all seven WRPs were significantly higher than their respective winter samples (paired t-test, p<0.05); however, there were no significant differences between mean summer and winter effluent concentrations (data not shown).

Influent and effluent concentrations for all compounds were consistent across all four sampling years; however, 84 percent of the influent and 41 percent of the effluent samples for NP2EO were below detection limits. Mean concentrations for samples below the detection limit were calculated using one-half the limit of quantification. Similarly, 56 percent of 4-t-OP and 52 percent of BPA effluent samples were also below detection limits. Nonylphenol monoethoxylate and 4-NP had the most number of influent and effluent samples above detection limits.

TABLE 2: MEAN¹ CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

	•	lphenol loxylate	Nonyl Monoeth	phenol oxylate	4-Nony	lphenol	4-tert-Oc	tylphenol	Bisphe	enol A
WRP ²	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
					μg L	1				
Stickney ³	15.0	3.2	43.8	2.6	57.7	1.3	1.3	0.13	3.0	0.18
Calumet	16.9	3.2	41.5	1.8	61.5	0.73	1.8	0.11	2.8	0.15
Hanover Park	9.5 ⁴	2.0	21.7	1.6	24.9	0.71	0.69	0.11	0.94	0.16
Lemont	9.5 ⁴	2.4	47.9	1.2	56.5	0.92	0.94	0.11	3.5	0.22
Egan	9.5 ⁴	1.7	35.3	0.96	34.3	0.50	0.80	0.10	4.4	0.12
Kirie	11.9	2.4	32.7	1.3	37.7	0.61	0.75	0.10	2.2	0.12
O'Brien	11.7	5.0	36.6	5.1	34.3	1.2	0.76	0.11	1.8	0.13

¹Mean of eight samples.

²Water Reclamation Plant.

³Mean of 16 samples. Separate samples were taken from the west side and southwest wastewater streams at the Stickney Water Reclamation Plant.

⁴Samples were non-detect but reported as one-half the limit of quantification.

TABLE 3: MEAN¹ REMOVAL EFFICIENCIES FOR NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A FROM THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006.

WRP ²	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
			%%		
Stickney	76	87	93	86	93
Calumet	73	89	97	91	90
Hanover Park	79	87	95	86	69
Lemont	75	93	97	89	81
Egan	82	96	98	90	83
Kirie	80	93	97	87	80
O'Brien	59 ³	89 ³	95	85	78

¹Mean of eight samples. ²Water Reclamation Plant.

³Mean of seven samples.

TABLE 4: RANGE IN CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN EFFLUENT SAMPLES IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006 AND COMPARISONS TO PUBLISHED EFFLUENT CONCENTRATIONS

Study			4-Nonylphenol	4-tert-Octylphenol	Bisphenol A
			μg L ⁻¹		
Present Study	1.7 – 5.0	0.96 - 5.1	0.50 - 1.3	0.10 - 0.13	0.12 - 0.22
Vidal-Dorsch et al., 2012	NA^1	NA	$ND^{2} - 7.20$	0.21 – 1.55	0.21 - 1.60
Vajda et al., 2011	0.44 - 5.0	1.5 – 9.8	0.24 – 2.0	0.03 - 0.20	0.01 - 0.04
Chen et al., 2011	NA	NA	0.46 – 4.9	0.01 - 0.17	0.09 - 0.27
Phillips and Chalmers, 2009	23	NA	14	0.91	NA
Stasinakis et al., 2008	ND – 17.4	ND – 6.89	ND – 0.90	NA	ND – 1.10
Hohne and Puttmann, 2008	NA	NA	ND – 14.4	ND – 0.39	ND - 7.6
1 NA = Data not available.					

 2 ND = Not detected.

Mean biosolids and sludge concentrations are summarized in <u>Table 5</u>. Concentrations in biosolids and sludge samples for individual sampling dates for each WRP for the duration of the sampling period are available in <u>Appendix B</u>. Mean biosolids and sludge concentrations for NP2EO and NP1EO ranged from 12.1 to 25.6 and 9.2 to 29.3 mg kg⁻¹, respectively (<u>Table 5</u>). These values were lower than 4-NP due, in part, to their degradation to 4-NP during the treatment process. Mean concentrations for 4-NP ranged from 9.8 to 861 mg kg⁻¹, and the highest was for the Hanover Park WRP (<u>Table 5</u>). Mean concentrations for 4-t-OP and BPA ranged from 1.1 to 8.2 and 2.3 to 12.0 mg kg⁻¹, respectively.

All of NP2EO and 77 percent of NP1EO biosolids and sludge samples were below detection limits. 4-Nonylphenol had the highest number of biosolids and sludge samples above detection limits. There was no seasonal variation in 4-NP concentrations in biosolids and sludge samples. Mean biosolids and sludge concentrations for NP2EO, NP1EO, 4-NP, 4-t-OP, and BPA from the District's WRPs were mostly within range or lower than concentrations reported in the peer-reviewed literature (<u>Table 6</u>). Kinney et al. (2006) analyzed nine different WRP biosolids products and reported 4-NP concentrations up to 1,520 mg kg⁻¹, nearly twice the concentration (861 mg kg⁻¹) observed for the Hanover Park WRP. Additionally, Xia et al. (2004) reported up to 1,380 mg kg⁻¹ 4-NP in biosolids sampled from 16 WRPs. Gehring et al. (2004) reported BPA concentrations up to 32.1 mg kg⁻¹ from 12 different WRPs, which is over twice the concentration (12.0 mg kg⁻¹) observed for the O'Brien WRP.

Alkylphenol ethoxylates, including NP2EO and NP1EO, and octylphenol ethoxylates degrade to 4-NP and 4-t-OP during wastewater treatment and in natural environments. Although 4-NP and 4-t-OP are considered as more toxic than NP2EO and NP1EO, they ultimately degrade to less toxic end products in aerobic sediments and soils. Similar to removal during wastewater treatment processes, 4-NP, 4-t-OP, and BPA partition into organic matter and sediments when effluent is discharged to rivers and streams. De Weert et al. (2010) demonstrated that 4-NP degrades in 8 to 84 days at the sediment-water interface, depending on the microbial composition in the sediment. Similarly, Cousins et al. (2002) demonstrated that BPA degrades in aerobic soils and water in 4.5 days. Degradation of 4-NP, 4-t-OP, and BPA in a laboratory study occurred in aerobic soil within seven days; however, the same compounds were more persistent under anaerobic conditions. Similarly, degradation in sediments for 4-NP and BPA was significantly reduced under anaerobic conditions (Ike et al., 2006; Ying, 2006).

Brown et al. (2009) investigated the degradation and fate of 4-NP in biosolids applied at agronomic rates to agriculture soils in a greenhouse study and reported that less than 15 percent of the initial concentration of 4-NP remained in the presence of winter wheat, movement through the top few cm of soil was negligible, and concentrations were not detected in wheat leaves at 45 days. Xia et al. (2010) reported that 4-NP remained in the top 30 cm of an agricultural soil receiving biosolids application for 33 years, and that concentrations were 30 to 50 times lower than cumulative loading estimates due, in part, to degradation over time. Gomez-Rico et al. (2008) reported that 84 percent of 4-NP degraded within nine months in biosolids-amended forest soils. The land application of biosolids would not be expected to contribute to the presence of 4-NP in surface water or groundwater due to its immobility in soil, or to accumulate in soil due to biodegradation in aerobic environments.

TABLE 5: MEAN¹ CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS AND SLUDGE SAMPLES² IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

WRP ³	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
		mg kg	g ⁻¹ (dry weight)		
Stickney	12.1	12.0	337	3.9	5.6
Calumet	17.5	9.2	469	5.5	2.3
Hanover Park	16.2	23.5	861	8.2	2.7
Lemont	20.8	12.1	12.2	1.1	3.4
Egan	19.1	10.0	452	5.8	3.5
Kirie	20.1	10.6	9.8	1.1	2.7
O'Brien	25.6	29.3	58.8	1.6	12.0

¹Mean of eight samples. ²Samples from Lemont and O'Brien are waste-activated sludge, and from Kirie are return sludge.

³Water Reclamation Plant.

TABLE 6: RANGE IN CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS AND SLUDGE SAMPLES IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006 AND COMPARISONS TO PUBLISHED BIOSOLIDS CONCENTRATIONS

Study	Nonylphenols Diethoxylates			4-tert-Octylphenol	Bisphenol A
Present Study	12.1 – 25	10 – 29.3	9.8 – 861	1.1 - 8.2	2.3 - 12.0
Venkatesan and Halden, 2013	32.8 – 153	34.3 – 103	405 – 861	ND^1	NA ²
Stasinakis et al., 2008	ND – 24.7	1.01 – 45.3	ND – 0.45	NA	ND – 1.75
Kinney et al., 2006	0.79 – 89.0	4.0 – 79.4	2.2 – 1,520	0.90 - 5.4	1.7 – 14.4
Gehring et al., 2004	NA – 25.6	NA	1.2 – 493	0.06 - 10.4	0.20 - 32.1
La Guardia et al., 2001	ND – 32.6	25.7 – 102	683 – 887	6.7 – 12.6	NA

 1 ND = Not detected.

 2 NA = Data not available.

The monitoring data show that NP2EO, NP1EO, 4-NP, 4-t-OP, and BPA are present in District WRP's influent and effluent at trace concentrations in the parts per trillion (ppt) to parts per billion (ppb) range. Concentrations of these compounds in the District's effluent were in the range of or lower than concentrations reported in the peer-reviewed literature. Due to the hydrophobic nature of these compounds, removal efficiencies are high across all seven of the District's WRPs, resulting in the partitioning of these compounds to sludge and biosolids. Concentrations of these compounds detected in biosolids range from parts per billion (ppb) to parts per million (ppm). Research has shown that these compounds are not mobile in soils when biosolids are land-applied at agronomic rates as a fertilizer. Therefore, these compounds are not expected to leach from soils to surface water when biosolids are land applied, and would not impact aquatic organisms. Additionally, these compounds were shown to readily biodegrade in aerobic environments, and would not be expected to accumulate over time in biosolids-amended soils.

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APPENDIX A

NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES: INDIVIDUAL SAMPLING DATES FROM THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

TABLE A-1: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE JOHN E. EGAN WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling	•	phenol oxylate	•	lphenol thoxylate	4-Non	ylphenol		ert- ohenol	Bisph	enol A
Date	Influent	Effluent	Influent			Effluent	Influent	Effluent	Influent	Effluent
	$\mu g L^{-1}$									
Feb 2003	9.5 ¹	1.1	38	0.55	47	0.32	0.50 ¹	0.06^{1}	8.0	0.06^{1}
July 2003	9.5 ¹	1.6	30	0.63 ¹	33	0.31 ¹	0.50^{1}	0.02	5.0	0.05
Feb 2004	9.5 ¹	1.2	79	0.63	50	0.28	0.50^{1}	0.01^{1}	9.0	0.17
Aug 2004	9.5 ¹	6.0^{1}	71	4.0^{1}	68	2.0^{1}	2.0	0.50^{1}	0.50^{1}	0.50^{1}
Feb 2005	9.5 ¹	1.0^{1}	5.0 ¹	0.50^{1}	11	0.36	0.50^{1}	0.05^{1}	11	0.05^{1}
Aug 2005	9.5 ¹	1.0^{1}	5.0^{1}	0.41	3.5 ¹	0.25 ¹	1.4	0.05^{1}	0.50^{1}	0.05^{1}
Feb 2006	9.5 ¹	1.0^{1}	34	0.50^{1}	26	0.25^{1}	0.50 ¹	0.05^{1}	0.50^{1}	0.05^{1}
July 2006	9.5 ¹	1.0^{1}	20	0.50^{1}	36	0.25^{1}	0.50^{1}	0.05 ¹	0.50^{1}	0.05^{1}

TABLE A-2: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling	Nonylı Dietho:		Nonylp Monoeth						Bisphenol A		
Date	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	
					μg I	μg L ⁻¹					
Feb 2003	9.5 ¹	7.2	28	6.7	39	1.3	1.0	0.06^{1}	0.50^{1}	0.11	
July 2003	9.5 ¹	1.7	42	3.2	40	0.32	0.50^{1}	0.06 ¹	0.50^{1}	0.09	
Feb 2004	9.5 ¹	9.1	42	6.6	25	1.8	0.50^{1}	0.07	7.0	0.01 ¹	
Aug 2004	9.5 ¹	6.0 ¹	49	4.0^{1}	62	2.0^{1}	1.0	0.50^{1}	0.50^{1}	0.50^{1}	
Feb 2005	9.5 ¹	12	5.0 ¹	15	14	2.5	0.50^{1}	0.06^{1}	0.50^{1}	0.06 ¹	
Aug 2005	27	1.2	83	1.1	58	0.25^{1}	1.6	0.05^{1}	0.50^{1}	0.05^{1}	
Feb 2006	9.5 ¹	1.7	20	3.1	18	1.1	0.50^{1}	0.05^{1}	0.50^{1}	0.05^{1}	
July 2006	9.5 ¹	1.0^{1}	23	0.50^{1}	18	0.25^{1}	0.50^{1}	0.05 ¹	4.0	0.2	

TABLE A-3: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE JAMES C. KIRIE WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling	•	vlphenol oxylate		ylphenol ethoxylate	4-Non	ylphenol		ert- phenol	Bispl	henol A
Date	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
Feb 2003	9.5 ¹	2.5	5.0 ¹	1.1	65	0.33	0.50^{1}	0.06^{1}	1.0	0.06^{1}
July 2003	9.5 ¹	1.9	25	1.1	25	0.31 ¹	0.50^{1}	0.03	9.0	0.09
Feb 2004	9.5 ¹	2.9	42	1.4	35	0.68	1.0	0.04	5.0	0.05
Aug 2004	29	6.0 ¹	57	4.0^{1}	53	2.0^{1}	1.0	0.50^{1}	0.50^{1}	0.50^{1}
Feb 2005	9.5 ¹	1.0^{1}	5.0 ¹	0.50^{1}	3.5 ¹	0.37	0.50^{1}	0.05^{1}	0.50^{1}	0.05^{1}
Aug 2005	9.5 ¹	1.0^{1}	38	0.50^{1}	33	0.25^{1}	0.50^{1}	0.05^{1}	0.50^{1}	0.11
Feb 2006	9.5 ¹	1.0^{1}	60	0.50^{1}	50	0.25 ¹	1.0	0.05^{1}	0.50^{1}	0.05^{1}
July 2006	9.5 ¹	3.1	29	1.5	37	0.68	1.0	0.05^{1}	0.50^{1}	0.05^{1}

TABLE A-4: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling		vlphenol noxylate	Nonylpl Monoetho		4-Nony	lphenol	4-te Octylt	ert- ohenol	Bisp	henol A
Date	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
					μg L ⁻	1				
Feb 2003	9.5 ¹	2.6	26	2.5	36	0.34	0.50^{1}	0.06^{1}	0.50^{1}	0.06^{1}
July 2003	9.5 ¹	1.2	17	1.4	30	0.31 ¹	0.50^{1}	0.01	0.50^{1}	0.11
Feb 2004	9.5 ¹	1.5	5.0 ¹	1.5	3.5 ¹	0.39	0.50^{1}	0.01^{1}	0.50^{1}	0.08
Aug 2004	9.5 ¹	6.0 ¹	46	4.0^{1}	46	2.0^{1}	1.0	0.50^{1}	0.50^{1}	0.50^{1}
Feb 2005	9.5 ¹	2.0^{1}	5.0 ¹	1.7	11	0.50^{1}	0.50^{1}	0.10 ¹	0.50^{1}	0.10^{1}
Aug 2005	9.5 ¹	1.1	51	0.70	50	0.25 ¹	1.5	0.05^{1}	0.50^{1}	0.18
Feb 2006	9.5 ¹	1.0^{1}	26	0.50^{1}	21	1.7	0.50^{1}	0.06	0.50^{1}	0.05^{1}
July 2006	9.5 ¹	1.0^{1}	5.0 ¹	0.69	3.5 ¹	0.25^{1}	0.50^{1}	0.05^{1}	0.50^{1}	0.22

TABLE A-5: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE CALUMET WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling	•	lphenol oxylate	Nonylp Monoeth		4-Nony	phenol		tert- lphenol	Bis	phenol A
Date	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
					μg L ⁻¹					
Feb 2003	35	3.7	57	2.0	129	0.50	1.0	0.06^{1}	3.0	0.06^{1}
July 2003	20	2.3	35	1.6	53	0.31 ¹	0.50^{1}	0.01	4.0	0.05
Feb 2004	9.5 ¹	4.4	11	2.3	10	0.99	0.50^{1}	0.09	0.50^{1}	0.21
Aug 2004	9.5 ¹	6.0^{1}	65	4.0^{1}	113	2.0^{1}	5.0	0.50^{1}	4.0	0.50^{1}
Feb 2005	9.5 ¹	5.3	5.0^{1}	2.1	11	1.1	0.50^{1}	0.10 ¹	2.0	0.10^{1}
Aug 2005	9.5 ¹	1.0^{1}	58	0.84	87	0.26	3.1	0.05	0.50^{1}	0.05^{1}
Feb 2006	9.5 ¹	1.5	19	0.90	19	0.33	0.50^{1}	0.05^{1}	4.0	0.11
July 2006	33	1.0^{1}	82	0.78	70	0.37	3.0	0.04	4.0	0.16

TABLE A-6: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE LEMONT WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling	Dietho	phenol oxylate	Monoe	vlphenol ethoxylate		ylphenol	Octyl	ert- phenol	Bisphe	
Date	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
					μg L ⁻	1				
Feb 2003	9.5 ¹	2.2	45	0.60	77	0.37	0.50^{1}	0.06^{1}	1.0	0.06 ¹
July 2003	9.5 ¹	1.3 ¹	20	0.63 ¹	28	0.49	0.50^{1}	0.02	3.0	0.06 ¹
Feb 2004	9.5 ¹	2.3	5.0^{1}	1.7	16	1.6	0.50^{1}	0.07	5.0	0.27
Aug 2004	9.5 ¹	6.0 ¹	42	4.0^{1}	90	2.0^{1}	2.0	0.50^{1}	2.0	0.50^{1}
Feb 2005	9.5 ¹	4.0	19	1.9	15	1.8	0.50^{1}	0.12	13.0	0.29
Aug 2005	9.5 ¹	1.0 ¹	74	0.46	85	0.25 ¹	1.5	0.05^{1}	0.50^{1}	0.37
Feb 2006	9.5 ¹	1.0^{1}	112	0.50^{1}	68	0.55	1.0	0.05	3.0	0.05^{1}
July 2006	9.5 ¹	1.0 ¹	66	0.05	73	0.39	1.0	0.04	0.50^{1}	0.17

TABLE A-7: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN INFLUENT AND EFFLUENT SAMPLES IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

			onylphen iethoxyla			onylphen noethoxy		4-1	Nonylpher	nol	(4-tert- Octylphene	ol	В	sisphenol	A
	Sampling Date	S. West Influent	West Influent	Effluent	S. West Influent	West Influent	Effluent	S. West Influent	West Influent	Effluent	S. West Influent	West Influent	Effluent	S. West Influent	West	
-									μg L ⁻¹							
	Feb 2003	34	9.5 ¹	4.0	42	22	2.7	79	44	1.2	0.50^{1}	0.50^{1}	0.06 ¹	1.0	9.0	0.06 ¹
	July 2003	24	9.5 ¹	1.1	71	60	0.63 ¹	76	78	0.31 ¹	1.0	2.0	0.06^{1}	2.0	4.0	0.17
A-7	Feb 2004	9.5 ¹	9.5 ¹	4.1	5.0 ¹	11	5.1	8.0	3.5 ¹	2.2	0.50^{1}	0.50 ¹	0.09	0.50^{1}	1.0	0.07
	Aug 2004	9.5 ¹	9.5 ¹	6.0^{1}	28	63	4.0^{1}	58	88	2.0^{1}	2.0	0.50 ¹	0.50^{1}	7.0	0.50^{1}	0.50 ¹
	Feb 2005	25	26	7.2	46	32	5.5	32	27	3.3	0.50^{1}	0.50 ¹	0.15	10	0.50^{1}	0.31
	Aug 2005	9.5 ¹	26	1.0^{1}	60	97	0.58	49	51	0.25 ¹	1.2	1.8	0.05^{1}	6.6	0.50^{1}	0.21
	Feb 2006	9.5 ¹	9.5 ¹	1.5	27	30	1.6	16	98	0.72	0.50^{1}	2.0	0.05^{1}	0.50^{1}	1.0	0.05 ¹
	July 2006	9.5 ¹	9.5 ¹	1.0 ¹	49	58	0.78	92	124	0.29	3.0	3.0	0.05^{1}	0.50^{1}	3.0	0.10

APPENDIX B

NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS AND SLUDGE SAMPLES: INDIVIDUAL SAMPLING DATES FROM THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

TABLE B-1: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS SAMPLES IN THE JOHN E. EGAN WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
			mg kg ⁻¹ (dry weight) -		
Feb 2003	19 ¹	10^{1}	552	5.6	3.5
July 2003	14^{1}	7.5^{1}	575	6.0	2.9
Feb 2004	17 ¹	9 .1 ¹	335	6.2	5.8
Aug 2004	30 ¹	16 ¹	595	7.6	6.3
Feb 2005	20^{1}	11^{1}	416	5.5	4.9
Aug 2005	211	11^{1}	405	4.5	1.1^{1}
Jan 2006	18 ¹	9.6 ¹	500	7.4	0.96 ¹
July 2006	12 ¹	6.5 ¹	242	3.7	2.4

TABLE B-2: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN WASTE ACTIVATED SLUDGE SAMPLES IN THE TERRENCE J. O'BRIEN WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

mg kg ⁻¹ (dry weight) Feb 2003 29^1 60 87 1.5^1 1.5^1 July 2003 63^1 33^1 81 3.3^1 81 Feb 2004 68^1 36^1 25^1 3.6^1 11 Aug 2004 7.5^1 18 86 1.3 0.40^1 Feb 2005 7.9^1 4.2^1 24 0.42^1 0.42^1 Aug 2005 7.8^1 21 85 1.4 0.41^1	Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
July 2003 63^1 33^1 81 3.3^1 81 Feb 2004 68^1 36^1 25^1 3.6^1 11 Aug 2004 7.5^1 18 86 1.3 0.40^1 Feb 2005 7.9^1 4.2^1 24 0.42^1 0.42^1 Aug 2005 7.8^1 21 85 1.4 0.41^1				mg kg ⁻¹ (dry weight)		
Feb 2004 68 ¹ 36 ¹ 25 ¹ 3.6 ¹ 11 Aug 2004 7.5 ¹ 18 86 1.3 0.40 ¹ Feb 2005 7.9 ¹ 4.2 ¹ 24 0.42 ¹ 0.42 ¹ Aug 2005 7.8 ¹ 21 85 1.4 0.41 ¹	Feb 2003	29^{1}	60	87	1.5 ¹	1.5^{1}
Aug 20047.5118861.30.401Feb 20057.914.21240.4210.421Aug 20057.8121851.40.411	July 2003	63 ¹	33 ¹	81	3.3 ¹	81
Feb 2005 7.9 ¹ 4.2 ¹ 24 0.42 ¹ 0.42 ¹ Aug 2005 7.8 ¹ 21 85 1.4 0.41 ¹	Feb 2004	68 ¹	36 ¹	25^{1}	3.6 ¹	11
Aug 2005 7.8 ¹ 21 85 1.4 0.41 ¹	Aug 2004	7.5 ¹	18	86	1.3	0.40^{1}
	Feb 2005	7.9^{1}	4.2^{1}	24	0.42^{1}	0.42^{1}
	Aug 2005	7.8^{1}	21	85	1.4	0.411
Jan 2006 11 30 44 0.56 0.56	Jan 2006	11^{1}	30	44	0.56^{1}	0.56^{1}
July 2006 12^1 34 38 0.63^1 0.63^1	July 2006	12 ¹	34	38	0.63 ¹	0.63 ¹

TABLE B-3: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN RETURN SLUDGE SAMPLES IN THE JAMES C. KIRIE WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
		n	ng kg ⁻¹ (dry weight)		
Feb 2003	32 ¹	17^{1}	12^{1}	1.7^{1}	1.7^{1}
July 2003	18^{1}	9.4 ¹	6.6 ¹	0.94 ¹	3.9
Feb 2004	29^{1}	15 ¹	11^{1}	1.5 ¹	5.0
Aug 2004	16 ¹	8.3 ¹	5.8 ¹	0.831	5.2
Feb 2005	15 ¹	8.1^{1}	5.6 ¹	0.811	0.81^{1}
Aug 2005	20^1	10 ¹	7.3 ¹	1.0^{1}	2.1
Jan 2006	16 ¹	8.2^{1}	25	0.82^{1}	0.82^{1}
July 2006	16 ¹	8.2^{1}	5.7 ¹	0.82^{1}	1.8

TABLE B-4: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS SAMPLES IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
			mg kg ⁻¹ (dry weight)		
Feb 2003	22 ¹	12 ¹	984	4.7	4.8
July 2003	22 ¹	12 ¹	739	4.6	2.8
Feb 2004	26^{1}	14^{1}	480	5.7	6.5
Aug 2004	16 ¹	44	853	8.6	2.8
Feb 2005	5.2 ¹	2.7^{1}	716	8.3	0.27^{1}
Aug 2005	6.0^{1}	33	1,031	8.9	0.311
Jan 2006	10^{1}	46	956	9.8	0.55^{1}
July 2006	22 ¹	26	1,131	15	4.0

TABLE B-5: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS SAMPLES IN THE CALUMET WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
			mg kg ⁻¹ (dry weight)		
Feb 2003	21^{1}	11^{1}	696	4.3	2.8
July 2003	11^1	5.9 ¹	500	3.5	1.3
Feb 2004	22^{1}	12 ¹	403	5.2	1.2^{1}
Aug 2004	17^{1}	8.8^{1}	422	5.6	2.5
Feb 2005	19 ¹	9.9 ¹	510	6.9	2.3
Aug 2005	19 ¹	10^{1}	487	5.2	2.7
Jan 2006	18 ¹	9.6 ¹	452	6.5	2.7
July 2006	13 ¹	7.0^{1}	280	6.6	3.2

TABLE B-6: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN WASTE ACTIVATED SLUDGE SAMPLES IN THE LEMONT WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
			mg kg ⁻¹ (dry weight)		
Feb 2003	26 ¹	14^{1}	9.5^{1}	1.4 ¹	4.1
July 2003	25^{1}	13 ¹	9.2 ¹	1.3 ¹	5.9
Feb 2004	33 ¹	18 ¹	12 ¹	1.8 ¹	5.6
Aug 2004	13 ¹	16	28	0.69 ¹	7.8
Feb 2005	21^{1}	11^{1}	20	1.1^{1}	1.1^{1}
Aug 2005	12 ¹	6.1 ¹	4.3 ¹	0.61 ¹	0.611
Jan 2006	21^{1}	11^{1}	7.6 ¹	1.1^{1}	1.1^{1}
July 2006	17^{1}	8.8^{1}	6.1 ¹	0.88^{1}	0.88^{1}

TABLE B-7: CONCENTRATIONS OF NONYLPHENOL DIETHOXYLATE, NONYLPHENOL MONOETHOXYLATE, 4-NONYLPHENOL, 4-TERT-OCTYLPHENOL, AND BISPHENOL A IN BIOSOLIDS SAMPLES IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED SEMI-ANNUALLY FROM FEBRUARY 2003 THROUGH JULY 2006

Sampling Date	Nonylphenol Diethoxylate	Nonylphenol Monoethoxylate	4-Nonylphenol	4-tert- Octylphenol	Bisphenol A
-			mg kg ⁻¹ (dry weight)		
Feb 2003	17 ¹	9.1 ¹	522	4.3	2.1
July 2003	11^{1}	37	131	1.5	0.57^{1}
Feb 2004	15 ¹	7.6 ¹	309	5.2	2.2
Aug 2004	10^{1}	12	422	5.3	1.6
Feb 2005	11^{1}	5.5 ¹	283	3.7	1.2
Aug 2005	11^1	5.8 ¹	314	2.9	15
Jan 2006	12 ¹	6.4 ¹	382	4.3	6.4
July 2006	10 ¹	12	333	4.2	16