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2010 Occurrence of Pharmaceutical and Personal Care Products (PPCPs) in Source Water of the New York City Water Supply

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Cannonsville Reservoir

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ABSTRACT

In response to the issue of emerging contaminants in drinking water, including those classified as pharmaceuticals and personal care products (PPCPs), in 2009, the New York City Department of Environmental Protection (DEP) conducted a one-year occurrence study to document the presence or absence of a target group of PPCPs in the source waters of the New York City Water Supply. This report summarizes the results of a follow up study that was conducted in 2010, with water samples again collected quarterly from the Catskill, Delaware, and Croton untreated source waters. In addition, a new site was added to sample for PPCP occurrence in water that had been treated with chlorine for primary disinfection. The purpose of adding this site to the sampling program was to determine whether the presence of a strong oxidant such as chlorine degraded or changed the mixture of the minute amount of PPCPs present in samples from the Delaware Aqueduct.

As in 2009, samples were again analyzed by two contract laboratories using newly developed and highly sensitive analytical methods to look for a target group of up to 72 analytes. Two industrial chemicals, bisphenol A (BPA) and perfluorooctane sulfonate (PFOS), were included in this target group of analytes. Due to the extremely low detection levels required for this study, DEP implemented strict quality control requirements for field sampling and laboratory analysis. The results indicate that some PPCPs are present in very low concentrations (low parts per trillion range) in the source waters of New York City, with a greater frequency of detection in the Croton source water as compared to the Catskill/Delaware source waters. A total of 14 individual PPCP compounds were detected in at least one sampling event; and while ten of these compounds were detected in the previous year, four compounds were new detections found in the Croton source water. In addition, six of the 16 compounds detected in 2009 were not detected in 2010, three of which were hormones. The PPCP compounds detected most frequently in 2010 included: butalbital, sulfamethoxazole, cotinine, caffeine, carbamazepine, and DEET. PFOS, an industrial chemical, was also detected frequently. The measured concentrations of the detected compounds were all well below the New York State generic standards for individual unspecified organic contaminants (UOCs) or principal organic contaminants (POCs) of 50,000 nanograms per liter (ng/L) and 5,000 ng/L, respectively. In addition, results of samples collected after chlorination indicated that primary disinfection had little effect on degradation of target compounds detected in this study. A screening level risk assessment conducted by DEP suggests that the concentrations of the detected PPCPs are well below levels that would pose a risk to the health of consumers of New York City's drinking water.

Although there are currently no State or federal mandatory testing or reporting requirements for PPCPs, the United States Environmental Protection Agency (*EPA) recently published a draft list of compounds that will need to be monitored by water utilities under the Unregulated Contaminant Monitoring Rule 3 (UCMR3). Several of the compounds included in UCMR3 are considered PPCPs including five analytes targeted by DEP as part of this occurrence study. DEP has been proactive in this effort, and will continue to assess the presence of PPCPs through EPA's Unregulated Contaminant Monitoring Rule (UCMR 3) monitoring requirements, starting in 2013.*

Background

The last decade has seen increased documentation of trace concentrations (low parts-per-trillion) levels of pharmaceuticals and personal care products (PPCPs) in surface water, groundwater, and finished drinking water. While PPCPs can originate from numerous sources, effluents from wastewater treatment plants (WWTPs) have been identified as a significant source to surface waters. PPCPs can enter WWTPs when people excrete pharmaceutical products or their metabolites, or flush unused medications down a drain or sewer system. The pharmaceutical drugs that have been detected nationally comprise a large range of emerging drinking water contaminants, including prescription and over-the-counter drugs, antibiotics, tranquilizers, antidepressants, and other organic chemicals. The personal care products that have been detected include but are not limited to: fragrances, disinfectants, sunscreen, preservatives, and surfactants or their metabolites (Kolpin et al., 2002).

In 2009, the New York City Department of Environmental Protection (DEP) proactively began a PPCP occurrence study that focused on 72 constituents selected based on results reported in national and regional occurrence studies carried out by the United States Geological Survey (USGS) (Kolpin et al., 2002; Phillips et al., 2010), and the NYSDOH (Wilson et al., 2006). Additional compounds associated with the contracted laboratories' analytical methods were also included. The sampling was conducted on a quarterly basis at three locations representative of the three untreated source waters of the New York City Water Supply System. A total of 16 PPCPs were detected over the course of the sampling period. The most commonly detected PPCPs were butalbital, sulfamethoxazole, carbamazepine, caffeine, cotinine, diazepam, gemfibrozil, and cis-testosterone. Perfluorooctane sulfonate (PFOS) an industrial chemical was also detected frequently. There was a greater frequency of detection in the Croton Water Supply's source water, compared to the Catskill/Delaware's source water. Although PPCPs are not yet regulated under the Safe Drinking Water Act, New York State has a generic standard for organic constituents that would include the PPCPs detected in 2009. The concentration of these compounds at all locations were well below the New York State generic standards for individual unspecified organic contaminants (UOCs) or principal organic contaminants (POCs)[i] of 50,000 nanograms per liter (ng/L) (10 NYCRR Part 5 – Public Water Systems) and 5,000 ng/L, respectively.

A screening level risk assessment conducted by DEP suggested that the concentrations of the PPCPs detected in 2009 were well below levels that would pose a risk to the health of consumers of NYC's drinking water. These results were consistent with USGS and NYSDOH regional findings; as well as national findings summarized by the Water Research Foundation (WateRF)[ii], "screening level risk assessments conducted to date have not indicated that the trace concentrations of pharmaceuticals detected in drinking water pose a risk to consumers" (Snyder et al., 2008; Wilson et al., 2006; Kolpin et al., 2002).

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[i] POCs are generally halogenated alkanes, ethers, benzenes, and some other classes of compounds as defined in the State Sanitary Code http://www.health.state.ny.us/regulations/nycrr/title_10/part_5/subpart_5-1_tables.htm. UOCs refer to any organic chemical not otherwise specified in the State Sanitary Code. **Tables 3 and 4** of this report list the applicable NYS standards for the compounds analyzed.

[ii] Formerly known as American Water Works Association Research Foundation

[iii] CROGH, the primary site for Croton raw water effluent from the New Croton Reservoir, could not be used as the source water keypoint for the Croton System because the Croton System was offline during the study. Instead, the

Purpose

The purpose of the 2010 PPCP study was to provide additional data on the occurrence of and temporal changes in PPCP concentrations in the untreated source waters of the New York City Water Supply, as well as to assess the impact of chlorination on the mixture of PPCPs at one location. Analyses were performed by the same two contract laboratories used in the 2009 study - Montgomery Watson (MWH) and its subcontractor Underwriters Laboratory (UL).

Site Selection and Sampling

Sampling Locations: The 2010 PPCP Quarterly Monitoring program began in March 2010, with the final quarter of sampling occurring in December 2010. Drinking water supply samples were collected in duplicate from DEP's three source water keypoints: CATLEFF, DEL18, and CRO1T.[iii] A fourth set of samples were collected from DEL19 after the water had been chlorinated (**Table 1**). Trip and field blanks were also collected for Quality Control (QC) purposes. A description of QC sampling can be found in subsequent sections.

Table 1: Sites for PPCP Monitoring Program

Site Code	Site Description	Reason for Site Selection
CATLEFF	Catskill Aqueduct, lower effluent chamber, untreated effluent from Kensico Reservoir	Raw source water keypoint sampling location. Locations are prior to the addition of chlorine used for primary disinfection.
DEL18	Delaware Aqueduct, Shaft 18 untreated effluent from Kensico Reservoir	
CRO1T	Croton Gatehouse 1, untreated effluent from New Croton Reservoir	
DEL19	Delaware Aqueduct, Shaft 19 chlorinated effluent from Kensico Reservoir	Sampling site post primary chlorination

Quality Control (QC) Samples: In addition to the sample and sample duplicates collected for each quarterly sampling event, the following QC samples were collected:

- Sample Duplicate. For each analytical method used, a sample and sample duplicate were collected by filling one bottle immediately after the other. The purpose of the sample duplicate is to test the overall precision of the analytical and sampling methods. Four samples and four duplicates were collected for each round of sampling.
- Field Blank. A field blank was collected with each round of sampling. The purpose of the field blank is to test for potential cross-contamination in sample handling and with sampling equipment. Field blanks were generated by pouring analyte-free water supplied by the contract laboratories into sample bottles in the field, utilizing the same equipment and processes as used to collect the water samples.
- Trip blank. A trip blank was collected with each round of sampling. The purpose of the trip blank is to test for potential cross-contamination during the transport and storage of

[iii] CROGH, the primary site for Croton raw water effluent from the New Croton Reservoir, could not be used as the source water keypoint for the Croton System because the Croton System was offline during the study. Instead, the sample was collected at Gate House 1 where there is a sample tap — the CRO1T site.

the water samples. Trip blanks were prepared by the contract laboratories using analyte-free water in the appropriate sample containers with the proper preservative. Trip blanks are taken out into the field and then returned to the contract lab for analysis without being opened.

- Laboratory Fortified Matrix (LFM) and Laboratory Fortified Matrix Duplicate (LFMD). These are water samples collected by DEP at one of the three sampling sites in a manner identical to the procedures used to collect the samples. The LFM and LFMD samples are used by the laboratories to spike known concentrations of target analytes or surrogate compounds. The LFM sample helps to assess analytical preparation and analysis bias using a raw water sample rather than distilled or laboratory water. Similarly, the LFMD is used to test analytical bias and precision with an actual raw water sample rather than with laboratory water.

As summarized in **Table 2** below**,** a total of 12 samples (including QC samples) were collected for each quarterly round of sampling. Alternating locations were selected each quarter from which to draw or collect samples for QC purposes. In one quarter, the QC samples were collected from CRO1T, the next quarter from CATLEFF, and so on.

Table 2: Samples Collected

	Sample	Sample Dup	Trip Blank	Field Blank	LFM	LFMD
Site 1	X	X	X[1]	X[1]	X[1]	X[1]
Site 2	X	X				
Site 3	X	X				
Site 4	X	X				

[1] At alternating locations at each sampling event.

Field Methods

The desired detection levels of the pilot program are generally in the ng/L (parts-per- trillion) range. The *"clean hands"* method (USEPA, 1996), was deemed successful in 2009 sampling so was repeated in 2010. The method sufficiently reduced exposures from airborne dust, dirt, and lint, as well as transference from human contact with the samples (e.g., dandruff, skin oils, sweat). Upon arrival at the sampling site, one member of the two-person sampling team was designated as *"dirty hands";* the second member was designated as *"clean hands."* All operations involving contact with the sample bottle and with transfer of the sample from the sample collection device to the sample bottle were handled by the individual designated as *"clean hands." "Dirty hands"* was responsible for all activities that did not involve direct contact with the sample. This is depicted in **Figure 1** below.

Figure 1: *"Dirty hands"* (right) assisting *"clean hands"* (left) with sample collection

Description of Analytical Methods

After collection, samples were sent to the contract laboratories MWH and UL for analysis and were analyzed by each laboratory using proprietary High Pressure Liquid Chromatography and Dual Mass Spectroscopy (LC/MS/MS) methods. The difference between the methods and calibrations by each lab resulted in a variation in the range, sensitivity, and performance of the methods when searching for a wide range of target analytes with varying physical-chemical properties. Each laboratory established minimum reporting levels (MRLs) for their LC/MS/MS methods. The MRL is the minimum level (concentration) that the laboratory can report accurately. Anything below the MRL is considered to be not detected. In several instances, the laboratories had different MRLs for the same target compounds.

Compounds Analyzed: Based upon the 2009 results, in 2010, DEP relied principally upon two methods for PPCP analyses: MWH Method EDC2SR and UL Method 220. MWH Method EDC2SCR (a peer-reviewed isotope dilution based solid phase extraction (SPE) LC-MS-MS method using a sensitive API4000 instrument) was used in all four quarters to analyze and quantify 21 compounds including most of the compounds detected frequently in the 2009 study. UL Method 220 was used in three of four quarters to cover a broader range of 44 PPCPs than provided by MWH including several of the PPCPs detected infrequently in 2009 such as lasalocid. In the first quarter of 2010, DEP used UL Method 221 instead of UL220, in part to see if any new compounds were picked up including at the chlorinated site. Gemfibrozil which was also detected by the MWH method, was the only PPCP detected with UL220, and as this PPCP was also detected using MWH Method EDC2SR20, the UL221 was dropped in subsequent quarters and replaced by UL220. Overall, the target analytes list included 38 individual compounds in the first quarter and 56 compounds in the remaining three quarters.

Tables 3 and 4 provide the general classes of compounds and analytes for which tests were performed utilizing the three different laboratory methods, and the MRL for each analyte. There was some overlap between the various methods; therefore some analytes were measured using more than one method (e.g. sulfamethoxazole).

Table 3: Selected PPCP Analysis Methods (MWH)

MWH Method	Compounds Analyzed (21 total) **	MRL ng/L
EDC2SCR	Acetaminophen	1
	Atenolol	5
	Bisphenol A (BPA)	10
	Butalbital	5
	Caffeine	3
	Carbamazepine	5
	Cotinine	1
	Diazepam	1
	Estrone	1
	Estradiol	1
	Ethinyl Estradiol - 17 alpha	5
	Fluoxetine	5
	Gemfibrozil	1
	Ibuprofen	1
	Iopromide	10
	Perfluorooctane Sulfonate - PFOS	0.2
	Progesterone	1
	Sulfamethoxazole	1
	Testosterone	1
	Triclosan	5
	Trimethoprim	1

** Some overlap of compounds analyzed by both labs

Table 4: Selected PPCP Analysis Methods (UL)

UL221 Method	Compounds Analyzed (20 total) **	MRL ng/L
UL221	Bezafibrate	0.5
	Chloramphenicol	5
	Chlorotetracycline	50
	Clofibric Acid	0.5
	Diclofenac	0.5
	Dilantin	2
	Doxycycline	50
	Gemfibrozil	0.5
	Ibuprofen	50
	Levothyroxine (Synthroid)	2
	Naproxen	2
	Oxytetracycline	500
	Penicillin G	2
	Penicillin V	2
	Prednisone	2
	Salicylic acid	50
	Tetracycline	500
	Theophylline	5
	Triclocarban	5
	Triclosan	50

** Some overlap of compounds analyzed by both labs

UL220 Method	Compounds Analyzed (44 total) **	MRL ng/L
UL220	Acetaminophen	5
	Antipyrine	1
	Atenolol	1
	Azithromycin	5
	Bacitracin	1000
	Caffeine	50
	Carbadox	5
	Carbamazepine	1
	Ciprofloxacin	50
	Cotinine	1
	DEET	5
	Dexamethasone	5
	Diazepam	1
	Diltiazem	0.1
	Enrofloxacin	50
	Erythromycin	1
	Fluoxetine	1
	Iopromide	50
	Lasalocid	1
	Lincomycin	0.1

UL220 Method	Compounds Analyzed (44 total) **	MRL ng/L
	Meprobamate	1
	Monensin	1
	Narasin	1
	Nicotine	5
	Norfloxacin	50
	Oleandomycin	1
	Paraxanthine	5
	Primidone	5
	Roxithromycin	1
	Salinomycin	0.1
	Simvastatin	5
	Sulfachloropyridazine	5
	Sulfadiazine	1
	Sulfadimethoxine	0.1
	Sulfamerazine	1
	Sulfamethazine	1
	Sulfamethizole	1
	Sulfamethoxazole	1
	Sulfasalazine	5
	Sulfathiazole	1
	Theobromine	50
	Trimethoprim	1
	Tylosin	1
	Virginiamycin M1	1

** Some overlap of compounds analyzed by both labs

Quality Control Issues

Quality Controls: The laboratories analyzed Quality Controls with every batch of samples. The results document the accuracy and precision at the time of the actual testing and to show that any compounds present in the samples, came only from the water being tested, and not from some other sources, such as trace contamination from sampling and analysis procedures.

Sample Duplicates: Sample duplicates were collected and processed in the same batches as the corresponding samples. The analytical results for sample duplicates were compared using the relative percent difference (RPD) between the sample duplicate and the sample. A relative percent difference of 20 percent or less for MWH, and 30 percent or less for UL, between the sample and its field duplicate was used as an indication of good overall precision. As in 2009, there was generally good agreement between a sample and its duplicate. One exception was acetaminophen which was detected at 68 ng/L at DEL19 in a sample, but at only 5 ng/L in the duplicate sample.

Trip/Field Blanks: One set of trip blanks and field blanks was collected for each analytical method at alternating sampling locations every sampling event. During the second quarter, the UL field blank collected at DEL18 was positive for atenolol, cotinine, and nicotine. During the third quarter sampling at CRO1T, the MWH field blank was positive for triclosan and the UL

field blank was positive for sulfadimethoxine. During the fourth quarter sampling at DEL19, the UL field blank was positive for norfloxacin and ciprofloxacin. The only other analyte detected in a field blank was cotinine which was present in the second quarter and analyzed by UL; also in the second quarter, cotinine was detected in a sample which was analyzed by MWH. Because these samples were analyzed by two different labs, in all likelihood the detections are unrelated. In addition, the fourth quarter detections of the norfloxacin and ciprofloxacin in the UL field blank for DEL19 were also detected in similar concentrations for the UL trip blank indicating some possible background interference or contamination of these two compounds. However, neither compound was found in the site samples. A total of seven compounds were detected in blanks, but only one of these detections (cotinine, second quarter) overlapped with the 14 reported sample detections. These few detections in the blanks were not unusual given the high sensitivity of the method, and none of the sample results were invalidated due to the limited blank contamination.

Results and Discussion

A sample and a sample duplicate were collected for each analytical method at all four sampling sites (CATLEFF, DEL18, CRO1T, and DEL19) each quarter. A total of 32 samples were collected for the 2010 sampling period not including QC samples. This resulted in the verified detection of 14 different compounds in the water supply samples. A list of detected compounds from 2009 and 2010 with their general use can be found in **Table 5**. An additional seven compounds were detected in blanks but only one of these detections (cotinine, second quarter) overlapped with the 14 reported sample detections.

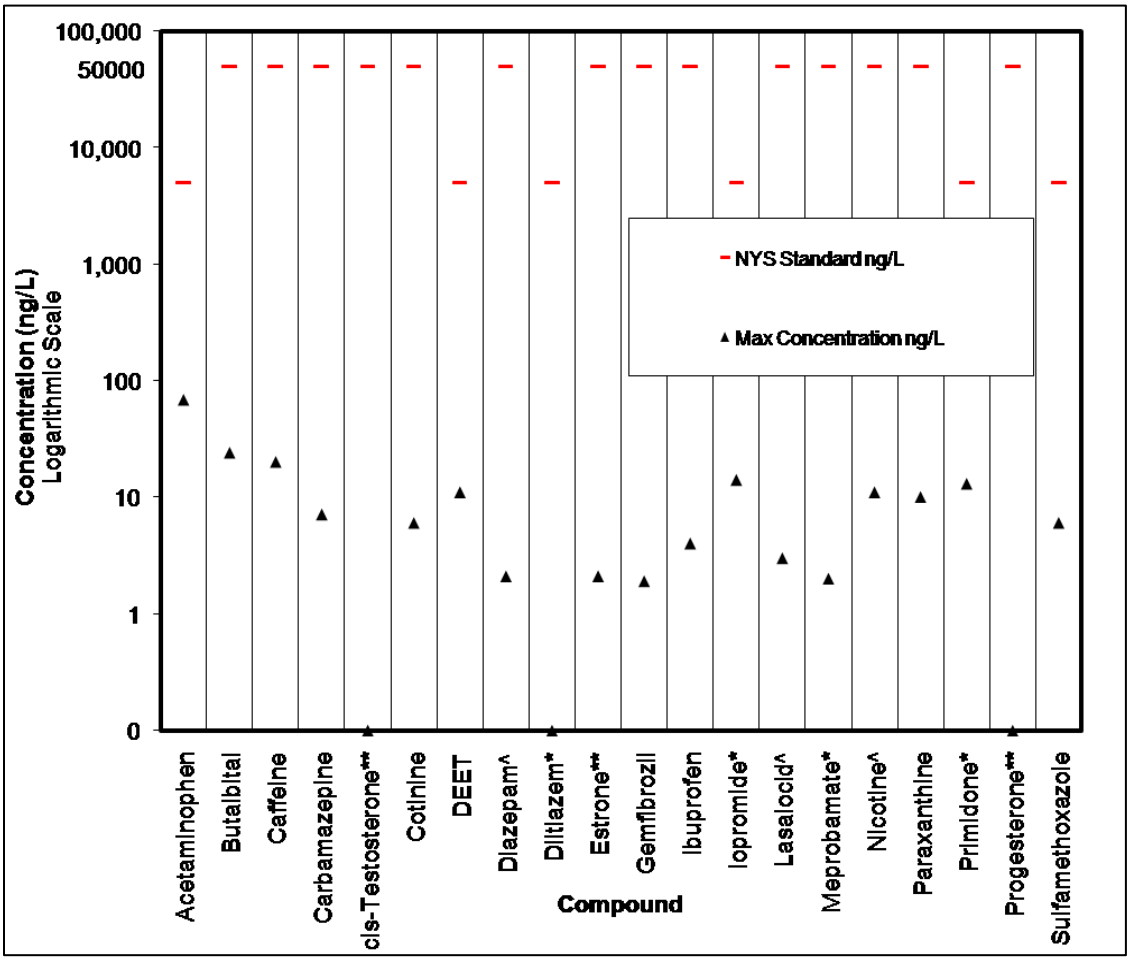
Measured concentrations were generally in the low parts-per-trillion range (ng/L), with most concentrations below 10 ng/L with all concentrations falling well below the New York State generic standard for UOCs of 50,000 ng/L and of 5,000 ng/L for principal organic compounds (POCs) (**Figure 2**). Many of the compounds that were detected were relatively close to or just above the minimum reporting limits for the method.

Table 5: Detected Compounds and General Use Category for 2010 and 2009

Compound	Type of Compound
Acetaminophen	fever reducer, nonprescription drug
Butalbital	barbiturate, pain reliever, prescription drug
Caffeine	stimulant
Carbamazepine	anti-convulsant, prescription drug
cis-Testosterone^	reproductive hormone
Cotinine	nicotine metabolite
DEET	insect repellent
Diazepam^	anti-anxiety/insomnia, prescription drug
Diltiazem*	antihypertensive
Estrone^	reproductive hormone
Gemfibrozil	cholesterol lowering, prescription drug
Ibuprofen	anti-inflammatory, nonprescription drug
Iopromide*	x-ray contrast media
Lasalocid^	antibiotic
Meprobamate*	sedative
Nicotine^	stimulant, alkaloid
Paraxanthine	stimulant, caffeine metabolite
Primidone*	antiepileptic
Progesterone^	reproductive hormone
Sulfamethoxazole	antibiotic

*Compounds detected only in 2010
^Compounds detected in 2009 but not in 2010
PFOS, a fluorosurfactant, was also detected

Figure 2: 2009-2010 Maximum Detected Concentrations of PPCP Compounds vs. NYS Standard Levels



*Compounds detected only in 2010
^Compounds detected in 2009 but not in 2010
PFOS was also detected with a maximum concentration of 2.3 ng/L (NYS Standard = 50,000 ng/L)

Summaries of the positive detections of PPCPs found in the samples from CROIT, DEL18, CATLEFF, and DEL19 are provided in; **Table 6, Table 7, Table 8, and Table 9,** respectively. As indicated in these tables, the frequency of occurrence was greater in the Croton System than in the Catskill/Delaware System and the chlorinated water site DEL19. For example, at least 14 PPCPs were detected in at least one sample in the Croton system whereas less than 3 PPCPs were detected in the Catskill/Delaware system sites. Five PPCPs were detected in every sample at CRO1T (either by MWH or UL): butalbital, sulfamethoxazole, caffeine, DEET, and cotinine.[iv] Carbamazepine was also detected at CRO1T in three of the four quarters sampled. In contrast, there was only sporadic detection of a few PPCPs at the Catskill/Delaware system sites, and no detection of butalbital, sulfamethoxazole or carbamazepine. Caffeine was the only PPCP that showed up at all four sites at least once.

[iv]PFOS, an industrial chemical, was also detected at all four locations, though at trace levels of less than a part-per-trillion (ng/L) in the Catskill/Delaware System and less than 3 ng/L in the Croton system

In the Croton System, caffeine and butalbital were detected at consistently higher concentration than most other compounds. As noted in **Table 6,** they were found at a peak concentration of 20 ng/L and 14 ng/L, respectively, and at a 100 percent detection frequency at CRO1T. From 2003 to 2006, USGS conducted a nationwide study to assess the occurrence and concentrations of organic wastewater compounds, and also detected butalbital in approximately 50 percent of all samples, with a maximum concentration in wastewater effluent as high as 500 ng/L (Phillips et al., 2007). In another nationwide study from 1999-2000 caffeine was detected in approximately 70 percent of samples (Kolpin et al, 2002).

The occurrence and concentrations of other frequently detected compounds in this study such as carbamazepine, cotinine, DEET, and sulfamethoxazole are consistent with the results from other investigations within New York State, New Jersey, and the New York City Watershed, (Wilson et al., 2006; Benotti et al., 2006; Stackleberg et al., 2007; Palmer et al., 2008) as well as other national studies (Kolpin et al., 2002; Benotti et al., 2009).

Table 10 summarizes the compounds detected in 2009 and 2010. **Tables 6-9** provide the 2010 results for each location sampled. Four new compounds were detected sporadically at CROIT: iopromide, primidone, diltiazem, and meprobamate. Diltiazem and meprobamate were detected at low levels at or near their MRLs while iopromide, an x-ray contrast media, and primidone, an antiepileptic, were detected at maximum concentrations on 14 ng/L and 13 ng/L, respectively. DEET was more prevalent in 2010, appearing in three out of four quarters versus just one quarter in 2009. Gemfibrozil which was detected at CRO1T in all four quarters in 2009, was detected in only one quarter in 2010. Diazepam which was detected in three of four quarters in 2009 was not detected in 2010.

Sporadic detections of acetaminophen, caffeine, cotinine, and DEET were observed at DEL18 and/or CATLEFF. Acetaminophen, ibuprofen, and caffeine were also detected sporadically at DEL19 which is a chlorinated site. The maximum concentration of acetaminophen at DEL19 was 68 ng/L which is the highest value recorded in either 2009 or 2010. However, the sample duplicate was only 5 ng/L so this result seems to be an outlier, and may have been a QC problem. Ibuprofen was detected in only one sample at Del19 but not it's duplicate. Three hormones that were detected in 2009 at extremely low levels; progesterone, estrone, and cis-testosterone were tested for in 2010 using method EDC2SCR but were not detected. Slightly higher MRLs were used by the contract laboratories in 2010, and this may account for the absence of these hormones. In addition, lasalocid which was detected in one quarter in 2009 in the Croton system was not detected at all in 2010 in the three of four quarters examined using method UL220.

Table 6: 2010 Summary of Positive PPCP Detections at the Croton Water Supply Source Water Testing Point (CRO1T)

Compound	MRL (ng/L)	1st Quarter		2nd Quarter		3rd Quarter		4th Quarter	
		CROGH	CROGH-DUP	CROGH	CROGH-DUP	CROGH	CROGH-DUP	CROGH	CROGH-DUP
Acetaminophen (1)	1	6.1	5.5						
Acetaminophen (2)	5			8	11				
Butalbital(1)	5	14	13	13	12	9.2	10	8.6	7.5
Caffeine (1)	3	16	18	20	20	18	16	12	11
Carbamazepine (1)	5							6.9	7.1
Carbamazepine (2)	1			3	3	3	3	5	5
Cotinine (1)	1	2.9	2.8	2.3	4.1	1.9	2.2	4	4.3
Cotinine (2)	1			3	2	2	2	3	3
DEET(2)	5			5	5	6	6	9	8
Diltiazem(2)	0.1					0.1	0.1		
Gemfibrozil(1)	1	1.5	1.5						
Gemfibrozil(3)	0.5	1.9	1.5						
Iopromide(1)	10	12	14						
Ibuprofen(1)	1		3.4						
Meprobamate(2)	1			1	1			2	2
Paraxanthine(2)	5			10	5			5	6
Primidone(2)	5					6		13	8
Sulfamethoxazole(2)	1			4	4	4	3	6	6
Sulfamethoxazole(1)	1	5.8	6.4	3.6	2.6	4.4	4.7	5.3	5.4

All concentrations listed in ng/L
(1) - MWH Method EDC2SCR
(2) - UL Method UL220
(3) - UL Method UL221
PFOS was also detected ranging from 0.2 to 2.3 ng/L

Table 7: 2010 Summary of Positive PPCP Detections at Delaware Water Supply Source Water Testing Point (DEL18)

Compound	MRL (ng/L)	1st Quarter		2nd Quarter		3rd Quarter		4th Quarter	
		DEL18	DEL18-DUP	DEL18	DEL18-DUP	DEL18	DEL18-DUP	DEL18	DEL18-DUP
Caffeine(1)	3			3.2	3.1				
Cotinine(2)	1							1	
DEET(2)	5			7	7				

All concentrations listed in ng/L
(1) - MWH Method EDC2SCR
(2) - UL Method UL220
(3) - UL Method UL221
PFOS was also detected ranging from 0.2 to 0.34 ng/L

Table 8: 2010 Summary of Positive PPCP Detections at the Catskill Water Supply Source Water Testing Point (CATLEFF)

Compound	MRL (ng/L)	1st Quarter		2nd Quarter		3rd Quarter		4th Quarter	
		CATLEFF	CATLEFF-DUP	CATLEFF	CATLEFF-DUP	CATLEFF	CATLEFF-DUP	CATLEFF	CATLEFF-DUP
Acetaminophen(2)	5			3	8				
Caffeine(1)	3			5.7	3.1				

All concentrations listed in ng/L
(1) - MWH Method EDC2SCR
(2) - UL Method UL220
(3) - UL Method UL221
PFOS was also detected ranging from ND to 0.38 ng/L

Table 9: 2010 Summary of Positive PPCP Detections at Catskill/Delaware Water Chlorinated Water Testing Point (DEL19)

Compound	MRL (ng/L)	1st Quarter		2nd Quarter		3rd Quarter		4th Quarter	
		DEL19	DEL19-DUP	DEL19	DEL19-DUP	DEL19	DEL19-DUP	DEL19	DEL19-DUP
Acetaminophen(1)	1				4.8				
Acetaminophen(2)	5			68	5				
Caffeine(1)	3			3.2	3.1	5.9	5.4		
Ibuprofen(1)	1						1.4		

All concentrations listed in ng/L
(1) - MWH Method EDC2SCR
(2) - UL Method UL220
(3) - UL Method UL221
PFOS was also detected ranging from 0.2 to 0.38 ng/L

Table 10: Summary of Positive PPCP Detections for 2009 and 2010 by Sample Site

	2009					2010					
Compound	CROGH	CATLEFF	DEL18	Trip Blank	Field Blank	DEL19	CROGH	CATLEFF	DEL18	Trip Blank	Field Blank
Acetaminophen	x		x			x	X	x			
Atenolol											x
Azithromycin**											
Bisphenol A					x						
Butalbital	x						X				
Caffeine	x		x			x	X	x	x		
Carbamazepine	x						X				
Ciprofloxacin										x	x
cis-Testosterone	x	x	x								
Cotinine	x	x	x				X		x		x
DEET	x								x		
Diazepam	x										
Diltiazem							X				
Estrone		x	x								
Gemfibrozil	x						X				
Ibuprofen	x	x	x		x	x	X				
Iopromide							X				
Lasalocid	x										
Meprobamate							X				
Nicotine	x	x	x								x
Norfloxacin										x	x
Paraxanthine	x						X				
Primidone							X				
Progesterone			x								
Sulfamethoxazole	x						X				
Sulfadimethoxine											x
Triclosan*											x

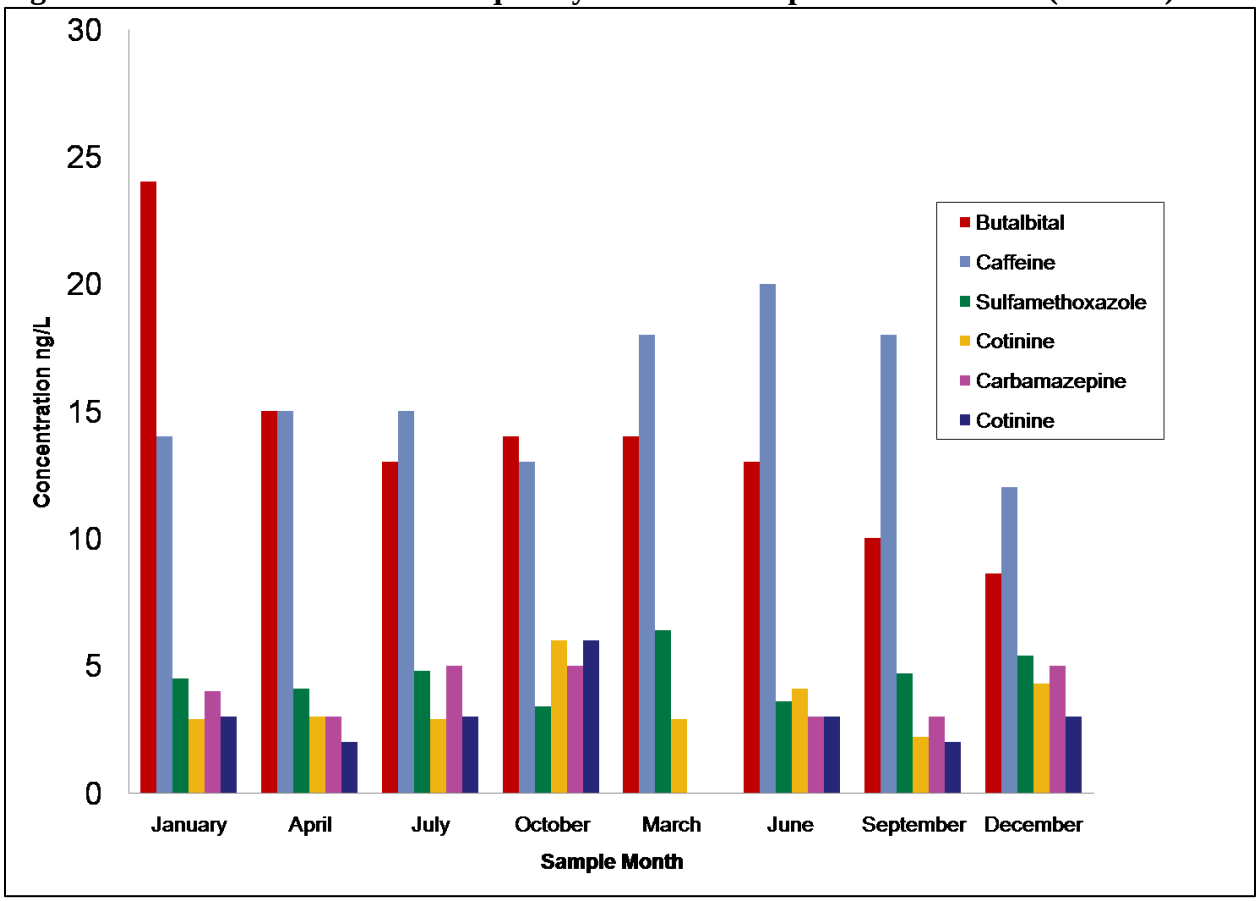
*Low percent recovery

** Detected in Lab Blank

PFOS was also detected at all sample sites in both 2009 and 2010

Seasonal variations are difficult to determine given the limited scope of this study. Studies focusing on fluctuations in concentration of PPCPs generally point to seasonal usage patterns, differences in contributing populations and input percentages of wastewater effluent to natural stream flow as primary reasons for fluctuation (Palmer et al., 2008; Takao et al., 2008; Loraine et al., 2006). Some studies point to a lack of seasonal variation for some PPCP constituents. For example, a 2006 study of effluent concentrations by *Brun et al.*, a found that carbamazepine is consistently present throughout the year at intermediate levels. **Figure 3** displays seasonal variations of PPCPs at CRO1T from 2009-2010. CRO1T seasonal concentration trends of butalbital observed in 2009 were not replicated in 2010; with the lowest concentration being detected in the winter of 2010 which is the inverse of the 2009 study. Caffeine concentrations peaked in June 2010, though a similar pattern was not observed in 2009. Cotinine trends were also not repeated. In 2009, lower concentrations of cotinine were detected during July the warmest sampling period; while in 2010, no trend was discernable. Carbamazepine and sulfamethoxazole continued to have no seasonal trend as they were detected at consistent levels at CRO1T during each sampling event. Generally speaking, the difference between peak values and minimum values over the period of study for each constituent did not vary by more than a factor of about two-fold. Overall, the data are too limited and inconsistent to draw any firm conclusions regarding seasonal trends.

Figure 3: Seasonal Variation of Frequently Detected Compounds at CRO1T (2009-10)



Health Implications

Although the human health risks associated with the presence of PPCPs in drinking water have not yet been thoroughly studied, several screening level risk assessments have concluded that no appreciable human health risk exists for the trace levels of PPCPs detected in this and other comparable studies (Bruce et al., 2010; Rahman et al., 2009; Snyder et al., 2008; Wilson et al., 2006; Schwab et al., 2005; Schulman et al., 2002). USEPA has summarized the different approaches that have been used to screen for human health risk from pharmaceuticals in drinking water (USEPA, 2008). In general, these approaches utilize existing toxicological data on acceptable therapeutic doses, or toxicological thresholds such as Acceptable Daily Intakes (ADIs), or Lowest or No Adverse Effect Levels (LOAELs or NOAELS), to establish some type of reference dose or point of departure to compare with screening level exposure estimates. In some cases, uncertainty factors are added to the "acceptable" toxicological reference value to account for intra- and inter-species differences in toxicity, as well as for gender, age or individual differences in susceptibility to toxicants. These numbers are then used to calculate screening level health risk metrics such as a Margin of Exposure (MOE). Otherwise stated, the MOE is the ratio of the no-observed adverse-effect-level (or other toxicological threshold such as an ADI) to the estimated exposure dose.

Table 11 provides DEP's application of this type of methodology. Specifically, DEP utilized a screening level approach similar to that used by Snyder (Snyder, Trenholm, et. al., 2008). This MOE approach compares the number of glasses of water that would have to be consumed to exceed a drinking water guideline (DWG) value derived by the authors from ADIs and other toxicological information. In most instances, the DWG is based on either the lowest therapeutic dose or the ADI.

The MOE for caffeine provides some perspective on the minute quantities of pollutants found in the study. For example, it would take almost 180 million 8-oz. cups of water at the maximum concentration of caffeine detected in this study of 20 ng/L to exceed a drinking water guideline value represented by the amount of caffeine in one 8-oz. cup of coffee. For the remaining compounds, the number of glasses of water required to exceed an acceptable daily intake, the MOE, is well over 1000, often by several orders of magnitude. Consistent with the conclusions of other screening level risk assessments, these large MOEs suggest that the risks to the health of New York City consumers are likely to be *de minimis.*

Table 11: Number of Glasses of Water Required to Exceed Derived Drinking Water Guideline

Detected Compound	NYS Standard[Ω] (ng/L)	∞Max Conc. (ng/L)	Toxicity Threshold	Units	Basis	Derived DWG (ng/L)	[#]No. of 8 oz. glasses of water/day to exceed DWG	Reference
Acetaminophen	5,000	68	50	µg/kg/day	ADI	175,000	21,772	v
Butalbital	50,000	14	5,000	µg/kg/day	MRTD	175,000,000	105,750,000	vi
Caffeine	50,000	20	100	mg/(8-oz-cup)		423,000,000	178,929,000	vii
Carbamazepine	50,000	7.1	200	mg/day	LTD	100,000	119,155	iii
cis-Testosterone	50,000	0.1	2	µg/kg/day	ADI	7,000	592,200	iii
Cotinine	50,000	4.3	20	mg/day	LTD	10,000	19,674	iii
DEET	5,000	9	0.1	mg/kg/day	ADI	3,500,000	3,290,000	viii
Diazepam^	50,000	2.1	5	mg/day	LTD	2,500	10,071	iii
Diltiazem*	5,000	0.1	1.7	µg/kg/day	ADI	60,000	5,076,000	iii
Estrone**	50,000	2.1	0.013	µg/kg/day	ADI	460	1,853	ix
Gemfibrozil	50,000	1.9	1,200	mg/day	LTD	600,000	2,671,579	iii
Ibuprofen	50,000	3.4	800	mg/day	LTD	400,000	995,294	iii
Iopromide*	5,000	14	21	µg/kg/day	ADI	750,000	453,214	iii
Lasalocid^	50,000	3	NI				NA	
Meprobamate*	50,000	2	40	µg/kg/day	MRTD	1,400,000	5,922,000	iv
Nicotine^	50,000	11	NI				NA	
Paraxanthine	50,000	10	NI				NA	
Primidone*	5,000	13	8.33	µg/kg/day	MRTD	291,550	189,732	iv
Progesterone	50,000	0.1	30	µg/kg/day	ADI	105,000	8,883,000	iii
Sulfamethoxazole	5,000	6	10	µg/kg/day	ADI	35,000	49,350	iii

[Ω]NYS standard for UOCs = 50,000 ng/L and POCs = 5,000 ng/L.
[#]No. of 8 oz glasses/day = [DWG (ng/L)* 2 (L/d)*4.23 8oz glasses]/L/(maximum water concentration (ng/L))
∞Max Concentration is for 2010 results except for compounds not detected in 2010
*Compounds detected only in 2010
^Compounds detected in 2009 but not in 2010
ADI = **Acceptable Daily Intake**. Maximum amount of a substance to which an individual can be exposed, on a daily basis over his or her life span, without causing any harmful effects.
DWG = **Drinking Water Guideline**. Health-based guideline values representing minimum requirements for drinking water safety. Values cited are from reference 1 unless otherwise noted.
LTD = **Lowest Therapeutic Dose**. The LTD which produces the desired clinical effect.
MRTD = **Maximum Recommended Therapeutic Dose**. The recommended maximum amount of a drug to be given to a patient without causing adverse health effects.
PHA = **Provisional Health Advisory**. PHAs are developed to provide information in response to an urgent or rapidly developing situation. They reflect reasonable, health-based hazard concentrations above which action should be taken to reduce exposure to unregulated contaminants in drinking water.
NI =No Information

[v]*Australian Guidelines for Water Recycling*, Augmentation of Drinking Water Supplies, May 2008, Environment Protection and Heritage Council, National Health and Medical Research Council, Natural Resource Management Ministerial Council.
[vi]U.S. Food and Drug Administration (FDA), Maximum Recommended Therapeutic Dose (MRTD) Database. http://www.fda.gov/aboutfda/centersoffices/cder/ucm092199.htm.
[vii]Gilbert SG. *A Small Dose of Toxicology – The Health Effects of Common Chemicals*. CRC Press, Boca Raton, February 2004.
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Conclusions

The results of 2010 study were similar to the pilot study conducted in 2009. As indicated in **Table 5**, a total of 14 PPCPs were detected in at least one sampling event during 2010. The compounds detected at CRO1T include: acetaminophen, ibuprofen, iopromide, DEET, paraxanthine, meprobamate, diltiazem, primidone, butalbital, sulfamethoxazole, carbamazepine, caffeine, cotinine, and gemfibrozil. Caffeine was also detected at the three other sites (DEL18, CATLEFF, and DEL19). There were also sporadic detections of DEET and cotinine at DEL18; acetaminophen at CATLEFF; and acetaminophen and ibuprofen at DEL19. A screening level assessment conducted by DEP indicates that the low concentrations of PPCPs detected in this study are well below levels that would pose a risk to the health on consumers of NYS's drinking water.

Some notable findings from DEP's 2010 results are the consistent detection of butalbital, caffeine, sulfamethoxazole, carbamazepine, and cotinine at CRO1T. Findings for 2010 were similar to those in the 2009 pilot study, with many of the same compounds reoccurring at similar levels (**Tables 6-9).** Some exceptions include diazepam, ibuprofen and gemfibrozil which were detected less frequently in 2010. In addition, acetaminophen was detected at three of four sampling sites in the 2nd quarter of 2010.

Positive detections of sulfamethoxazole, gemfibrozil, carbamazepine, caffeine, ibuprofen, cotinine, and diazepam at CRO1T are similar to results seen in other studies of wastewater compounds in surface waters (Kolpin et al., 2002; Benotti et al., 2009; Stackleberg et al., 2007; Heberer et al., 2001). Other studies have identified wastewater treatment plants as a considerable point source of pharmaceutical and organic compound pollution into receiving waters (Benotti et al., 2006; Phillips et al., 2007; Phillips et al., 2010). Wastewater treatment plants in the Croton and Catskill/Delaware Watersheds may be potential sources of pharmaceutical and personal care product compounds in surface waters (Palmer et al., 2008). The greater frequency of detection in the Croton's source water compared to the Catskill/Delaware's source water can possibly be attributed to greater wastewater inputs relative to natural stream flows and higher population density surrounding the Croton watershed.

In 2010, samples were also collected at DEL19 to detect any difference in PPCP concentrations after water has been chlorinated. DEL19 was receiving water from the Catskill and Delaware system at the time of sampling; therefore, comparison was made between both CATLEFF and DEL18 to DEL19. While some treatment and disinfection techniques have been known to partially or wholly remove, degrade or transform certain PPCP related compounds; no discernable change was observed at DEL19. Of the compounds detected at DEL 18, CATLEFF and DEL19, the literature from bench scale and other studies suggests that some of the detected compounds may be affected more readily by chlorine disinfection than others. Caffeine, cotinine, and ibuprofen have generally low reactivity with free chlorine under ambient pH conditions, whereas acetaminophen is removed or changed into new products by the addition of chlorine (Stackleberg et al., 2007) (Snyder et al., 2007)(Bender, 2006) . The results of DEP's study indicated that chlorine oxidation had little noticeable effect on the reduction of caffeine concentrations. At DEL18, cotinine was detected at the MRL in one 4th quarter sample, and was undetected at DEL19; however, there is minimal evidence in support of chlorination decreasing the concentration of cotinine.

The measured concentrations of the PPCP target analytes at all four sampling locations were all well below the New York State generic standards for UOCs or POCs of 50,000 ng/L and 5,000 ng/L, respectively.

In addition to the PPCPs that were detected, PFOS, an industrial chemical was also detected at all four sampling locations. The results are similar to 2009 with the highest concentrations detected at CRO1T and with a maximum concentration of 2.3 ng/L. The PFOS levels detected at all four sampling locations were well below EPA's Provisional Health Advisory level of 200 ng/L in drinking water (USEPA, 2009).

DEP believes that the overall quality of the data provided by the contract labs was acceptable. The few problems with blank contamination indicate the importance of collecting QC samples such as trip blanks and field blanks to help ensure that positive detections attributable to samples are not the result of trace contamination due to field or laboratory procedures.

Going forward DEP will use the experience gained through this study to prepare for UCMR3 sampling in 2013. Several of the compounds that are required to be monitored for under UCMR3 were part of our target analytes. Under UCMR3, EPA will be updating and providing standard analytical methods to ensure the results of this national effort meet appropriate and standardized levels of quality.

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