Metropolitan Water Reclamation District of Greater Chicago

MONITORING AND RESEARCH DEPARTMENT

REPORT NO. 09-60

LEVELS OF TRICLOCARBAN AND TRICLOSAN IN THE INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE FROM THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS

SEPTEMBER 2009

Metropolitan Water Reclamation District of Greater Chicago
100 East Erie Street Chicago, IL 60611-2803 (312) 751-5600

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By

Lakhwinder S. Hundal
Soil Scientist II

Kuldip Kumar
Soil Scientist I

Anna Liao
Instrumentation Chemist IV

Albert E. Cox
Soil Scientist III

Thomas C. Granato
Assistant Director of Monitoring and Research
Environmental Monitoring and Research Division

Monitoring and Research Department
Louis Kollias, Director

September 2009

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ACKNOWLEDGMENTS

The authors thank Dr. Rolf Halden and his research group at the Johns Hopkins University for providing analyses of triclocarban and triclosan. The authors acknowledge the staff of the District's Maintenance and Operations Department for their assistance in sample collection from the seven water reclamation plants. The authors thank Richard Lanyon, Executive Director of the District, Louis Kollias, Director of Monitoring and Research, and Mary Khalil, former Assistant Director of Analytical Laboratory Division (retired) for their support in establishing the TCC and TCS monitoring study. Special thanks are extended to Kathleen Quinlan for formatting the report.

DISCLAIMER

The mention of trade names of specific products does not constitute endorsement of them by the Metropolitan Water Reclamation District of Greater Chicago.

SUMMARY

Triclocarban (TCC) and triclosan (TCS) are commonly used antimicrobial additives in many household and personal care products. There is no documented evidence that TCC or TCS in personal care products provide any benefit to consumers. However, a majority of the personal care products sold in the United States still contain these antimicrobial additives. The antimicrobials are being released continuously into the wastewater stream via routine domestic activities and reach the wastewater treatment plants (WWTPs). During the wastewater treatment process a majority of these chemicals (>97%) is removed from the liquid phase through partitioning into biosolids. Concerns have been raised that TCC and TCS may enter the terrestrial environment via land application of biosolids. Trace levels of TCC and TCS have been reported in the WWTP effluents and detectable levels have been found in the effluent-dominated streams across the United States. Both TCC and TCS can be toxic to aquatic organisms and are likely to persist in the environment.

Generally, TCC and TCS are not routinely monitored by WWTPs because they are not regulated by the United States Environmental Protection Agency (USEPA). However, due to the increased concerns expressed by many researchers and the public, the Metropolitan Water Reclamation District of Greater Chicago (District) initiated a program in August 2005 for semi-annual monitoring of TCC and TCS in the influents, effluents, and biosolids samples from its seven water reclamation plants (WRPs).

The mean concentrations of TCC in influents, effluents, and biosolids collected from the District's WRPs ranged from 2.2 to 7.0 µg/L, 0.09 to 0.30 µg/L, and 14.0 to 38.0 mg/kg, respectively. The mean concentrations of TCS in influent, effluent, and biosolids samples ranged from 4.1 to 7.6 µg/L, 0.03 to 0.14 µg/L, and 4.0 to 28.5 mg/kg, respectively. Overall, the mean effluent concentrations of TCC were slightly greater than TCS but there were no noteworthy trends in the concentrations of TCC and TCS over time during the sampling period. The levels of TCC and TCS found in the District's influents, effluents, and biosolids were considerably lower than the levels reported in the peer-reviewed literature. Overall, the District's WRPs show high removal of both TCC and TCS from the liquid phase.

INTRODUCTION

Triclocarban (TCC; 3-[4-chlorophenyl]-1-{3,4-dichlorophenyl}urea) and triclosan (TCS; 5-chloro-2-{2,4-dichlorophenoxy}phenol) are high production volume (HPV) chemicals and are frequently used as antimicrobial additives in many daily use household and personal care products such as soaps, detergents, deodorants, toothpaste, cosmetics, etc. (TCC Consortium, 2002; Bair-Anderson et al. 2008). There is no documented evidence that the use of soaps containing TCS or TCC provide any health benefit or protection from infectious diseases to the consumers (Tan et al., 2002; Aiello et al., 2007). Yet, a majority of the personal care products sold in the U.S. still contain these antimicrobial additives. These chemicals are continuously released into the wastewater stream via routine domestic activities. Recent research demonstrated that upon reaching the WWTPs, approximately 97% of TCC and 98% of TCS are removed from the aqueous stream through partitioning into solids and microbially-mediated transformation processes (Heidler et al., 2006; Heidler and Halden 2007). Since a large fraction of the TCC and TCS removed can be recovered in biosolids, concerns have been raised that land application of biosolids may be a significant route through which TCC and TCS may enter the terrestrial environment. However, more attention has been focused on the trace levels of TCC and TCS detected in the WWTP effluents because detectable levels of these chemicals have been reported in the effluent-receiving streams across the United States (Kolpin et al., 2002).

Both TCC and TCS have come under scrutiny by the USEPA and the United States Food and Drug Administration due to environmental concerns. While there is evidence suggesting that TCC and TCS can be toxic to aquatic organisms, concerns regarding bioaccumulation and antimicrobial resistance arise due to their likely persistence in the environment. Both TCC and TCS have been shown to bioaccumulate in algae and snails exposed to WWTP effluents, though TCC generally exhibited greater bioaccumulation than TCS or methyl-triclosan, a biodegradation product of TCS (Coogan et al., 2007; Coogan, and La Point, 2008). Some researchers have postulated that the toxicity of these compounds may increase as they biodegrade in the environment (Halden and Paull, 2005). However, to our knowledge no definitive evidence exists that confirms this hypothesis.

TCC and TCS are not regulated by the USEPA and thus are not included in the routine National Pollution Discharge Elimination System (NPDES) monitoring program. But due to the increased media attention and concerns expressed by leading researchers, the District fostered collaboration with Dr. Rolf Halden of John Hopkins University (currently at the Arizona State University) in 2005 and initiated semi-annual monitoring of TCC and TCS in the influents, effluents, and waste-activated sludge or biosolids samples from its seven WRPs. This report summarizes the findings of this research.

METHODS AND MATERIALS

Sample Collection

Influent, effluent, and waste-activated sludge (Lemont, Kirie and North Side WRPs) or anaerobically digested biosolids (Stickney, Calumet, Egan and Hanover Park WRPs) samples were collected twice a year from the District's seven WRPs starting in August 2005 through January 2009. Composite samples of influent and effluent (3,600 mL) were collected over 24-h time period by collecting a 600 mL sample every 4-h time interval. A single grab sample of waste-activated sludge or biosolids was taken at the end of the 24-h composite time. At the Stickney WRP, influent samples from the southwest and west side wastewater streams were collected separately. The samples were shipped on ice to Johns Hopkins University where the samples were fortified with isotope-labeled standards (500 ng/L of $^{13}C_6$-TCC and $^{13}C_6$-TCS for influent and waste-activated sludge or biosolids samples; 100 ng/L of $^{13}C_6$-TCC and $^{13}C_6$-TCS for effluent samples), and stored at – 20°C prior to analysis.

Sample Preparation

Influent and effluent samples were centrifuged at 2000g for 20 min to remove any particulate matter. The supernatant was passed through a solid-phase extraction (SPE) cartridge and eluted with organic solvents (5 mL, 1:1 methanol/acetone). Waste-activated sludge and biosolids samples (10 mL) were dried at room temperature by using forced air and then extracted three times with 5 mL of 1:1 methanol/acetone mixture. The extracts were dried at room temperature by using forced air, reconstituted in high performance liquid chromatography (HPLC) grade 1:1 methanol/acetone, and then centrifuged at 2000g for 20 min to remove any remaining particulate matter as described by Halden and Paull, 2004. Aliquots of influent and sludge samples were diluted as needed with HPLC grade water containing 10 mM acetic acid and then fortified with internal standards.

Analytical Methods

Specific details of the analytical technique used are given elsewhere (Halden and Paull, 2005). Briefly, TCC and TCS were analyzed using a liquid chromatography electrospray ionization mass spectrometry (LC/ESI/MS) technique. Chromatography was carried out on a Waters 2795 LC unit equipped with an autosampler. Compounds were eluted using a gradient method (70% acetonitrile, 30% water) and detected using a quadrupole mass spectrometer (Quattro MicroMass triple quadrupole) in negative electron spray ionization (ESI) mode. The mass spectrometer was operated in a selective ion monitoring (SIM) mode and quantitation was performed using a linear calibration curve using seven calibration levels. TCC-d_7 and $^{13}C_{12}$-TCS (20 ng/L) were used as internal standards. Additional details of quality assurance and quality control protocols followed are given elsewhere (Halden and Paull, 2004; Halden et al., 2001).

RESULTS AND DISCUSSION

The mean concentrations of TCC and TCS in influent, effluent, and waste-activated sludge or biosolids samples collected from the District's seven WRPs during August 2005 through January 2009 are summarized in Table 1. These concentrations for individual sampling dates for each WRP for the duration of the sampling period are presented in Tables 2 – 8 and percent solids data are given in Table A1-1. Overall, the mean effluent concentrations of TCC were slightly greater than TCS (Table 1). There were no note-worthy trends in the concentrations of TCC and TCS over time during the sampling period (Tables 2 – 8). The highest mean concentrations of TCC in the influent and effluent samples were observed in the Lemont WRP, whereas the highest mean concentration of TCC in waste-activated sludge or biosolids was observed in the Hanover Park WRP samples. The highest mean concentrations of TCS in the influent and effluent samples were observed in the Hanover Park and Egan WRPs, and the North Side WRP, respectively, whereas the highest mean TCS concentration in waste-activated sludge or biosolids was observed in the Hanover Park WRP.

Concentrations of TCC in WWTPs influent and effluent presented in the peer-reviewed literature were found to range from 0.4 to 50 µg/L and 0.1 to 6 µg/L, respectively (Bendz et al., 2005; Halden and Paull, 2005; Lishman et al., 2006). The mean concentrations of TCC in the District's WRPs influent and effluent ranged from 2.2 to 7.0 µg/L and 0.09 to 0.30 µg/L, respectively (Table 1). The concentrations of TCC in WWTPs waste-activated sludge and biosolids (dry weight basis) reported in literature were found to range from 2.2 to 4.8 mg/kg and 3.1 to 51.0 mg/kg, respectively (Heidler et al., 2006; Kinney et al, 2006; Chu and Metcalfe, 2007). However, the mean concentrations of TCC in waste-activated sludge or biosolids produced by the District's WRPs ranged from 14.0 to 38.0 mg/kg (Table 1).

Concentrations of TCS in WWTPs influent and effluent reported in peer-reviewed literature were found to range from 1.86 to 26.8 µg/L and 0.027 to 2.7 µg/L, respectively (McAvoy et al., 2002; Bester, 2003; Kanda et al., 2003; Sabaliunas et al., 2003; Bendz et al., 2005; Halden and Paull, 2005; Thompson et al., 2005; Lishman et al., 2006; Waltman et al., 2006; Heidler and Halden, 2007). The mean concentrations of TCS in the District's WRPs influent and effluent ranged from 4.1 to 7.6 µg/L and 0.03 to 0.14 µg/L, respectively (Table 1). The concentrations of TCS in WWTPs waste-activated sludge and biosolids (dry weight basis) reported in peer-reviewed literature ranged from 0.58 to 14.7 mg/kg and 0.09 to 32.9 mg/kg, respectively (McAvoy et al., 2002; Singer et al., 2002; Bester, 2003; Morales et al., 2005; Kinney et al, 2006; Chu and Metcalfe, 2007; Ying and Kookana, 2007). The mean concentrations of TCS in waste-activated sludge or biosolids produced by the District's WRPs ranged from 4.0 to 28.5 mg/kg (Table 1). Clearly, the levels of TCC and TCS found in the influent, effluent, and waste-activated sludge or biosolids of the District's WRPs are closer to the low end of the concentration ranges reported in literature.

Overall, the District's WRPs showed high removal of both TCC and TCS from the liquid phase. The average liquid phase removal rates for TCC and TCS at the District's seven WRPs ranged from 93.2% to 97.8% and 96.6% to 99.6%, respectively (Figure 1). The levels of TCC and TCS in waste-activated sludge or biosolids suggest that removal of TCC and TCS from the

influent occurs largely through partitioning into solids during the treatment process (Table 1). Our data show that compared to TCC, TCS concentrations tended to be higher in the influent, but lower in the effluent, and waste-activated sludge or biosolids (Table 1). Percent removal rates for TCS were consistently higher than TCC at all WRPs (Figure 1). These relationships suggest that the contribution of microbial degradation to removal of these compounds from the liquid phase is probably greater for TCS than for TCC.

TABLE 1: MEAN[1] CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE OR BIOSOLIDS SAMPLES IN THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
WRP[2]	Influent	Effluent	Biosolids[3]	Influent	Effluent	Biosolids[3]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
Stickney	4.1[4]	0.09	14.0	4.8[4]	0.09	8.8
Calumet	3.3	0.19	18.1	6.2	0.11	4.8
North Side	2.2	0.15	15.6	4.1	0.14	10.3
Hanover Park	5.1	0.21	38.0	7.6	0.08	28.5
Lemont	7.0	0.30	20.2	6.2	0.12	4.0
Kirie	2.5	0.11	19.3	5.7	0.08	4.5
Egan	4.6	0.10	24.6	7.6	0.03	21.2

[1] Mean of 8 samples.
[2] Water reclamation plant.
[3] Dry weight basis, Lemont, Kirie, and North Side are waste-activated sludge samples.
[4] Mean of 16 samples. Separate samples were taken from the west side and southwest wastewater streams.

TABLE 2: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND BIOSOLIDS SAMPLES IN THE STICKNEY WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent[1]	Effluent	Biosolids[2]	Influent[1]	Effluent	Biosolids[2]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/24/2005	3.0	0.09	30.7	5.9	0.08	10.9
2/02/2006	4.0	0.18	10.9	3.7	0.08	12.6
7/20/2006	3.6	0.12	16.9	3.4	0.14	10.0
1/24/2007	3.3	0.01	7.5	7.9	0.11	7.9
7/18/2007	4.4	0.11	11.6	3.5	0.03	7.9
1/24/2008	6.5	0.11	8.5	7.0	0.11	4.6
7/24/2008	3.6	0.09	11.9	3.9	0.09	6.1
1/21/2009	4.9	0.04	14.0	3.4	0.11	10.7

[1]Mean of 2 samples collected from the west side and southwest wastewater streams.
[2]Dry weight basis.

TABLE 3: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND BIOSOLIDS SAMPLES IN THE CALUMET WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent	Effluent	Biosolids[1]	Influent	Effluent	Biosolids[1]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/24/2005	1.5	0.30	35.7	8.0	0.17	3.9
2/02/2006	3.1	0.17	18.5	5.2	0.09	5.9
7/20/2006	3.0	0.12	14.5	4.3	0.04	5.1
1/24/2007	3.5	0.16	9.7	8.8	0.08	1.6
7/18/2007	2.8	0.26	14.6	3.2	0.12	0.5
1/24/2008	2.5	0.29	12.0	10.8	0.21	4.0
7/24/2008	3.7	0.07	19.1	6.2	0.08	14.1
1/21/2009	6.2	0.11	20.8	2.8	0.08	2.9

[1]Dry weight basis.

TABLE 4: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE SAMPLES IN THE NORTH SIDE WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent	Effluent	Sludge[1]	Influent	Effluent	Sludge[1]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/16/2005	3.6	0.20	24.4	4.7	0.31	9.4
1/26/2006	2.1	0.23	18.5	3.1	0.09	11.5
7/13/2006	2.1	0.16	16.3	3.3	0.09	11.3
1/17/2007	2.3	0.07	10.2	4.4	0.05	5.9
7/11/2007	1.5	0.21	13.2	2.1	0.02	9.5
1/24/2008	1.9	0.19	12.0	5.3	0.10	9.5
7/24/2008	1.5	0.05	15.2	4.7	0.05	12.9
1/21/2009	2.6	0.11	15.2	4.8	0.40	12.3

[1]Dry weight basis.

TABLE 5: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND BIOSOLIDS SAMPLES IN THE HANOVER PARK WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent	Effluent	Biosolids[1]	Influent	Effluent	Biosolids[1]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/16/2005	6.4	0.17	33.3	8.3	0.01	28.8
1/26/2006	5.4	0.30	42.4	4.1	0.07	29.9
7/13/2006	7.4	0.20	37.4	12.4	<0.01	26.4
1/17/2007	4.4	0.23	25.9	5.3	0.06	22.2
7/11/2007	4.1	0.17	27.2	5.8	<0.01	39.9
1/24/2008	4.3	0.21	21.4	8.4	0.09	13.2
7/24/2008	4.0	0.16	68.9	6.4	<0.01	55.5
1/21/2009	5.1	0.21	47.2	9.8	0.38	11.8

[1]Dry weight basis.

TABLE 6: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE SAMPLES IN THE LEMONT WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent	Effluent	Sludge[1]	Influent	Effluent	Sludge[1]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/24/2005	3.9	0.28	27.0	8.6	0.23	1.4
2/01/2006	15.2	0.07	7.1	11.3	0.04	2.8
7/20/2006	5.1	0.48	24.9	5.9	0.05	7.7
1/24/2007	10.4	0.51	13.0	6.0	0.11	2.9
7/18/2007	7.5	0.14	7.2	7.1	0.11	2.3
1/30/2008	2.9	0.11	3.9	5.9	0.11	2.1
7/31/2008	2.5	0.36	28.9	3.7	0.06	6.8
1/28/2009	8.1	0.41	49.5	1.1	0.21	5.7

[1]Dry weight basis.

TABLE 7: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND WASTE-ACTIVATED SLUDGE SAMPLES IN THE KIRIE WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent	Effluent	Sludge[1]	Influent	Effluent	Sludge[1]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/16/2005	2.9	0.05	19.2	6.5	<0.01	5.4
1/26/2006	2.5	0.18	26.8	2.3	0.08	5.9
7/13/2006	1.9	0.10	15.2	6.7	<0.01	5.4
1/17/2007	1.3	0.06	25.7	4.8	0.04	3.9
7/11/2007	1.6	0.10	14.1	3.7	0.02	1.7
1/24/2008	3.0	0.17	12.4	5.8	0.21	3.8
7/24/2008	4.1	0.13	15.4	8.5	0.04	4.7
1/21/2009	2.9	0.11	25.5	6.9	0.27	4.9

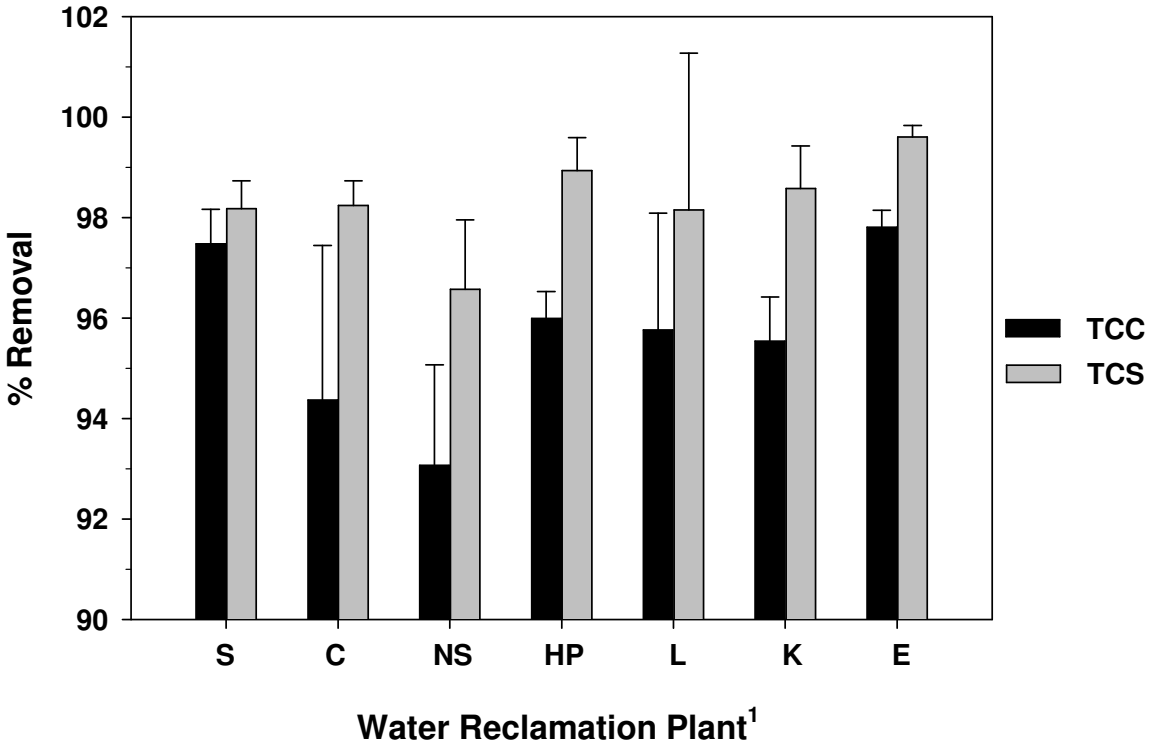
[1]Dry weight basis.

TABLE 8: CONCENTRATIONS OF TRICLOCARBAN AND TRICLOSAN IN INFLUENT, EFFLUENT, AND BIOSOLIDS SAMPLES IN THE EGAN WATER RECLAMATION PLANT COLLECTED FROM AUGUST 2005 THROUGH JANUARY 2009

	Triclocarban			Triclosan		
Sampling Date	Influent	Effluent	Biosolids[1]	Influent	Effluent	Biosolids[1]
	µg/L	µg/L	mg/kg	µg/L	µg/L	mg/kg
8/16/2005	6.7	0.16	17.4	11.0	<0.01	16.0
1/26/2006	4.5	0.14	45.9	3.4	<0.01	25.0
7/13/2006	4.0	0.11	20.5	5.8	<0.01	16.1
1/17/2007	4.3	0.07	22.2	8.9	0.02	18.9
7/11/2007	4.6	0.07	18.0	5.8	0.01	33.6
1/24/2008	4.9	0.13	15.4	10.3	0.12	10.6
7/24/2008	2.6	0.06	25.6	7.5	<0.01	23.7
1/21/2009	5.0	0.06	31.9	8.4	0.08	25.9

[1]Dry weight basis.

FIGURE 1: MEAN PERCENT REMOVAL RATES OF TRICLOCARBAN AND TRICLOSAN FROM THE LIQUID PHASE DURING WASTEWATER TREATMENT AT THE DISTRICT'S SEVEN WATER RECLAMATION PLANTS[1]



[1]S = Stickney, C = Calumet, NS = North Side, HP = Hanover Park, L = Lemont, K = Kirie, E = Egan.
TCC = Triclocarban.
TCS = Triclosan.

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APPENDIX - AI

TABLE A1-1: TOTAL SOLIDS CONTENT IN WASTE-ACTIVATED SLUDGE AND BIOSOLIDS SAMPLES COLLECTED FROM THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO'S SEVEN WATER RECLAMATION PLANTS FROM AUGUST 2005 THROUGH JANUARY 2009

Sampling Date	Stickney	Calumet	North Side	Hanover Park	Lemont	Kirie	Egan
8/16/2005	NA	NA	0.7	1.6	NA	1.6	1.8
8/24/2005	3.0	2.1	NA	NA	0.8	NA	NA
1/26/2006	NA	NA	0.8	1.5	NA	0.6	2.2
2/02/2006	2.9	2.0	NA	NA	1.0[1]	NA	NA
7/13/2006	NA	NA	0.7	1.5	NA	0.5	2.2
7/20/2006	3.7	3.1	NA	NA	0.4	NA	NA
1/17/2007	NA	NA	0.7	2.5	NA	0.7	2.0
1/24/2007	2.7	1.8	NA	NA	0.5	NA	NA
7/11/2007	NA	NA	0.5	1.0	NA	0.4	1.5
7/18/2007	4.4	2.0	NA	NA	1.0	NA	NA
1/24/2008	NA	NA	0.9	1.1	NA	0.7	1.7
1/31/2008	2.4	1.7	NA	NA	0.8	NA	NA
7/24/2008	NA	NA	0.7	0.4	4	0.7	2.7
7/31/2008	3.6	1.7	NA	NA	0.4	NA	NA
1/21/2009	NA	NA	0.9	1.9	NA	0.8	3.6
1/28/2009	2.7	1.4	NA	NA	0.6	NA	NA

[1]Samples collected on 2/01/2006.
NA = Not applicable.