



United States
Environmental Protection
Agency

Occurrence of Contaminants of Emerging Concern in Wastewater From Nine Publicly Owned Treatment Works

August 2009

U.S. Environmental Protection Agency
Office of Water (4303T)
1200 Pennsylvania Avenue, NW
Washington, DC 20460

EPA-821-R-09-009

CONTENTS

	Page
1. INTRODUCTION	1
2. BACKGROUND.....	2
3. STAGED STUDY	4
3.1 Stage 1 (2005-2006).....	4
3.2 Stage 2 (2007-2008).....	6
4. SAMPLE COLLECTION	11
4.1 Sampling Locations and Procedures.....	11
4.1.1 Sampling Locations	11
4.1.2 Sampling Procedures	15
4.2 Quality Assurance.....	17
4.3 Quality Control Procedures.....	17
4.3.1 Equipment Blanks.....	18
4.3.2 Field Duplicates	18
4.3.3 Laboratory Quality Control Volumes.....	18
4.3.4 Trip Blanks.....	18
4.3.5 Quality Control Sample Summary.....	19
4.4 Sample Collection, Preservation, and Storage.....	19
5. SUMMARY OF OCCURRENCE DATA	26
5.1 PPCPs.....	27
5.2 Sterols and Hormones.....	33
5.3 Alkylphenols, APEs, and BPA	35
5.4 PBDEs.....	36
5.5 Pesticides.....	37
6. REFERENCES	40
Appendix A: Contaminants of Emerging Concern (CECs) Analyte list	
Appendix B: Analytical Methods Overview	
Appendix C: CECs Analytical Results	

1. INTRODUCTION

The primary objective of EPA's Nine POTW Study (the Study) was to investigate the occurrence of Contaminants of Emerging Concern (CECs) in untreated and fully treated wastewater at publicly owned treatment works (POTWs). EPA also initially investigated the occurrence of conventional, non-conventional, and toxic pollutants, including many of the pollutants in EPA's 1982 "50-POTW Study" (EPA, 1982). For the first four POTWs in this Study, EPA selected facilities that treated industrial discharges because POTWs receiving significant volumes of discharges from pharmaceutical or other manufacturing facilities might be expected to receive a significant quantity and variety of CECs.

After reviewing analytical data from the first four POTWs, EPA realized that the available analytical methods were not developed enough to meet the needs of the Study. As a result, prior to collecting samples for the remaining POTWs, EPA developed three analytical methods to detect the occurrence of CECs in POTW wastewaters. These three methods are: EPA 1694 for pharmaceuticals and personal care products, EPA 1698 for steroids and hormones, and EPA 1699 for pesticides.

During the development of these three methods, EPA reevaluated the design of the Study and decided to focus the remaining resources on documenting the occurrence of CECs in wastewater, and ceased collecting data for the other non-CEC parameters previously analyzed. EPA also revised the POTW selection criteria to focus on facilities with specific sources of residential flow expected to contain higher concentrations of CECs.

This report presents the findings from the Nine POTW Study for informational purposes. Caution should be used in interpreting the results. For several reasons, the study does not lend itself to national conclusions. The Study had two very distinct stages, each with different analytical methods, sampling techniques and POTW selection criteria. The nine POTWs in this study are not statistically representative of all POTWs in the country, nor would they be expected to statistically represent particular subpopulations of POTWs. The analytical methods were under development during much of this study, which resulted in certain data quality issues during laboratory analysis. Additionally, sample collection efforts were not designed to calculate removal of CECs from POTWs. Although some reduction in concentration levels of many CECs was observed in the effluent, the data are not sufficient to draw any conclusions about treatment effectiveness.

2. BACKGROUND

In 2005, EPA began studying environmental contamination by pharmaceuticals, detergents, natural and synthetic hormones, and other chemicals. These contaminants are commonly referred to collectively as contaminants of emerging concern.

Many CECs are believed to enter municipal wastewater through bathing, cleaning, laundry, and the disposal of unused pharmaceuticals and human waste. Municipal wastewater is treated by POTWs, which typically employ biological treatment and disinfection processes and, in some instances, a form of advanced treatment. These processes are designed to reduce the load of organic pollutants and pathogens in the treated wastewater discharged to the environment; however, POTW treatment systems are not designed to specifically remove CECs. Thus, any removals of CECs which may occur are incidental to these treatment processes.

To assess the occurrence of CECs in POTW influent and effluent, and to test and develop new analytical methods with which to measure these emerging contaminants, between September 2005 and July 2008, EPA collected samples for one day at each of nine POTWs. This study is referred to in this report as the “Nine POTW Study”, or “the Study.” The CECs in this study include the following classes of chemicals:

- *Pharmaceuticals and Personal Care Products (PPCPs)* – A variety of pharmaceuticals and personal care products are used by individuals for personal health or cosmetic reasons. Pharmaceuticals include over-the-counter medication (e.g., aspirin, acetaminophen, and pseudoephedrine) as well as medications prescribed by a physician (e.g., Lipitor[®], albuterol, amoxicillin). Most ingested pharmaceuticals are only partially metabolized, so a portion is excreted, unmetabolized, in urine or feces. Metabolized and unmetabolized pharmaceuticals are discharged in domestic sewage.

Personal care products include chemicals such as soaps, detergents, shampoo, cosmetics, sun-screen products, fragrances, insect repellants, and antibacterial compounds. An example of a personal care product is triclosan, a potent wide-spectrum antibacterial and antifungal agent. Personal care products enter domestic wastewater from bathing, laundry, and household cleaning.

- *Steroids and Hormones (S/H)* – Steroids and hormones include both naturally occurring compounds and synthetic analogues that are structurally related to one another. Hundreds of distinct steroids are found in plants and animals. Sterols, which are steroid-based alcohols, are the most abundant of the steroids. The most common sterol in vertebrates is cholesterol, which is found in cell membranes and also serves as a central intermediate in the biosynthesis of many biologically active steroids, including bile acids, corticosteroids, and sex hormones.

Hormones are intercellular chemical messengers. They are synthesized and secreted from a cell and act in low concentrations by binding to a stereospecific target-cell receptor to activate a response. Some hormones are classified by chemical structure as steroids. Steroid hormones include the sex hormones, which are, among others, natural estrogens, synthetic estrogens such as EE2 (17 alpha-

ethinyl estradiol), progesterone, and testosterone. Other hormones are polypeptides or amino acid-derived compounds. Plant steroids can mimic animal sex hormones.

- *Alkylphenols and Alkylphenol Ethoxylates (APEs)* – Alkylphenol ethoxylates (APEs) are synthetic surfactants used in some detergents and cleaning products. The most common APEs are nonylphenol ethoxylates (NPEs), derived from nonylphenol (NP), which is an alkylphenol. Octylphenol ethoxylates (OPEs), derived from octylphenol (OP), are also common.
- *Bisphenol A (BPA)*, also known as 4,4'-isopropylidenediphenol, is an organic compound used primarily to make polycarbonate plastic and epoxy resins. Polycarbonate is used in eyeglass lenses, medical equipment, water bottles, CDs, DVDs, and many other consumer products. Among the many uses for epoxy resins are can coatings, industrial floorings, automotive primers, and printed circuit boards.
- *Polybrominated Diphenyl Ethers (PBDEs)* – Polybrominated diphenyl ethers (PBDEs) are structurally similar to Polychlorinated biphenyl. PBDEs are major components of commercial formulations often used as flame retardants in furniture foam (e.g., pentaBDE), plastics for TV cabinets, consumer electronics, wire insulation, back coatings for draperies and upholstery (e.g., decaBDE), and plastics for personal computers and small appliances (e.g., octaBDE). These chemicals slow ignition and rate of fire growth.
- *Pesticides* – Pesticides are any of a large number of unrelated chemicals that are used to prevent, destroy, or repel a living organism that occurs where it is not wanted (i.e., a pest). Pesticides are often referred to according to the type of pest they control (e.g., insecticides, rodenticides, fungicides). Pesticides include organochlorine, organophosphorus, triazine, and pyrethroid pesticides.

3. STAGED STUDY

During this Study, EPA collected samples from nine POTWs located in eight states. These POTWs are referred to in this report as Plants A through I. All of the sampled POTWs used variations of the activated sludge process, including some degree of nitrification. All POTWs disinfected some or all of their treated effluent prior to discharge or land application.

The Study was carried out in two stages. In the first stage (Plants A through D), EPA examined the occurrence of contaminants of emerging concern (CECs), including endocrine disrupting compounds and other potentially harmful compounds. After sampling these first four POTWs in 2005 and 2006, EPA concluded that it needed more sensitive and selective analytical methods. Method development took place from 2006-2008. See Appendix B for an overview of EPA's CECs analytical methods. With development of better analytical methods well underway, and with changes in both POTW site selection criteria and sampling approach (as described in Section 3.2 of this report), EPA began the second stage of the Study in September 2007 (Plants E through I).

3.1 Stage 1 (2005-2006)

The first stage of the Study was designed to determine the occurrence of pollutants at POTWs based on one day of sampling. In addition to obtaining this relatively limited information on pollutant occurrence, EPA anticipated that the Study results would identify candidate POTWs for future performance sampling should such a decision be made to do so in the future. Samples taken during this first stage were analyzed for priority pollutants in order to build on the information collected during EPA's 1982 "50-POTW Study¹." During Stage 1, EPA also evaluated the available analytical methods for detecting CECs in wastewater.

During Stage 1, EPA sampled POTWs with a high proportion of flow from industries believed to be discharging high concentrations of PPCPs. EPA used the 2004 Clean Watershed Needs Survey (CWNS), the 2004 Permit Compliance System (PCS) database, and the 2002 Toxic Release Inventory (TRI) to gather information on influent contributions, treatment unit operations, and performance. EPA verified information about selected POTWs gathered using these data sources through discussions with the facilities themselves. Using this information, EPA identified facilities with average influent loadings and typical discharge patterns. The four POTWs sampled during Stage 1 were selected because they met some or all of the following criteria:

- Influent flow greater than 1 million gallons per day (MGD).
- High industrial wastewater flow contribution from a variety of different industries as well as targeted industries that were expected to contribute CECs. Targeted industries include: manufacture of organic chemicals, plastics, and synthetic fibers; pharmaceutical manufacturing and testing; meat and poultry processing; dairy products processing; hospitals and health care facilities; and landfills.
- Adequate POTW performance (effluent BOD₅ concentration less than 20 mg/L).

¹ The focus of this report is on CECs and information collected on the priority pollutants is not presented.

- Use of a variety of treatment technologies including, technologies for nutrient (total nitrogen and total phosphorus) removal, various disinfection technologies (ultraviolet (UV) light, ozonation and chlorine disinfection).

During Stage 1 of the Study, EPA monitored for a lengthy list of pollutants including:

- Pollutants that were part of the 1982 50-POTW Study, including:
 - Volatile and semivolatile organic compounds,
 - Metals (total and dissolved) and,
 - Classical pollutants.
- Additional organic compounds:
 - Other volatile and semivolatile organic compounds,
 - Polybrominated diphenyl ethers (PBDEs),
 - Polychlorinated dioxins and furans, and
 - Chlorinated biphenyl congeners (PCBs);
- Microbiological pollutants; and
- Analytes for which methods were under development, including:
 - Pharmaceuticals and personal care products,
 - Steroids and hormones,
 - Alkylphenol ethoxylates (APEs), and
 - Pesticides and herbicides.

Stage 1 Facilities

Each of the four POTWs selected for Stage 1 is described in more detail below. Table 3-1 found at the end of this section summarizes the unit process descriptions and operating characteristics of all nine POTWs participating in the Study.

Plant A treats a relatively large proportion of industrial wastewater (18 percent of total flow is from industrial sources) and receives 3.5 million pounds per year of TRI-listed chemicals, the majority of this industrial flow coming from a pharmaceutical manufacturer. Sources of domestic wastewater treated at the plant include two colleges with on-campus housing (a university with 28,600 students and a college with 1,300 students). The total flow to Plant A is 26 MGD.

Plant A is a conventional activated sludge facility with powdered activated carbon (PAC) addition. The wastewater treatment process consists of mechanical bar screening at the comminutors, grit removal, ferric chloride addition for phosphorus precipitation, primary clarification, PAC addition to remove refractory organic compounds, aeration, secondary clarification, sand filtration, chlorine disinfection using chlorine gas, and dechlorination using bisulfite. Final treated effluent is discharged to a river.

Plant B receives flow from a variety of sources that potentially contribute CECs and other pollutants. The facility receives approximately 10 to 15 percent of influent flow from industrial sources and commercial facilities, as well as wastewater from three hospitals, domestic septage, and landfill leachate. The total flow to Plant B is 27 MGD.

Plant B is a conventional activated sludge facility. Wastewater treatment includes screening, grit removal, primary clarification, aeration, and secondary clarification. After secondary clarification, the facility conducts chlorine disinfection using sodium hypochlorite and dechlorination using sodium bisulfite, and the final treated effluent is discharged to a river. This plant does not use any biological nutrient removal or other more advanced processes.

Plant C receives significant industrial flow from sources that potentially contribute both CECs and other pollutants. The facility receives approximately five to seven percent of influent flow from industrial sources and commercial facilities and treats domestic wastewater, landfill leachate, and septage from local haulers. The total flow to Plant C is 17 MGD.

Plant C is also a conventional activated sludge facility. Wastewater treatment consists of screening, grit removal, ferric chloride addition to remove phosphorus (as necessary), primary clarification, aeration, secondary clarification, and UV disinfection. The final effluent is discharged into a lake.

Plant D receives wastewater from multiple targeted industries and uses a wide variety of treatment processes. This POTW receives approximately 17 percent of flow from industrial sources and commercial facilities, as well as wastewater from two hospitals and several universities, septage, and landfill leachate. The total flow to Plant D is 34 MGD.

Plant D is an advanced secondary and tertiary treatment facility, using several treatment unit operations in two treatment trains. Wastewater enters the facility and undergoes screening, grit removal, and primary clarification. After primary treatment, the wastewater is split into two treatment trains. The wastewater in Train 1 (56 percent of flow) first undergoes aluminum sulfate addition for phosphorus removal, then passes through pure oxygen aeration basins, secondary clarification, nitrification aeration basins, tertiary clarification, and mixed media denitrification filters before mixing with the secondary effluent from Train 2 prior to disinfection. Train 2 (44 percent of flow) treatment consists of biological phosphorus removal aeration basins with aluminum sulfate addition to assist in phosphorus removal, secondary clarifiers, and sand filters. The Train 1 and Train 2 effluents are mixed prior to ozone disinfection. The final effluent is discharged to a creek.

3.2 Stage 2 (2007-2008)

During Stage 1 of the Study, EPA determined that the analytical methods needed further development. This development took place during 2006-2008, and resulted in three new analytical methods, EPA Methods 1694, 1698, and 1699.

With method development well underway, Stage 2 sampling resumed during 2007-2008. By this time, there was more interest in directing available resources to document occurrence of CECs in POTW influent and effluent with the use of single-day (screening) sampling episodes.

Thus, in Stage 2 EPA did not analyze for any pollutants beyond what had been identified as CECs in Stage 1.

In Stage 2 EPA also changed the selection criteria for POTWs. Because CEC discharges can result from excretion of ingested CECs, EPA changed the selection criteria from facilities with higher industrial flow to those with higher residential flow.

To identify the populations of interest for Stage 2, EPA reviewed literature on the use of medications that may be found in wastewater. The Slone Epidemiology Center at Boston University has conducted an annual survey of medication use in the United States since 1998. The surveys indicated that in a given week, over 80 percent of adults in the United States take at least one medication (prescription or nonprescription drug, vitamin/mineral, or herbal/natural supplement). Medication use varies considerably by age. People over 65 are the largest consumers of medication. Of this age group, 57 to 59 percent take at least five medications in a week, and 17 to 19 percent take at least 10 (Slone, 2006). Thus, higher influent concentrations of pharmaceuticals may be found at POTWs that serve a higher than average proportion of population age 65 or older.

The Slone survey conducted in 1998-1999 (Kaufman, 2002) identified two population groups that frequently use hormone medications. In that study, 21 percent of women aged 45 to 65 and 17 percent of women over 65 used conjugated estrogens, presumably as part of hormone replacement therapy (HRT). Since 1998, the use of HRT has declined dramatically, but as of 2004, 12 percent of women over 50 continued to use this therapy. Higher POTW influent concentrations of estrogens may be found at POTWs serving a greater than average population of women over 50, though this effect may have decreased with the decline of HRT use.

The second population group that frequently uses hormone medications is women aged 18 to 44. In the 1998-99 Slone survey, 14 percent of this group used EE2 (17 alpha-ethinyl estradiol), the synthetic hormone that is the active ingredient in birth control pills (Kauffman, 2002). Data published by the National Center for Health Statistics show that 32 percent of all women between the ages of 20 and 24 used the birth control pill for contraception in 2002 (Chandra, 2005). According to the 2000 Census, 21 percent of college students are women in this age group compared to 6.5 to 6.8 percent of the general population (Census, 2007). As a result, the use of birth control pills among the college population may be greater than among the general population.

For Stage 2 site selection, EPA identified areas with high concentrations of college-aged populations or populations over 65. To identify such areas, EPA used information from the U.S. Census Bureau (population by age and total populations by county) and the U.S. News and World Report (college settings and student populations). EPA selected five facilities for Stage 2 sampling using the following criteria, in no particular order:

- Use of selected advanced treatment technology;
- Use of separate sewers, which segregates stormwater away from sewers directing wastewater to the POTW; and
- Service area includes a large college (at least 10,000 students) or more than 15 percent of population 65 or over.

Stage 2 Facilities

The five facilities selected for sampling during Stage 2 are described below. Table 3-1, found at the end of this section, summarizes the unit process descriptions and operating characteristics of the nine POTWs participating in the Study.

Plant E receives wastewater from a large university and surrounding towns. The POTW diverted town flow to another nearby POTW during the sampling episode, so samples collected represent only university-derived wastewater. Flow from the town was diverted far enough in advance of EPA's sampling, to allow for the system to treat and discharge all non-university flow prior to the sampling episode. The plant receives approximately 2 MGD from the university.

Plant E is a multi-train secondary treatment facility that operates three secondary treatment trains and disinfects using chlorine gas. The influent passes through grit removal, rag removal, primary aeration, and primary clarification. From here, treatment is split into three trains. The wastewater in Train 1 (25 percent of flow) is passed through trickling filters prior to rejoining with Train 2 wastewater (25 percent of flow), which passes through an anoxic tank before joining Train 1 wastewater. The combined secondary effluent from Trains 1 and 2 undergo secondary aeration and clarification with poly-aluminum chloride addition for phosphorus removal. Train 3 (50 percent of flow) is a two-step conventional activated sludge treatment process. The first step is a two-ring activated sludge basin; the inner zone provides aeration and the outer zone provides anoxic conditions. The second step, also a two-ring activated sludge basin, provides reaeration in the inner zone and clarification in the outer zone. Combined secondary effluent from Trains 1 and 2 and secondary effluent from Train 3 are all combined prior to disinfection using chlorine gas. The final effluent is used for spray irrigation on agricultural fields.

Plant F is located in a county with an above-average percentage of population over age 65. Twenty-two percent of the population in the county is over age 65, compared to the national average of 12.4 percent. Total flow to Plant F is 6.7 MGD.

Plant F is one of a few POTWs in the United States to use a five-stage Bardenpho™ treatment system with only bar screening and grit removal prior to the process. The Bardenpho™ system consists of five zones: anaerobic, anoxic, aerobic, anoxic, and reaeration. This process is designed to provide efficient, economical removal of BOD₅, TSS, nitrogen, and phosphorus. The Bardenpho™ system is followed by clarification, sand filters, and UV disinfection. Final effluent is either discharged to a river or reused for irrigation and cooling in the plant's service area.

Plant G treats flow mainly from a university and the off-campus student population for a total of 1.5 MGD. Plant G's flow spikes significantly during and after rain events; therefore, EPA planned and conducted the sampling episode to take place during a dry time of the year to prevent loading dilution.

Plant G has an oxidation ditch system (an extended aeration activated sludge process) with final discharge to a river. Treatment consists of bar screening, grit removal, fine screening, oxidation ditch treatment, secondary clarification, chlorine disinfection (sodium hypochlorite), and dechlorination (sodium bisulfite).

Plant H is located in a community with an above-average percentage of population over age 65. According to the 2000 U.S. Census data, 73 percent of the population served by Plant H is over age 65. The surrounding area is home to many active adult communities and retirement homes. Total flow to the facility is approximately 2 MGD.

Plant H also uses an oxidation ditch system. Treatment consists of screening, grit removal, oxidation ditch treatment, secondary clarification, tertiary media filters, and chlorine disinfection using sodium hypochlorite. The facility maintains chlorine residual in the treated effluent because it is discharged to groundwater percolation beds. The facility is permitted to discharge to a river, but rarely does so.

Plant I is also located in a community with an above-average percentage of population over age 65. According to the 2000 U.S. Census data, 25 percent of the population in the county where the POTW is located is over age 65, compared to the national average of 12.4 percent. The site contact noted that approximately 50 to 75 percent of the influent flow to Plant I is possibly from persons over 65. The surrounding area is home to many active adult communities and retirement homes. Total flow to the facility is approximately 1 MGD.

Plant I operates three sequencing batch reactors (SBRs) to achieve secondary treatment. Treatment consists of screening, grit removal, SBR activated sludge treatment, sand filtration, and chlorine disinfection with elemental chlorine. The final effluent is disposed of on on-site spray irrigation fields. The facility is also permitted to discharge to a swamp, but rarely does so.

Table 3-1. Descriptions of Participating POTWs

Plant	Primary Treatment	Secondary Treatment	Sludge Retention Time (SRT)	Nitrification	Sand or Media Filters	Type of Disinfection
A	Clarification with ferric chloride addition	Conventional activated sludge and PAC addition	20 days	Yes	Yes	Chlorine
B	Clarification	Conventional activated sludge	12 days	Yes	No	Chlorine
C	Clarification with ferric chloride (as necessary)	Conventional activated sludge	3.5 days	Yes	No	UV
D	Clarification	Train 1: Chemical phosphorus removal and pure oxygen activated sludge (with separate stage nitrification and denitrification) (56% flow)	2.1 days	Yes	Yes	Ozone
		Train 2: Activated sludge with an anoxic zone for biological phosphorus removals and chemical phosphorus removal (44% flow)	0.1 days	No	Yes	
E	Aeration and clarification	Train 1: Trickle filters (25% flow)	N/A	Yes	No	Chlorine
		Train 2: Anoxic tank (25% flow)		Yes		
		Train 3: 2-step activated sludge (50% flow)	Step 1: 19.4 days			
F	None	5-stage Bardenpho process (anaerobic, anoxic, aerobic, anoxic, aerobic)	16 days	Yes	Yes	UV
H	None	Oxidation ditch	12.4 days	Yes	Yes	Chlorine

4. SAMPLE COLLECTION

4.1 Sampling Locations and Procedures

During Stage 1, EPA collected samples of influent, final effluent, and at intermediate points in the treatment process. The wastewater was sampled periodically over a 24-hour period and the samples were composited for analysis. EPA also collected grab samples of either primary or combined sludge.

During most of Stage 2, EPA collected samples of influent and final effluent only. EPA's objective during Stage 2 was to identify POTWs with high influent concentrations of CECs, not to estimate daily influent load. POTW influent flows follow a characteristic diurnal pattern, with lowest flows in the very early morning hours and peak flows in mid-morning. Influent BOD₅ concentration follows the flow variation (Metcalf and Eddy, 1978), and EPA assumed that CECs concentrations, like the BOD₅ concentrations, peak with the flow. EPA decided it could identify POTWs with high influent concentrations of CECs by collecting one-time grab samples during periods of peak flow. For this reason, during the last three sampling episodes, samples were collected as grab samples or manual grab composite samples. EPA did not collect any sludge samples during Stage 2.

During Stage 2 there was a seasonal component to scheduling sampling events. The targeted populations were anticipated to have predictable seasonal migratory patterns. EPA sampled POTWs with large college populations during the school year, in order to get flow associated with their student population. Similarly, EPA sampled POTWs with a high percentage of populations over 65 during times this population was anticipated to be at their residences. EPA avoided sampling these POTWs during summer months and major holidays, when the populations may travel to escape warm summer climates or visit family.

EPA reviewed historical monthly flow patterns for the Stage 2 facilities in order to determine when the daily flow to the specific POTWs declined, indicating that the target populations may have left the area. All of the sampling in Stage 2 took place outside periods of reduced flow. See Table 4-1 for the sampling dates.

Table 4-1. POTW Sampling Episode Dates

	Plant								
	A	B	C	D	E	F	G	H	I ^a
Sampling Date	9/23/2005	6/21/2006	8/9/2006	9/13/2006	11/12/2007	11/15/2007	4/8/2008	4/10/2008	7/29/2008

^a While this sampling event took place during summer months, the historical information indicated flow remained fairly consistent during the summer with peaks in October and drops off in November and December, two major holiday months.

4.1.1 *Sampling Locations*

At four plants, EPA collected influent samples after bar screening and grit removal, but before primary treatment and recycle stream mixing when feasible (Plants A, B, D, and H). At the remaining plants, EPA collected influent samples prior to the grit chamber (Plants E, F, and G) or influent channel bar screen (Plants C and I).

At Plants A, F, G, H, and I, EPA collected samples only at the influent and final effluent locations.

At Plants B and C, EPA collected samples of intermediate secondary effluent in addition to samples of influent and final effluent. Secondary effluent samples were collected directly after secondary clarification but before disinfection.

Because Plant D uses a variety of treatment units, EPA collected samples at several intermediate points.

Due to the long SRT of the two-step activated sludge treatment system at Plant E, EPA chose to sample the influent to and effluent from the activated sludge treatment system at this facility.

Figures 4-1 through 4-9 present process schematics for each POTW. Sampling points are noted on each of the plant schematics.

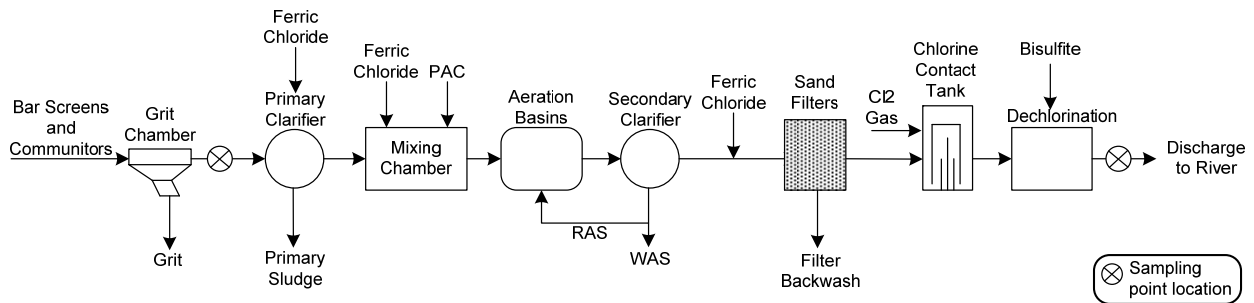


Figure 4-1. Process Schematic of Plant A

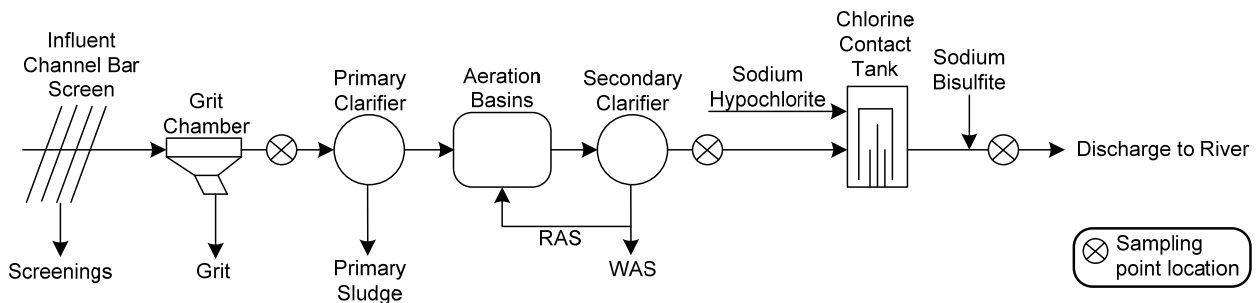


Figure 4-2. Process Schematic of Plant B

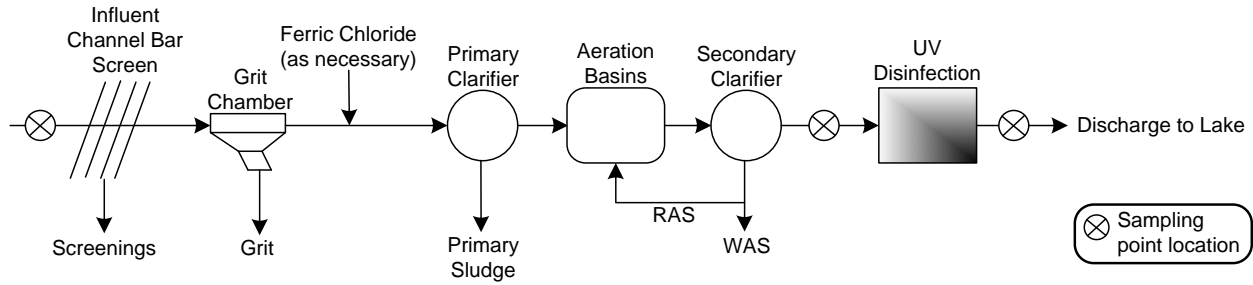


Figure 4-3. Process Schematic of Plant C

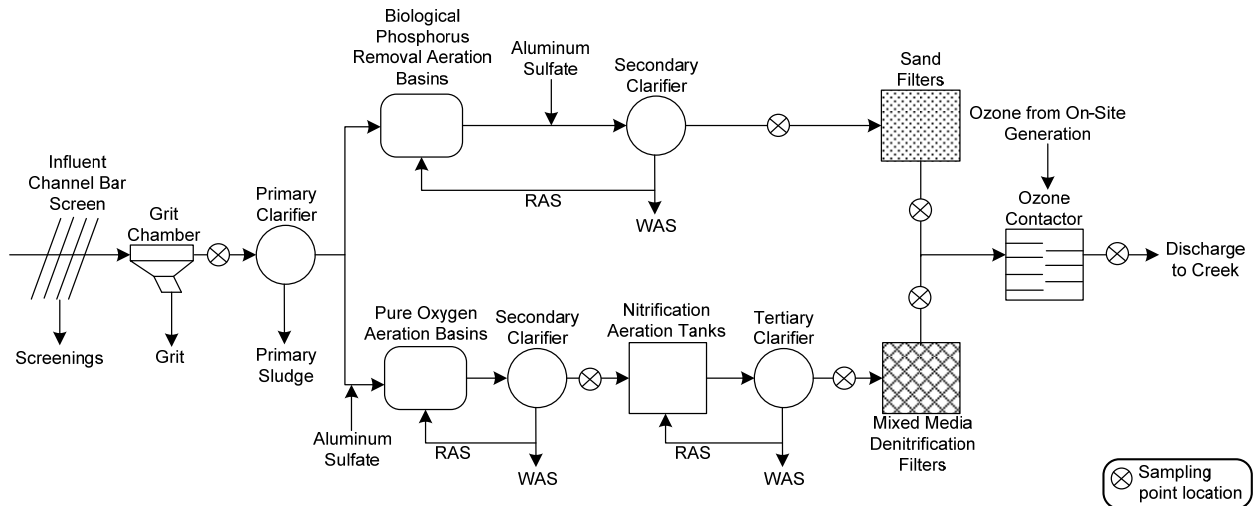


Figure 4-4. Process Schematic of Plant D

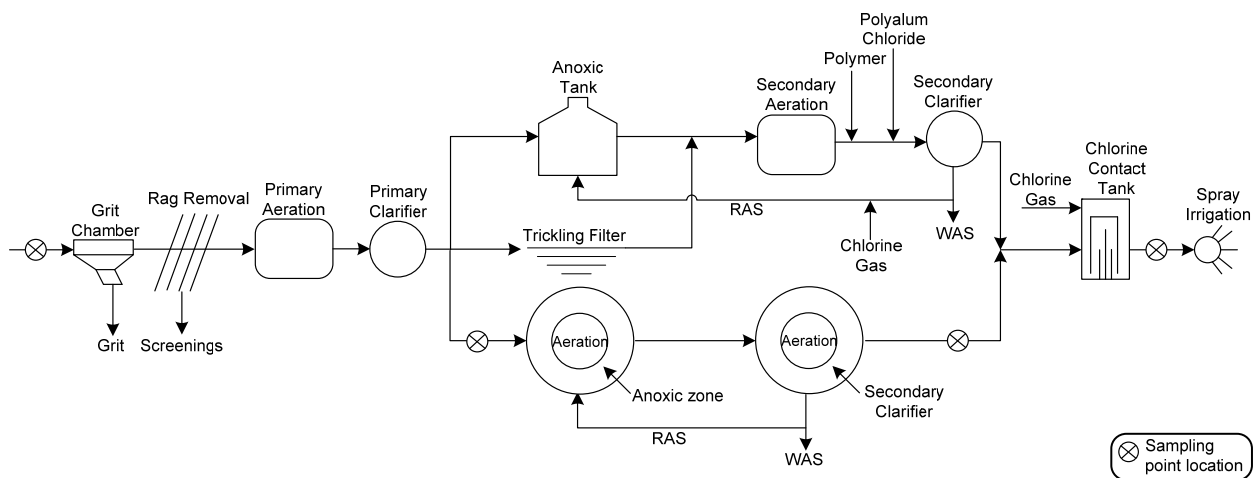


Figure 4-5. Process Schematic of Plant E

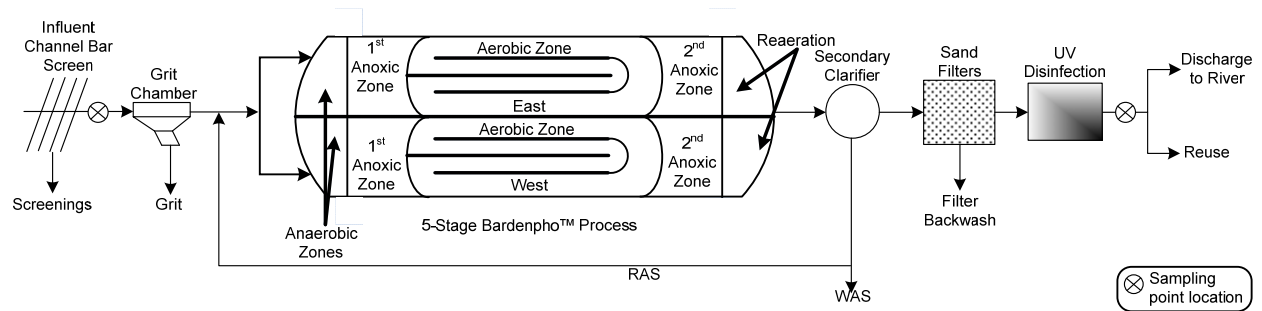


Figure 4-6. Process Schematic of Plant F

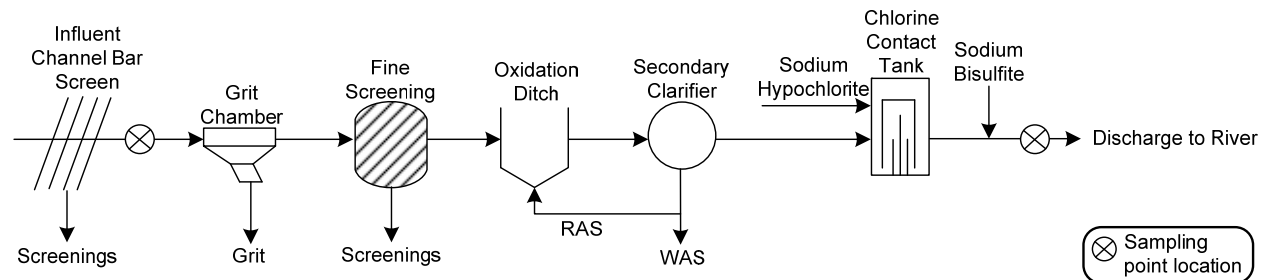


Figure 4-7. Process Schematic of Plant G

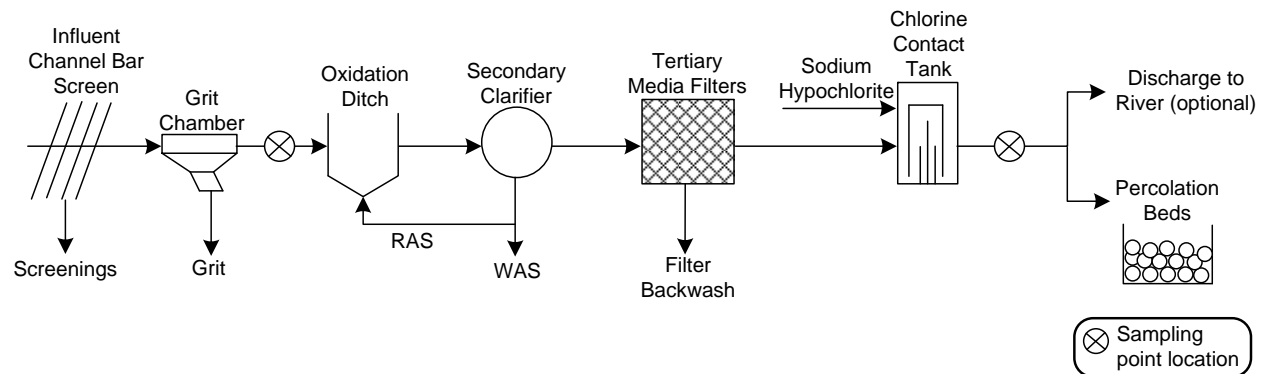


Figure 4-8. Process Schematic of Plant H

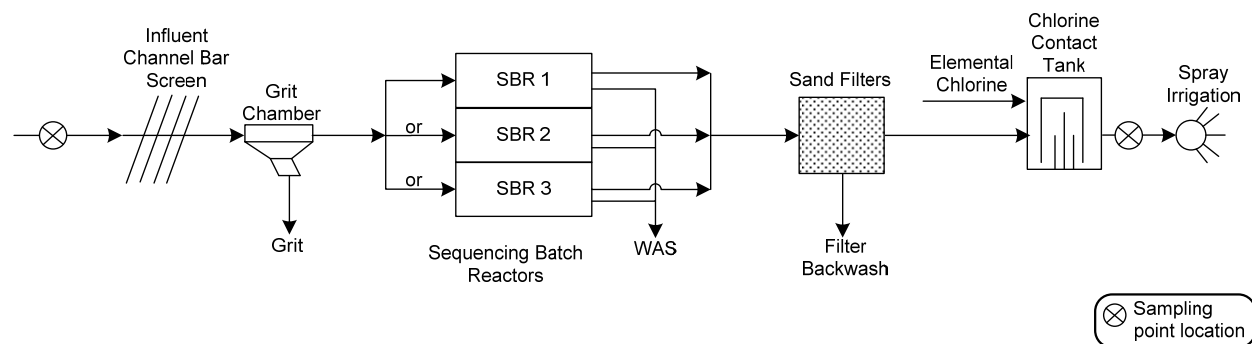


Figure 4-9. Process Schematic of Plant I

4.1.2 Sampling Procedures

EPA collected samples over a 24-hour period at Plants A through F, as one-time grab samples at Plants G and H, and as multiple-hour manual grab composites at Plant I.

24-Hour Sampling Episodes

At Plants A, B, C, D, E, and F, EPA collected samples over a 24-hour sampling period. Samples were collected using either an automatic compositor or a grab sampling bottle dipper. A composite sample is a mixture of discrete samples collected over a specific period of time (e.g., 24 hours) intended to represent the characteristics of the flow at the sampling point over the entire sampling period.

To collect a representative sample of the varying influent flow to POTWs, EPA used one of the following techniques:

- Time-weighted sampling: aliquots collected at 15-minute intervals;
- Flow-weighted sampling: automatic compositors connected to flow meters, aliquots collected after predetermined amount of flow passed; or
- Manual time-/flow-weighted sampling: typical flow pattern determined, aliquots collected at intervals ranging from 10 to 20 minutes based on high-, medium-, and low-flow periods.

Time-weighted sampling. At Plant A, automatic compositors were programmed to collect sample aliquots every 15 minutes throughout the entire 24-hour sampling period. The time-based schedule was intended to reflect the changing wastewater stream as flow changed.

Flow-weighted sampling. Because the time-based schedule used at Plant A did not seem to reflect periods of high flow, EPA used a flow-weighted sampling strategy at Plant B. Automatic compositors collected sample aliquots when a preset volume of sample passed through the influent channel, as signaled by a flow meter. However, the flow meters used at POTW B were sometimes unreliable, and the signal was not always constant.

Manual time-/flow-weighted sampling. For sampling at Plants C, D, E, and F, EPA used historical daily flow data and trends to create a manual time-/flow-weighted sampling scheme. A larger volume of sample was collected during periods of higher flow. This sampling technique required EPA to adjust the sampling interval on the automatic compositors four times within the 24-hour sampling period. Sampling aliquot intervals were calculated prior to the sampling episode. When possible, time-/flow-weighted sampling intervals were adjusted on site, as updated flow data were provided by POTW personnel.

The automatic compositors dispensed samples into multiple 10-liter jars over the 24-hour sampling period. The contents of the multiple jars were poured into a large 15-gallon plastic container lined with Teflon® bag(s). The sample was mixed with a Teflon® stirring rod and pumped into the sample containers using a peristaltic pump.

Because automatic sampling equipment could interfere with analyte recoveries for some of the non-CEC methods, a subset of these samples were collected directly into sample containers. Grab samples were collected four times (at 6-hour intervals) during the 24-hour sample collection period using a bottle dipper. The grab samples were physically composited by the analytical laboratory prior to analysis.

One-Time Grab Sampling Episodes

EPA changed from composite sampling to grab sample collection at Plants G and H. Grab sampling was based on the premise that CECs concentrations, like the BOD₅ concentrations, peak with the flow. EPA decided it could identify POTWs with high influent concentrations of CECs by collecting one-time grab samples during periods of peak flow.

EPA identified the peak POTW flow periods (the one to two hours during which POTW flow was at its highest) to get the highest percentage of human derived waste in the influent. The one-time grab samples were collected directly into the sample containers using a bottle dipper when the plant influent flow was at its peak.

Manual Grab Composite Episodes

At Plant I, EPA collected time-weighted, manual grab composite samples at the influent and effluent. Because Plant I operates SBRs and effluent flow is not constant, EPA chose not to collect one-time grab samples similar to POTWs G and H. Influent and effluent samples were collected as time-weighted, manual composite aliquots over one SBR fill period and decant period, respectively. The sampled SBR fill period occurred during the peak POTW flow period.

At the influent, the manual composite samples were collected into a sample bottle from a sample tap then poured into a 10-L composite sample jar. At the end of the compositing period, the mixed sample was poured from the 10-L jar directly into sample bottles. At the effluent, EPA used a bottle dipper to collect sample aliquots into a sample bottle. These bottle aliquots were poured into two 10-L composite sample jars. At the end of the compositing period, mixed sample was poured directly from the 10-L jars into sample bottles. EPA's sample collection procedures are summarized in Table 4-2.

Table 4-2. Sample Collection Procedures

Plant	Influent	Intermediate Sampling Point(s)	Final Effluent	Sludge
A	Time-weighted 24-hour composite and 4-interval grab	None	Time-weighted 24-hour composite and 4-interval grab	One-time grab
B	Flow-weighted 24-hour composite and 4-interval grab	Flow-weighted 24-hour composite and 4-interval grab	Flow-weighted 24-hour composite and 4-interval grab	One-time grab
C	Time-/flow-weighted 24-hour composite and 4-interval grab	Time-/flow-weighted 24-hour composite and 4-interval grab	Time-/flow-weighted 24-hour composite and 4-interval grab	One-time grab
D	Time-/flow-weighted 24-hour composite and 4-interval grab	5 time-/flow-weighted 24-hour composites and 4-interval grabs	Time-/flow-weighted 24-hour composite and 4-interval grab	One-time grab
E	Time-/flow-weighted 24-hour composite and 4-interval grab	2 time-/flow-weighted 24-hour composites and 4-interval grabs	Time-/flow-weighted 24-hour composite and 4-interval grab	None
F	Time-/flow-weighted 24-hour composite and 4-interval grab	None	Time-/flow-weighted 24-hour composite and 4-interval grab	None
G	One-time grab	None	One-time grab	None
H	One-time grab	None	One-time grab	None
I	Time-weighted, 3-hour grab composites	None	Time-weighted, 40-minute grab composites	None

4.2 Quality Assurance

EPA wrote a Quality Assurance Project Plan (QAPP) to document the project-specific policies, organization, objectives, functional activities, and specific quality assurance/quality control (QA/QC) procedures for the Nine POTW Study. EPA developed the initial QAPP at the start of Stage 1 (QAPP Versions 1 and 2), and revised it prior to the start of Stage 2 sampling (QAPP Version 3). The QAPP complies with *EPA Requirements for QA Project Plans (QA/R-5)* (EPA, 2001) and presents the criteria used to review the sampling data collected in the Study.

4.3 Quality Control Procedures

Quality assurance/quality control (QA/QC) procedures applicable to the nine sampling episodes are outlined in the *Quality Assurance Project Plan for the POTW Study* (ERG, 2006; ERG, 2008). QC practices include collecting and analyzing the following:

- Equipment blanks;
- Field duplicates;
- Laboratory QC volumes; and/or
- Trip blanks.

4.3.1 Equipment Blanks

To assess contamination from sampling equipment in the field, EPA collected equipment blanks for the automatic compositors and bottle dippers used at each sampling episode.

For the automatic compositor, EPA collected an equipment blank by pumping Ultra Pure Blank Water DI+™ through the entire automatic compositor system directly into sample containers; the automatic compositor system included tubing, a 15-gallon plastic tank lined with Teflon® bag(s), a Teflon® stir rod, and, in some cases, a strainer. The tubing was purged prior to the collection of equipment blanks. EPA also used Ultra Pure Blank Water DI+™ to collect bottle dipper equipment blanks by placing the sample container into the bottle dipper apparatus as is done during field sampling and pouring the Ultra Pure Blank Water DI+™ directly into the sample containers.

EPA analyzed equipment blanks for select analytes during each sampling episode. Equipment blanks collected at Plants A, B, C, and D were analyzed for semivolatile organic compounds and metals. Equipment blanks at Plants E, F, G, H, and I were analyzed for the CECs target analytes. EPA compared the concentrations of CECs detected in the equipment blanks to the concentrations of CECs detected in the associated samples. See Appendix C for details of how EPA adjusted the sample results based on the significance of blank contamination.

4.3.2 Field Duplicates

EPA collected duplicate samples to assess the variability in sample collection, handling, preparation, and analysis. Duplicate samples were collected from the same location, at the same time, and stored and analyzed independently.

EPA collected field duplicates using separate automatic compositors or duplicate grab samples collected at the same time as other grab samples. Field duplicates were not collected at Plants A, D, G, H, and I. Field duplicates collected at Plants B, C, E, and F were analyzed for all CECs target analytes. Results of analysis of duplicate samples have been averaged for presentation in Appendix C of this report.

4.3.3 Laboratory Quality Control Volumes

As part of standard laboratory QC, matrix effects on analytical performance are assessed through the analysis of matrix spikes and laboratory duplicates. For nonisotope dilution procedures, these analyses are conducted on 10 percent of the samples from a given matrix (e.g., aqueous, sludge) within a sampling event. Laboratory QC volumes were taken from composite volumes collected and poured into separate sample bottles at the same time as sample aliquots were prepared, or they were collected as grab samples.

EPA collected laboratory QC volumes for at least some of the CECs target analytes at all nine POTWs.

4.3.4 Trip Blanks

At some POTWs, trip blanks were collected and analyzed for select analytes to evaluate possible contamination during shipment and handling of samples. Sample containers were filled

with Ultra Pure Blank Water DI+™ and shipped with empty sample containers to the POTW and then with the collected samples to the analytical laboratories. Trip blanks collected at Plants A, B, C, and D were analyzed for volatile organic compounds. Trip blanks collected for Plants E and F were analyzed for alkylphenols, APEs, and BPA.

4.3.5 Quality Control Sample Summary

Samplers were to collect one equipment blank for every 10 samples collected, but no less than one per sampling episode. EPA collected 19 total equipment blanks for 31 total samples in the Nine POTW Study, and met the QAPP QC requirements.

In addition, samplers were to collect one duplicate sample per 10 samples collected for the program. EPA collected 10 duplicate samples for the 31 total samples, and met the QAPP requirements.

Table 4-3 lists the QC samples collected for each target analyte family at each POTW sampled during the program.

Table 4-3. QC Samples at each POTW for each CECs Analyte Family

Analyte Family	Plant								
	A	B	C	D	E	F	G	H	I
PPCPs	MS/MSD	MS/MSD EfDup	MS/MSD EfDup	MS/MSD	EfDup EEBlank InDup IEBlank	EfDup EEBlank InDup IEBlank	EEBlank	EEBlank	EEBlank
Steroids and Hormones	MS/MSD	MS/MSD EfDup	MS/MSD EfDup	MS/MSD	EfDup EEBlank InDup IEBlank	EfDup EEBlank InDup IEBlank	EEBlank	EEBlank	EEBlank
Alkylphenols, APEs, and BPA		EfDup	EfDup		MS/MSD EfDup EEBlank InDup IEBlank Trip B	MS/MSD EfDup EEBlank InDup IEBlank Trip B	MS/MSD EEBlank	MS/MSD EEBlank	MS/MSD EEBlank
PBDEs		InDup	InDup		EfDup InDup	EfDup InDup	EEBlank	EEBlank	EEBlank
Pesticides		MS/MSD InDup	MS/MSD InDup	MS/MSD	MS/MSD EfDup EEBlank InDup IEBlank	MS/MSD EfDup EEBlank InDup IEBlank	MS/MSD EEBlank	MS/MSD EEBlank	MS/MSD EEBlank

MS/MSD – Matrix spike/matrix spike duplicate.

EfDup – Effluent duplicate.

EEBlank – Effluent equipment blank.

InDup – Influent duplicate.

IEBlank – Influent equipment blank.

Trip B – Trip blank.

4.4 Sample Collection, Preservation, and Storage

Analytical methods used to measure pollutants in the wastewater samples collected in this study typically include instructions for collecting, preserving, and storing wastewater samples. These specifications are designed to prevent degradation and loss of the target analytes. Several

methods direct samplers to maintain samples at a certain temperature or to treat samples with preserving chemicals. For example organic analytes containing chlorine are preserved with sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). Dissolved thiosulfate converts hypochlorite to chloride ion and prevents oxidation of the organic analytes in the sample. For the CECs analytical methods, preservation and storage requirements and recommendations are still under development.

Because EPA was continuing to develop analytical methods during the Nine POTW Study, sample preservation and storage specifications were in flux. The methods used by EPA to detect PPCPs, steroids/hormones and pesticides suggest some of the target analytes may degrade rapidly in aqueous samples. The methods recommended beginning sample extraction within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation (especially if extracting within 48 hours is not practical), in which case samples should be extracted within 48 hours of removal from the freezer. Samples should arrive at the laboratory at $\leq 6^\circ\text{C}$.

Both methods used to analyze for nonylphenols and alkylphenol ethoxolates required that samples analyzed for alkylphenols, APEs, and BPA be stored between 0 and 4°C from the time of collection to extraction. Samples must be extracted within 28 days of collection and completely analyzed within 40 days of extraction. All samples met the hold times for extraction and analysis. The laboratory did not report sample temperatures upon receipt.

EPA Method 1614 does not specify holding times associated with PBDEs in aqueous sample matrices. The method notes that, if stored in the dark at $< 6^\circ\text{C}$, aqueous samples may be stored for up to one year.

The following tables present the sample collection, preservation, and storage conditions used for samples analyzed for CECs:

- Table 4-4: PPCPs;
- Table 4-5: Steroids and Hormones;
- Table 4-6: Alkylphenols, APEs, and BPA;
- Table 4-7: PBDEs; and
- Table 4-8: Pesticides and Herbicides.

Table 4-4. PPCPs: Sample Collection, Preservation, and Storage

Plant	A	B	C	D	E	F	G	H	I
Method	AXYS MLA-052 Rev. 2	AXYS MLA-052 Rev. 2	AXYS MLA-052 Rev. 2	AXYS MLA-052 Rev. 2	EPA 1694	EPA 1694	EPA 1694	EPA 1694	EPA 1694
Sample Container for Collection	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L plastic with Teflon lid	1-L plastic with Teflon lid	1-L amber plastic with Teflon lid	1-L amber plastic with Teflon lid	1-L silanized, amber glass with Teflon lid
Field Preservation	Cool, 0-4°C	Cool, 4°C	Cool, 4°C	Cool, 4°C	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present
Temperature Upon Receipt at Lab	3-9°C	9 – 12°C	5-11°C,	12-14°C	2°C	2°C	4°C	11°C	5°C
Lab Preservation	NH ₄ -EDTA	None	None	None	None	None	None	None	None
Storage Procedure	Sample transferred to 4-L bottle to prevent breakage and stored frozen	Refrigerated	Refrigerated	4°C	Stored frozen for 50 days prior to extraction	Stored frozen for 45 days prior to extraction	Stored frozen for 5 days prior to extraction	Not frozen; extracted upon receipt	UNK
Time from Collection to Extraction	UNK	Samples extracted 12 days after receipt	Samples extracted 3 days after receipt	Samples extracted 3 times, 70 days 89 days 98 days after collection	Samples were extracted within 48 hours of thaw	Samples were extracted within 48 hours of thaw	Samples extracted immediately after thawing	Samples in transit 5 days before extract; no lab storage	Samples were extracted two days after collection
Time from Extraction to Analysis	UNK	7 to 29 days	1 to 6 days	5 to 43 days	UNK	UNK	UNK	UNK	Within method-specified time

UNK – Unknown. Not reported by laboratory or not recorded in data review narrative.

^a For Plants E and F, EPA received verbal confirmation from the laboratory that samples arrived at or below 6°C.

Table 4-5. Steroids and Hormones: Sample Collection, Preservation, and Storage

Plant	A	B	C	D	E	F	G	H	I
Method	AXYS MLA-057 Rev. 01	AXYS MLA-057 Rev. 01	AXYS MLA-057 Rev. 01	AXYS MLA-057 Rev. 01	EPA 1698	EPA 1698	EPA 1698	EPA 1698	EPA 1698
Sample Container	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L plastic with Teflon lid	1-L plastic with Teflon lid	1-L amber plastic with Teflon lid	1-L amber plastic with Teflon lid	1-L silanized, amber glass with Teflon lid
Field Preservation	No headspace; cool, 4°C	Cool, 4°C	Cool, 4°C	Cool, 4°C	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present
Temperature Upon Receipt at Lab	3-9°C	9 – 14°C	9 – 11°C	15 – 16°C	2°C	2°C	3°C	16°C	4°C
Storage Procedure	Stored frozen	UNK	UNK	UNK	Stored frozen	Stored frozen	Stored at -20°C	Stored at -20°C	UNK
Time from Collection to Extraction	5 months	34 days	4–5 days	60 days	57 days (extracted within 48 hours after thaw)	52 days (extracted within 48 hours after thaw)	Within method-specified time	Within method-specified time	Within contract-specified time
Time from Extraction to Analysis	≤40 days	18 days	8–17 days	8 days	UNK	UNK	Within method-specified time	Within method-specified time	Within contract-specified time

UNK – Unknown. Not reported by laboratory or not recorded in data review narrative.

Table 4-6. Alkylphenols, APEs, and BPA: Sample Collection, Preservation, and Storage

Plant	A	B	C	D	E	F	G	H	I
Method	CRL-MS004	CRL-MS004	CRL-MS004	CRL-MS004	CRL-MS004; ASTM D 7076-06 ^a	CRL-MS004; ASTM D 7076-06 ^a	CRL-MS004; ASTM D 7076-06 ^a	CRL-MS004; ASTM D 7076-06 ^a	CRL-MS004; ASTM D 7076-06 ^a
Sample Container	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid
Field Preservation	Cool, 4°C; HCl to pH <2	Cool, 4°C; H ₂ SO ₄ to pH <2	Cool, 4°C; H ₂ SO ₄ to pH <2	Cool, 4°C; H ₂ SO ₄ to pH <2	Cool, ≤6°C; H ₂ SO ₄ to pH <2	Cool, ≤6°C; H ₂ SO ₄ to pH <2	Cool, ≤6°C; H ₂ SO ₄ to pH <2	Cool, ≤6°C; H ₂ SO ₄ to pH <2	Cool, ≤6°C; H ₂ SO ₄ to pH <2
Temperature Upon Receipt at Lab	UNK	UNK	UNK	UNK	UNK	UNK	UNK	UNK	UNK
Storage Procedure	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	UNK
Time from Collection to Extraction	Within the 28-day hold time	Within the 28-day hold time	Within method-specified time	Within method-specified time	Within method-specified time	Within method-specified time	All arbitrary hold times were met	All arbitrary hold times were met	7 days
Time from Extraction to Analysis	Within 45 days of extraction	Within the 40-day hold time	Within method-specified time	Within the 40-day hold time	Within the 40-day hold time	Within the 40-day hold time	All arbitrary hold times were met. It has been shown in cases where re-extraction was done, the concentration does not change noticeably within a 2-4 week period if preserved properly.	All arbitrary hold times were met. It has been shown in cases where re-extraction was done, the concentration does not change noticeably within a 2-4 week period if preserved properly.	19 days

UNK – Unknown. Not reported by laboratory or not recorded in data review narrative.

^a ASTM D 7076-06 used on selected samples.

Table 4-7. PBDEs: Sample Collection, Preservation, and Storage

Plant	A	B	C	D	E	F	G	H	I
Method	EPA 1614 draft	EPA 1614 draft	EPA 1614 draft	EPA 1614 draft	EPA 1614	EPA 1614	EPA 1614	EPA 1614	EPA 1614
Sample Container	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid
Field Preservation	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present;	Cool, 0-6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present
Temperature Upon Receipt at Lab	UNK	9-14°C	8-11°C	14°C	3°C	4°C	3°C	11°C	UNK
Storage Procedure	No storage required; extracted and analyzed immediately at lab	No storage required; extracted and analyzed immediately at lab	No storage required; extracted and analyzed immediately at lab	No storage required; extracted and analyzed immediately at lab	Stored frozen	Stored frozen	UNK	UNK	UNK
Time from Collection to Extraction	Within method-specified time	Within method-specified time	UNK	UNK	63 days (extracted within 48 hours of thaw)	63 days (extracted within 48 hours of thaw)	Within contract-specified time	Within contract-specified time	Within contract-specified time
Time from Extraction to Analysis	Within method-specified time	Within method-specified time	UNK	UNK	UNK	UNK	Within contract-specified time	Within contract-specified time	Within contract-specified time

UNK – Unknown. Not reported by laboratory or not recorded in data review narrative.

Table 4-8. Pesticides: Sample Collection, Preservation, and Storage

Plant	A	B	C	D	E	F	G	H	I
Method	AXYS MLA035 and MLA 037	AXYS MLA035 and MLA 037	AXYS MLA035 and MLA 037	AXYS MLA035 and MLA 037	EPA 1699	EPA 1699	EPA 1699	EPA 1699	EPA 1699
Sample Container	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber glass with Teflon lid	1-L amber plastic with Teflon lid	1-L amber plastic with Teflon lid	1-L amber glass with Teflon lid
Field Preservation	Cool, 0-4°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, 4°C	Cool, 4°C	Cool, 4°C	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present	Cool, ≤6°C; 80 mg/L (8 mL/L) Na ₂ S ₂ O ₃ when chlorine was present
Temperature Upon Receipt at Lab	3-9°C	9-14°C	3-8°C	14-16°C	3°C	1°C	2°C	12°C	3-5°C
Storage Procedure	Transferred to 4L glass jar and stored frozen	4°C	4°C	4°C	Stored frozen; extracted within 48 hr of thaw	Stored frozen; extracted within 48 hr of thaw	4°C	4°C	UNK
Time from Collection to Extraction	Approx 6 months	7 days	3 days	35 days	56-58 days after collection	53-55 days after collection ^a	Within method-specified time	Within method-specified time	UNK
Time from Extraction to Analysis	Less than 40 days	8 days	8 days	26 days	UNK	UNK	Within 40 days of extraction	Within 40 days of extraction ^b	UNK

UNK – Unknown. Not reported by laboratory or not recorded in data review narrative.

^a Sample 71322 reanalysis extracted 76 days after collection.

^b Sample 71470: heptachlor, dacthal, simazine, hexazinone, and chlorpyrifos re-analyzed diluted 50 days past extraction.

5. SUMMARY OF OCCURRENCE DATA

This section summarizes the occurrence of CECs in POTW influent and effluent samples collected in this study. With each analytical method the laboratory could detect, but not necessarily quantify, several CECs. Results for which an analyte was detected but failed to meet method QC specifications are reported as DET. No numerical value is associated with detected (DET) results. Numerical values are associated with quantifiable results, which are CECs detected at concentrations greater than the laboratory's reporting limits, and for which there were no laboratory or data quality problems.

Several classes of CECs were detected and quantified in POTW influents. Quantifiable amounts of PPCPs, sterols, alkylphenol ethoxylates (APEs), flame retardants (PBDEs), and pesticides were detected in treated effluent. Hormones and BPA were not quantified in any effluent sample.

Table 5-1 presents the analytes detected and quantified in the Nine POTW Study and the percent of sampled POTWs in which they were detected or quantified in influent and effluent. Because of the ongoing development of the analytical methods, not all of the same CECs were analyzed for in every POTW. Thus, the denominator in the tables can vary from analyte to analyte.

The remainder of this section is divided into subsections for the following CECs analyte classes:

- PPCPs;
- Sterols and hormones;
- Alkylphenols, APEs, and BPA;
- PBDEs; and
- Pesticides.

These subsections present the compounds detected in POTW influent and effluent samples. These subsections also present tables comparing the compounds detected and quantified in the influent and effluent based on the general difference between the concentrations quantified. Analytes are placed into one of the three following categories:

1. Detected or quantified in at least one influent sample and no effluent samples;
2. Quantified² in at least one influent sample and one or more effluent samples at concentrations at least one order of magnitude less than at least one influent sample; and
3. Quantified³ in at least one influent sample and one or more effluent samples at concentrations similar to influent samples.

^{2,3} Only analytes quantified in both influent and effluent samples are included in these comparisons. For example, the pharmaceutical albuterol was quantified in five influent samples and detected but failed to meet QC specifications (DET) in effluent samples. Albuterol is not included in Table 5-2 which compares pharmaceutical influent and effluent concentrations.

For example, the pharmaceutical acetaminophen, which was detected in all influent samples and no effluent samples, was placed in the first category. The pharmaceutical ciprofloxacin was quantified in five influent samples, one of which was 15,100 ng/L, and three effluent samples, one of which was 36.7 ng/L. Ciprofloxacin was placed in the second category. EPA cautions that this influent/effluent comparison is not based on the difference between influent/effluent pairs, and is presented only to indicate the CECs for which concentrations in the effluent were less than those in the influent. EPA did not design these sampling episodes with the intention of determining treatment effectiveness of the POTWs, nor were sludge samples analyzed for this study. Consequently, the extent that the concentration differences represent biological and/or chemical degradation versus partitioning to sludges is not known. More information on the analytical methods used in this study is in Appendix B.

5.1 PPCPs

Of the 72 PPCPs in the Nine POTW Study, 44 were detected in at least one sample of POTW influent collected. Of the 44 PPCPs detected, 27 were in 75% or more of the samples in which the particular PPCP was analyzed (see Table 5-1).

In POTW effluent samples, 33 PPCPs were detected in at least one sample. Of the 33 PPCPs detected, 16 were detected in less than 25% of the effluent samples analyzed for the PPCP (see Table 5-1).

Thirteen PPCPs were detected or quantified in at least one influent sample and no effluent samples. Thirteen PPCPs were quantified in at least one influent sample and one or more effluent samples at concentrations at least one order of magnitude less than influent samples. Seven PPCPs were quantified in at least one influent sample and one or more effluent samples at concentrations similar to influent samples. Table 5-2 compares the PPCPs detected in POTW influent and effluent samples.

Table 5-1. Analytes Detected in POTW Influent and Effluent

Analyte	Percent Occurrence in Sampled POTW Influent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)	Percent Occurrence in Sampled POTW Effluent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)
PPCPs: Antibiotics		
4-Epitetracycline (ETC)	100% (5 of 5)	0% (0 of 5)
Chlorotetracycline (CTC)	11% (1 of 9)	11% (1 of 9)
Doxycycline	67% (6 of 9)	11% (1 of 9)
Minocycline	20% (1 of 5)	0% (0 of 5)
Tetracycline (TC)	78% (7 of 9)	11% (1 of 9)
Sulfadiazine	20% (1 of 5)	20% (1 of 5)
Sulfadimethoxine	25% (2 of 8)	13% (1 of 8)
Sulfamerazine	50% (4 of 8)	0% (0 of 8)
Sulfamethazine	38% (3 of 8)	13% (1 of 8)
Sulfamethizole	13% (1 of 8)	13% (1 of 8)
Sulfamethoxazole	100% (8 of 8)	88% (7 of 8)
Sulfathiazole	25% (2 of 8)	0% (0 of 8)
Ciprofloxacin	78% (7 of 9)	44% (4 of 9)
Clarithromycin	100% (5 of 5)	60% (3 of 5)
Erythromycin	89% (8 of 9)	56% (5 of 9)
Ofloxacin	100% (5 of 5)	20% (1 of 5)
Tylosin	0% (0 of 9)	11% (1 of 9)
Azithromycin	100% (5 of 5)	40% (2 of 5)
Cefotaxime	0% (0 of 5)	20% (1 of 5)
Cloxacillin	20% (1 of 5)	0% (0 of 5)
Lincomycin	56% (5 of 9)	22% (2 of 9)
Penicillin V	40% (2 of 5)	0% (0 of 5)
Trimethoprim	100% (9 of 9)	33% (3 of 9)
Virginiamycin	22% (2 of 9)	0% (0 of 9)
PPCPs: Group 1, other than Antibiotics		
1,7-Dimethyl xanthine	100% (9 of 9)	11% (1 of 9)
Acetaminophen	100% (9 of 9)	0% (0 of 9)
Caffeine	100% (9 of 9)	0% (0 of 9)
Carbamazepine	100% (5 of 5)	80% (4 of 5)
Codeine	63% (5 of 8)	13% (1 of 8)
Cotinine	89% (8 of 9)	33% (3 of 9)
Dehydronifedipine	80% (4 of 5)	60% (3 of 5)
Diltiazem	100% (9 of 9)	44% (4 of 9)
Diphenhydramine	60% (3 of 5)	40% (2 of 5)
Fluoxetine	78% (7 of 9)	56% (5 of 9)

Table 5-1 (Continued)

Analyte	Percent Occurrence in Sampled POTW Influent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)	Percent Occurrence in Sampled POTW Effluent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)
Miconazole	100% (5 of 5)	0% (0 of 5)
Thiabendazole	80% (4 of 5)	80% (4 of 5)
PPCPs: Group 3		
Gemfibrozil	100% (9 of 9)	78% (7 of 9)
Ibuprofen	100% (9 of 9)	0% (0 of 9)
Naproxen	100% (5 of 5)	20% (1 of 5)
Triclocarban	100% (5 of 5)	80% (4 of 5)
Triclosan	100% (5 of 5)	0% (0 of 5)
Warfarin	44% (4 of 9)	0% (0 of 9)
PPCPs: Group 4		
Albuterol	67% (6 of 9)	22% (2 of 9)
Cimetidine	100% (9 of 9)	22% (2 of 9)
Metformin	88% (7 of 8)	88% (7 of 8)
Ranitidine	100% (8 of 8)	25% (2 of 8)
Sterols		
Beta Sitosterol	100% (9 of 9)	44% (4 of 9)
Beta Stigmastanol	100% (5 of 5)	40% (2 of 5)
Campesterol	100% (5 of 5)	40% (2 of 5)
Cholestanol	100% (9 of 9)	78% (7 of 9)
Cholesterol	100% (9 of 9)	67% (6 of 9)
Coprostanol	100% (9 of 9)	89% (8 of 9)
Desmosterol	100% (9 of 9)	44% (4 of 9)
Epicoprostanol	100% (9 of 9)	67% (6 of 9)
Ergosterol	88% (7 of 8)	50% (4 of 8)
Stigmasterol	100% (9 of 9)	67% (6 of 9)
Hormones		
17 Alpha Estradiol	22% (2 of 9)	0% (0 of 9)
17 Alpha-Dihydroequilin	22% (2 of 9)	0% (0 of 9)
17 Beta Estradiol	33% (3 of 9)	0% (0 of 9)
Alpha-Zearalanol	50% (2 of 4)	0% (0 of 4)
Androstenedione	60% (3 of 5)	0% (0 of 5)
Androsterone	100% (5 of 5)	0% (0 of 5)
Beta Estradiol 3-Benzoate	33% (3 of 9)	0% (0 of 9)
Equilin	22% (2 of 9)	0% (0 of 9)
Estriol	100% (5 of 5)	0% (0 of 5)
Estrone	56% (5 of 9)	0% (0 of 9)
Mestranol	11% (1 of 9)	0% (0 of 9)

Table 5-1 (Continued)

Analyte	Percent Occurrence in Sampled POTW Influent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)	Percent Occurrence in Sampled POTW Effluent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)
Norethindrone	33% (3 of 9)	0% (0 of 9)
Norgestrel	22% (2 of 9)	0% (0 of 9)
Progesterone	20% (1 of 5)	0% (0 of 5)
Testosterone	89% (8 of 9)	0% (0 of 9)
Alkylphenols and APEs		
Nonylphenol Diethoxylates (NP2EO)	22% (2 of 9)	0% (0 of 8)
Nonylphenol Monoethoxylates (NP1EO)	22% (2 of 9)	11% (1 of 9)
Octylphenol (OC)	44% (4 of 9)	0% (0 of 9)
Nonlyphenols (NP)	100% (9 of 9)	11% (1 of 9)
PBDEs		
PBDE-28+PBDE-33	100% (9 of 9)	44% (4 of 9)
PBDE-47	100% (9 of 9)	89% (8 of 9)
PBDE-99	100% (9 of 9)	89% (8 of 9)
PBDE-100	100% (9 of 9)	78% (7 of 9)
PBDE-153	100% (9 of 9)	67% (6 of 9)
PBDE-154	100% (9 of 9)	56% (5 of 9)
PBDE-183	100% (9 of 9)	22% (2 of 9)
PBDE-209	100% (8 of 8)	33% (3 of 9)
Pesticides: Organochlorine		
2,4'-DDD	22% (2 of 9)	0% (0 of 9)
2,4'-DDT	11% (1 of 9)	0% (0 of 9)
4,4'-DDD	22% (2 of 9)	0% (0 of 9)
4,4'-DDE	89% (8 of 9)	0% (0 of 9)
4,4'-DDT	22% (2 of 9)	0% (0 of 9)
Aldrin	11% (1 of 9)	0% (0 of 9)
Alpha-BHC	11% (1 of 9)	0% (0 of 9)
Alpha-chlordane	89% (8 of 9)	0% (0 of 9)
Beta-BHC	11% (1 of 9)	0% (0 of 9)
Chlorothalonil	17% (1 of 6)	0% (0 of 7)
Cis-Nonachlor	33% (3 of 9)	0% (0 of 9)
Dieldrin	89% (8 of 9)	56% (5 of 9)
Endosulphan I	11% (1 of 9)	11% (1 of 9)
Endosulfan sulfate	0% (0 of 9)	11% (1 of 9)
Gamma-BHC	44% (4 of 9)	33% (3 of 9)
Gamma-chlordane	89% (8 of 9)	11% (1 of 9)
Heptachlor	11% (1 of 9)	0% (0 of 9)
Heptachlor Epoxide	44% (4 of 9)	11% (1 of 9)

Table 5-1 (Continued)

Analyte	Percent Occurrence in Sampled POTW Influent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)	Percent Occurrence in Sampled POTW Effluent (Number of POTWs at which analyte was detected of Number of POTWs for which analyte was analyzed ^a)
Hexachlorobenzene	25% (2 of 8)	0% (0 of 8)
Methoxychlor	11% (1 of 9)	44% (4 of 9)
Perthane	33% (3 of 9)	0% (0 of 9)
Trans-Nonachlor	78% (7 of 9)	0% (0 of 9)
Pesticides: Organophosphorus		
Chlorpyrifos	67% (6 of 9)	0% (0 of 8)
Chlorpyrifos-oxon	11% (1 of 9)	22% (2 of 9)
Diazinon	67% (6 of 9)	56% (5 of 9)
Diazinon oxon	0% (0 of 9)	22% (2 of 9)
Disulfoton sulfone	11% (1 of 9)	0% (0 of 9)
Malathion	22% (2 of 9)	0% (0 of 9)
Methamidophos	11% (1 of 9)	11% (1 of 9)
Methyl-chlorpyrifos	11% (1 of 9)	0% (0 of 9)
Methyl-parathion	11% (1 of 9)	0% (0 of 9)
Pesticides: Pyrethroid		
Cis-Permethrin	80% (4 of 5)	0% (0 of 5)
Cypermethrins	78% (7 of 9)	0% (0 of 9)
Permethrin	89% (8 of 9)	22% (2 of 9)
Trans-Permethrin	100% (5 of 5)	0% (0 of 5)
Pesticides: Triazine		
Atrazine	89% (8 of 9)	100% (9 of 9)
Cyanazine	0% (0 of 9)	11% (1 of 9)
Desethyl atrazine	89% (8 of 9)	89% (8 of 9)
Hexazinone	11% (1 of 9)	22% (2 of 9)
Metribuzin	0% (0 of 9)	22% (2 of 9)
Simazine	44% (4 of 9)	56% (5 of 9)

^a Does not include excluded results. Includes detected (DET) and quantified results. See Appendix C.

Table 5-2. Comparison of PPCPs Detected in POTW Influent and Effluent^{b,c}

Detected in at Least One Influent Sample <u>AND</u>	Quantified in at Least One Influent Sample <u>AND</u>	
No Effluent Samples	One or More Effluent Samples at Concentrations at Least One Order of Magnitude Less than at Least One Influent Sample	One or More Effluent Samples at Concentrations Similar to Influent Samples
Antibiotics		
4-Epitetracycline (ETC) Minocycline ^a Sulfamerazine Sulfathiazole Cloxacillin ^a Penicillin V ^a Virginiamycin ^a	Doxycycline Sulfamethoxazole Ciprofloxacin Clarithromycin Erythromycin-Total Ofloxacin	Chlorotetracycline (CTC) Tetracycline (TC) Sulfadiazine Trimethoprim
Analytical Group 1, other than Antibiotics		
Acetaminophen Caffeine Miconazole	Cotinine	Carbamazepine Fluoxetine Thiabendazole
Analytical Group 3		
Ibuprofen Triclosan Warfarin ^a	Gemfibrozil Naproxen Triclocarban	None
Analytical Group 4		
None	Cimetidine Metformin Ranitidine	None

^a All influent results for which the analyte was detected are DET.

^b Tylosin and cefotaxime were detected in POTW effluent and no samples of POTW influent. They are not included on this table.

^c Eleven pharmaceuticals were detected in at least one influent and one effluent sample but were not quantified in both influent and effluent samples. Consequently, their influent and effluent concentrations could not be compared and they are not included on this table.

Many of the PPCPs laboratory results failed to meet the QC specifications of the analytical method. Of the 441 results for which the concentration was reported above the laboratory-reported detection limit, 54 percent of the results were not qualified in any way and are considered accurate. The remaining 46 percent of the results were reported only as detected (DET). No numerical value should be attached to these results because of poor spike recovery or other laboratory problems. See Table 5-3 for additional statistics on detections and quantifications for PPCP samples.

Table 5-3. Numbers of PPCPs Detected in POTW Influent and Effluent

Analyte Group	Number of Analytes	Number of Analytes Detected in at Least One Sample of					
		POTW Influent			POTW Effluent		
		DET ^a	Quantified ^b	Total Detected	DET ^a	Quantified ^b	Total Detected
Antibiotics	47	6	16	22	5	12	17
Group 1, other than antibiotics	15	1	11	12	4	5	9
Group 3	6	1	5	6	0	3	3
Group 4	4	0	4	4	1	3	4
Total	72	8	36	44	10	23	33

^a Analytes for which all POTW influent/effluent sample results were DET.

^b At least one sample of POTW influent/effluent was detected with no QC issues.

5.2 Sterols and Hormones

All evaluated sterols were detected in all POTW influent samples³. Sterols were detected in fewer samples of POTW effluent than influent, but all evaluated sterols were detected in at least one sample of effluent (see Table 5-1).

Concentrations of all evaluated sterols were much lower in the POTW effluent samples than in the influent samples (see Table 5-4).

Hormones were detected in fewer POTW influent samples than sterols, and when detected, the concentrations of hormones were typically much lower than concentrations of sterols. Evaluated hormones were not detected in any effluent samples (see Tables 5-4).

³ With the exception of one analyte (ergosterol) at one plant.

Table 5-4. Comparison of Sterols and Hormones Detected in POTW Influent and Effluent

Detected in at Least One Influent Sample <u>AND</u>	Quantified in at Least One Influent Sample <u>AND</u>	
	One or More Effluent Samples at Concentrations at Least One Order of Magnitude Less than at Least One Influent Sample	One or More Effluent Samples at Concentrations Similar to Influent Samples
Sterols^b		
None	Beta Sitosterol Beta Stigmastanol Campesterol Cholestanol Cholesterol Coprostanol Desmosterol Epicoprostanol Stigmasterol	None
Hormones		
17 Alpha-Estradiol ^a 17 Alpha-Dihydroequilin ^a 17 Beta-Estradiol ^a Alpha-Zearalanol ^a Androstenedione Androsterone Beta-Estradiol-3-Benzoate ^a Equilin Estriol Estrone Mestranol ^a Norethindrone Norgestrel ^a Progesterone Testosterone	None	None

^a All influent results for which the analyte was detected are DET.

^b Ergosterol was quantified in at least one influent sample but was not quantified in effluent samples. Consequently its influent and effluent concentrations could not be compared and it is not included on this table.

Many of the sterol and hormone analytical results failed to meet method QC specifications. Of the 240 results for which the concentration was reported above the laboratory-reported detection limit, 58 percent of the results were not qualified in any way and are considered accurate. The remaining 42 percent of the results were reported only as detected (DET). No numerical value should be attached to these results because of poor spike recovery or other laboratory problems. See Table 5-5 for additional statistics on detections and quantifications of Steroid and Hormone samples.

Table 5-5. Numbers of Sterols and Hormones Detected in POTW Influent and Effluent

Analyte Group	Number of Analytes	Number of Analytes Detected in at Least One Sample of					
		POTW Influent			POTW Effluent		
		DET ^a	Quantified ^b	Total Detected	DET ^a	Quantified ^b	Total Detected
Sterols	10	0	10	10	1	9	10
Hormones	17	6	9	15	0	0	0

^a Analytes for which all POTW influent/effluent sample results were DET.

^b At least one sample of POTW influent/effluent was detected with no QC issues.

5.3 Alkylphenols, APEs, and BPA

BPA was not detected in the influent or effluent of any sampled plant. NP was detected in each plant influent, additionally, the reporting limits for NP were significantly lower than the EPA Water Quality Criteria of 6,600 ng/L. Moreover, nonlyphenol monoethoxylates (NP1EO), nonlyphenol diethoxylates (NP2EO), and octylphenol (OP) were occasionally detected. OP and NP2EO were never detected in the effluent (see Tables 5-6).

Table 5-6. Comparison of Alkylphenols, APEs, and BPA Detected in POTW Influent and Effluent

Detected in at Least One Influent Sample <u>AND</u>	Quantified in at Least One Influent Sample <u>AND</u>	
No Effluent Samples	One or More Effluent Samples at Concentrations at Least One Order of Magnitude Less than at Least One Influent Sample	One or More Effluent Samples at Concentrations Similar to Influent Samples
OP NP2EO	NP1EO NP	None

Some of the analytical results failed to meet method QC specifications. Of the 31 results for which the concentration was reported above the laboratory-reported detection limit, 65 percent of the results were not qualified in any way and are considered accurate. The remaining 35 percent of the results were reported only as detected (DET). No numerical value should be attached to these results because of poor spike recovery or other laboratory problems. See Table 5-7 for additional statistics on detections and quantifications of alkylphenols, APEs and BPA.

Table 5-7. Numbers of Alkylphenols, APEs, and BPA Detected in POTW Influent and Effluent

Analyte Group	Number of Analytes	Number of Analytes Detected in at Least One Sample of					
		POTW Influent			POTW Effluent		
		DET ^a	Quantified ^b	Total Detected	DET ^a	Quantified ^b	Total Detected
Alkylphenols, Alkylphenol Ethoxylates (APEs), and Bisphenol A	5	0	4	4	0	2	2

^a Analytes for which all POTW influent/effluent sample results were DET.

^b At least one sample of POTW influent/effluent was detected with no QC issues.

5.4 PBDEs

There are 209 PBDE congeners. For the Nine POTW Study, results were reported for nine PBDEs of interest (EPA, 2007a). Note that PBDE-28 and PBDE-33 are treated as one analyte in this report because the analytical laboratory reported results for PBDE-28 + PBDE-33.

All PBDE congeners evaluated in this study were detected in all POTW influent samples. The concentrations of the analyzed congeners are very similar plant to plant, despite the differences in the populations served by the POTWs. Concentrations of all congeners of interest are much lower in the samples of POTW effluent than in samples of POTW influent (Table 5-8).

Table 5-8. Comparison of PBDEs Detected in POTW Influent and Effluent

Detected in at Least One Influent Sample <u>AND</u>	Quantified in at Least One Influent Sample <u>AND</u>	
No Effluent Samples	One or More Effluent Samples at Concentrations at Least One Order of Magnitude Less than at Least One Influent Sample	One or More Effluent Samples at Concentrations Similar to Influent Samples
None	PBDE-28 + PBDE-33 PBDE-47 PBDE-99 PBDE-100 PBDE-153 PBDE-154 PBDE-183 PBDE-209	None

The concentration of PBDE-209 in preparation blanks was often greater than the method quantitation levels (EPA Method 1614 quantitation level of 2 ppt). Because PBDE-209 sample results for POTW effluents are close to the method quantitation level, for effluent samples from four plants, it was impossible to determine if PBDE-209 detected in the effluent was the result of contamination. Consequently, the effluent sample concentrations were reported as not-quantified at the quantitation level, adjusted for sample size. See Appendix C.

A relatively small percentage of the PBDEs analytical results failed to meet method QC specifications in comparison to some of the other CECs analyte families. Of the 156 results for which the concentration was reported above the laboratory-reported detection limit, 87 percent of the results were not qualified in any way and are considered accurate. The remaining 13 percent of the results were reported only as detected (DET). No numerical value should be attached to these results because of poor spike recovery or other laboratory problems. See Table 5-9 for additional statistics on detections and quantifications of PBDEs.

Table 5-9. Numbers of PBDEs Detected in POTW Influent and Effluent

Analyte Group	Number of Analytes	Number of Analytes Detected in at Least One Sample of					
		POTW Influent			POTW Effluent		
		DET ^a	Quantified ^b	Total Detected	DET ^a	Quantified ^b	Total Detected
PBDEs	8	0	8	8	0	8	8

^a Analytes for which all POTW influent/effluent sample results were DET.

^b At least one sample of POTW influent/effluent was detected with no QC issues.

5.5 Pesticides

Of the 62 pesticides, 37 were detected in at least one sample of POTW influent collected in this study. Eighteen (18) pesticides were detected in at least one sample of POTW effluent (Table 5-1).

Twenty-three (23) pesticides were detected or quantified in at least one influent sample and no effluent samples. Five pesticides were quantified in at least one influent sample and one or more effluent samples at concentrations at least one order of magnitude less than influent samples. Five pesticides were quantified in at least one influent sample and one or more effluent samples at concentrations similar to influent samples. Table 5-10 compares the pesticides detected in POTW influent and effluent samples.

Table 5-10. Comparison of Pesticides Detected in POTW Influent and Effluent^{b,c}

Detected in at Least One Influent Sample <u>AND</u>	Quantified in at Least One Influent Sample <u>AND</u>	
	One or More Effluent Samples at Concentrations at Least One Order of Magnitude Less than at Least One Influent Sample	One or More Effluent Samples at Concentrations Similar to Influent Samples
Organochlorine		
2,4'-DDD 2,4'-DDT 4,4'-DDD 4,4'-DDE 4,4'-DDT Aldrin Alpha-BHC Alpha-chlordane Beta-BHC Chlorothalonil Cis-nonachlor Heptachlor ^a Hexachlorobenzene Perthane Trans-nonachlor	Dieldrin Gamma-chlordane	Gamma-BHC Heptachlor Epoxide
Organophosphorus		
Chlorpyrifos Disulfoton sulfone ^a Malathion Methyl-chlorpyrifos ^a Methyl-parathion	Diazinon	None
Pyrethroid		
Cis-Permethrin Cypermethrins Trans-Permethrin	Permethrin (sum of cis and trans)	None
Triazine		
	Desethyl atrazine	Atrazine Hexazinone Simazine

^a All influent results for which the analyte was detected are DET.

^b Endosulfan sulfate, diazinon oxon, metribuzin, and cyanazine were detected in POTW effluent and no samples of POTW influent. They are not included on this table.

^c Endosulfan I, metholxychlor, chlorpyrophos oxon, and methamidophos were detected in at least one influent and one effluent sample. They were not quantified in both influent and effluent samples, however. As a result, their influent and effluent concentrations could not be compared and they are not included on this table.

A relatively small percentage of the pesticides analytical results failed to meet method QC specifications in comparison to some of the other CECs analyte families. Of the 238 results for which the concentration was reported above the laboratory-reported detection limit, 81 percent of the results were not qualified in any way and are considered accurate. The remaining 19 percent of the results were reported only as detected (DET). No numerical value should be attached to these results because of poor spike recovery or other laboratory problems. See Table 5-11 for additional statistics on detections and quantifications of pesticides.

Table 5-11. Numbers of Pesticides Detected in POTW Influent and Effluent

Analyte Group	Number of Analytes	Number of Analytes Detected in at Least One Sample of					
		POTW Influent			POTW Effluent		
		DET ^a	Quantified ^b	Total Detected	DET ^a	Quantified ^b	Total Detected
Organochlorine	34	3	18	21	0	7	7
Organophosphorus	17	4	4	8	1	3	4
Pyrethroid	4	0	4	4	0	1	1
Triazine	7	0	4	4	1	5	6
Total	62	7	30	37	2	16	18

^a Analytes for which all POTW influent/effluent sample results were DET.

^b At least one sample of POTW influent/effluent was detected with no QC issues.

6. REFERENCES

- ASTM, 2006. ASTM International. Standard Test Method for Determination of Nonylphenol, Bisphenol A, p-tert-Octylphenol, Nonylphenol Monoethoxylate and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass Spectrometry, ASTM D7065 – 06. 2006.
- Census, 2007. U.S. Census Bureau. School Enrolment, Table A-5a. The Population 14 to 24 Years Old by High School Graduate Status, College Enrollment, Attainment, Sex, Race, and Hispanic Origin: October 1967 to 2005. <http://www.census.gov/population/socdemo/school/TableA-5a.xls>. Accessed 16 October 2007.
- Chandra, 2005. Chandra A, G.M. Martinez, W.D. Mosher, J.C. Abma, J. Jones. “Fertility, Family Planning, and Reproductive Health of U.S. Women: Data from the 2002 National Survey of Family Growth.” National Center for Health Statistics. *Vital and Health Statistics* 23 (25). http://www.cdc.gov/nchs/products/pubs/pubd/series/sr23/pre-1/sr23_25.htm. Accessed 15 October 2007.
- EPA, 1982. U.S. Environmental Protection Agency. *Fate of Priority Pollutants in Publicly Owned Treatment Works*. EPA 440/1-82/303. September 1982.
- EPA, 2001. U.S. Environmental Protection Agency. EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5. March 2001. <http://www.epa.gov/QUALITY/qs-docs/r5-final.pdf>. Accessed 13 November 2008.
- EPA, 2007a. U.S. Environmental Protection Agency, Office of Science and Technology. Method 1614: Brominated Diphenyl Ethers in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA 821-R-07-005. August 2007.
- EPA, 2007b. U.S. Environmental Protection Agency, Office of Science and Technology. Method 1694: Pharmaceuticals and Personal Care Products in Water, Soil, Sediment, and Biosolids by HPLC/MS/MS, EPA 821-R-08-002. December 2007.
- EPA, 2007c. U.S. Environmental Protection Agency, Office of Science and Technology. Method 1698: Steroids and Hormones in Water, Soil, Sediment, and Biosolids by HRGC/HRMS, EPA 821-R-08-003. December 2007.
- EPA, 2007d. U.S. Environmental Protection Agency, Office of Science and Technology. Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, EPA 821-R-08-001. December 2007.
- ERG, 2006. Eastern Research Group, Inc. Quality Assurance Project Plan for the POTW Study. 19 May 2006.
- ERG, 2008. Eastern Research Group, Inc. Quality Assurance Project Plan for the POTW Study. 13 June 2008.

- Kaufman, 2002. D.W. Kaufman, J.P. Kelly, L. Rosenberg, T.E. Anderson, A.A. Mitchell. "Recent patterns of medication use in the ambulatory adult population of the United States: The Slone Survey." *JAMA* 2002; 287:337-44.
- Metcalf and Eddy, 1978. Metcalf and Eddy, Inc. *Wastewater Engineering: Treatment/Disposal/Reuse*, Second Edition. Boston: McGraw Hill. 1978.
- Slone, 2006. Slone Center for Epidemiology at Boston University. *Patterns of Medication Use in The United States, 2006: A Report from the Slone Survey*. <http://www.bu.edu/slone/SloneSurvey/SloneSurvey.htm>. Accessed 15 October 2007.

Appendix A

CONTAMINANTS OF EMERGING CONCERN (CECs) ANALYTE LIST

Table A-1. PPCP Analyte List

Analyte	Classification	AXYS Method MLA-052 (Plants A,B,C,D)	EPA Method 1694 (Plants E,F,G,H,I)
Antibiotics			
4-Epianhydrochlortetracycline (EACTC)	Chlorotetracycline degradate		x
4-Epianhydrotetracycline (EATC)	Chlorotetracycline degradate		x
4-Epichlortetracycline (ECTC)	Chlorotetracycline degradate		x
4-Epioxytetracycline (EOTC)	Oxytetracycline degradate		x
4-Epitetracycline (ETC)	Tetracycline degradate		x
Anhydrochlortetracycline (ACTC)	Chlorotetracycline degradate		x
Anhydrotetracycline (ATC)	Chlorotetracycline degradate		x
Chlorotetracycline (CTC)	Tetracycline antibiotic	x	x
Demeclocycline	Tetracycline antibiotic		x
Doxycycline	Tetracycline antibiotic	x	x
Isochlortetracycline (ICTC)	Chlorotetracycline degradate		x
Minocycline	Tetracycline antibiotic		x
Oxytetracycline (OTC)	Tetracycline antibiotic	x	x
Tetracycline	Tetracycline antibiotic	x	x
Sulfachloropyridazine	Sulfonamide antibiotic	x	x
Sulfadiazine	Sulfonamide antibiotic		x
Sulfadimethoxine	Sulfonamide antibiotic	x	x
Sulfamerazine	Sulfonamide antibiotic	x	x
Sulfamethazine	Sulfonamide antibiotic	x	x
Sulfamethizole	Sulfonamide antibiotic	x	x
Sulfamethoxazole	Sulfonamide antibiotic	x	x
Sulfanilamide	Sulfonamide antibiotic		x
Sulfathiazole	Sulfonamide antibiotic	x	x
Ciprofloxacin	Fluoroquinolone antibiotic	x	x
Clarithromycin	Macrolide antibiotic		x
Clinafloxacin	Quinoline antibiotic		x
Enrofloxacin	Fluoroquinolone antibiotic	x	x
Erythromycin	Macrolide antibiotic	x	x
Lomefloxacin	Quinoline antibiotic		x
Norfloxacin	Quinoline antibiotic	x	x
Ofloxacin	Quinoline antibiotic		x
Roxithromycin	Macrolide antibiotic	x	x
Sarafloxacin	Fluoroquinolone antibiotic	x	x
Tylosin	Macrolide antibiotic	x	x
Ampicillin	β -lactam antibiotic	a	a
Azithromycin	Macrolide antibiotic		x

Table A-1 (Continued)

Analyte	Classification	AXYS Method MLA-052 (Plants A,B,C,D)	EPA Method 1694 (Plants E,F,G,H,I)
Carbadox	Quinoxaline antibiotic	x	x
Cefotaxime	Cephalosporin antibiotic		x
Cloxacillin	β -lactam antibiotic		x
Flumequine	Quinolone antibiotic		x
Lincomycin	Lincosamide antibiotic	x	x
Ormetoprim	Macrolide antibiotic		x
Oxacillin	β -lactam antibiotic		x
Oxolinic acid	Quinolone antibiotic		x
Penicillin G	β -lactam antibiotic		x
Penicillin V	β -lactam antibiotic		x
Trimethoprim	Pyrimidine antibiotic	x	x
Virginiamycin	Macrolide antibiotic	x	x
Analytical Group 1, other than antibiotics			
1,7-Dimethylxanthine	Antispasmodic, caffeine metabolite	x	x
Acetaminophen	Antipyretic, analgesic	x	x
Caffeine	Stimulant	x	x
Carbamazepine	Anticonvulsant		x
Codeine	Opiate	x ^b	x
Cotinine	Nicotine metabolite	x	x
Dehydronifedipine	Nifedipine metabolite		x
Digoxigenin	Immunohistochemical marker steroid	x	x
Digoxin	Cardiac glycoside	x	x
Diltiazem	Antihypertensive	x	x
Diphenhydramine	Antihistamine		x
Fluoxetine	SSRI antidepressant	x	x
Miconazole	Antifungal agent		x
Norgestimate	Hormonal contraceptives	x ^b	x
Thiabendazole	Fungicide and parasiticide		x
Analytical Group 3			
Gemfibrozil	Antilipemic	x	x
Ibuprofen	Analgesic	x	x
Naproxen	NSAID		x
Triclocarban	Antimicrobial		x
Triclosan	Antimicrobial		x
Warfarin	Anticoagulant	x	x

Table A-1 (Continued)

Analyte	Classification	AXYS Method MLA-052 (Plants A,B,C,D)	EPA Method 1694 (Plants E,F,G,H,I)
Analytical Group 4			
Albuterol	Antiasthmatic	x	x
Cimetidine	Anti-acid reflux	x	x
Metformin	Anti-diabetic drug	x	x
Ranitidine	Anti-acid reflux	x	x
Total		40	73

^a Although listed in Method 1694, ampicillin was not included in either Stage 1 or Stage 2 of the Nine POTW Study.

^b Codeine and norgestimate were included in AXYS Method MLA-052 for the analysis of Plant B, C, and D samples, but not for Plant A samples.

Note all compounds listed in Table A-1 are pharmaceuticals with the exception of triclosan and triclocarban, both antimicrobial compounds. Samples from Plants E – I were analyzed for triclosan and triclocarban, while samples from Plants A, B, C, and D were not.

Table A-2. Steroids and Hormones Analyte List

Analyte	AXYS Method MLA-057 Rev 01 (Plants A,B,C,D)	EPA Method 1698 (Plants E,F,G,H,I)
Sterols		
Beta Sitosterol	x	x
Beta Stigmastanol		x
Campesterol		x
Cholestanol	x	x
Cholesterol	x	x
Coprostanol	x	x
Desmosterol	x	x
Epi-coprostanol	x	x
Ergosterol	x	x
Stigmasterol	x	x
Hormones		
17 Alpha-Dihydroequilin	x	x
17 Alpha-Estradiol	x	x
17 Alpha-Ethinyl Estradiol ^a		x
17 Beta-Estradiol	x	x
Alpha-Zearalanol	x	
Androstenedione		x
Androsterone		x
Beta-Estradiol-3-Benzoate	x	x
Desogestrel	x ^b	x
Equilenin		x
Equilin	x	x
Estriol		x
Estrone	x	x
Ethinylestradiol ^a	x	
Mestranol	x	x
Norethindrone	x	x
Norgestrel	x	x
Progesterone		x
Testosterone	x	x
Total	21	27

^a Ethinylestradiol and 17 Alpha-Ethinyl Estradiol are synonyms.

^b Desogestrel was included in AXYS Method MLA-057 Rev 01 for the analysis of Plant A samples, but not for Plants B, C, or D.

Table A-3. Alkylphenols, APEs, and BPA Analyte List

Method Number	Full Name	Abbreviated Name
MS004 (Plants A-I) and ASTM Method D 7065-06 (Plants E-I)	Nonylphenols ^a	NP
	Nonylphenol Monoethoxylates ^a	NP1EO
	Nonylphenol Diethoxylates ^a	NP2EO
	Octylphenol	OP
	Bisphenol A	BPA

^a Nonylphenols, nonylphenol monoethoxylates, and nonylphenol diethoxylates are mixtures of branched isomers. The concentrations reported are the totals for the isomer mixtures.

Table A-4. PBDE Analyte List (Plants A-I)

Full Name	Abbreviated Name	Number
2,4,4'-Tribromodiphenyl ether plus 2',3,4-Tribromodiphenyl ether	2,4,4'-TrBDE plus 2',3,4-TrBDE	PBDE-28 + PBDE-33 ^a
2,2',4,4'- Tetrabromodiphenyl ether	2,2',4,4'-TeBDE	PBDE-47
2,2',4,4',5- Pentabromodiphenyl ether	2,2',4,4',5-PeBDE	PBDE-99
2,2',4,4',6-Pentabromodiphenyl ether	2,2',4,4',6-PeBDE	PBDE-100
2,2',4,4',5,5'- Hexabromodiphenyl ether	2,2',4,4',5,5'-HxBDE	PBDE-153
2,2',4,4',5',6- Heptabromodiphenyl ether	2,2',4,4',5',6-HxBDE	PBDE-154
2,2',3,4,4',5',6- Heptabromodiphenyl ether	2,2',3,4,4',5',6-HpBDE	PBDE-183
Decabromodiphenyl ether	DeBDE	PBDE-209

^a PBDE 28 and PBDE 33 have the same retention time on the DB-5HT gas chromatography column and cannot be quantified separately. For this reason, the concentration of PBDE 28 + PBDE 33 presented in this report represents the total concentration for these two TrBDE congeners.

Table A-5. Pesticides Analyte List

Analyte	AXYS Method MLA-035 Rev 04 Plant A	AXYS Method MLA-035 Rev 04 Plants B- D	EPA Method 1699 Plants E - I
Organochlorine			
2,4'-DDD	x	x	x
2,4'-DDE	x	x	x
2,4'-DDT	x	x	x
4,4'-DDD	x	x	x
4,4'-DDE	x	x	x
4,4'-DDT	x	x	x
Aldrin	x	x	x
Alpha-BHC	x	x	x
Alpha-chlordane	x	x	x
Beta-BHC	x	x	x
Captan	x	x	x
Chlorothalonil	x	x	x
Cis-nonachlor	x	x	x
Dacthal	x	x	x
Delta-BHC	x	x	x
Dieldrin	x	x	x
Endosulfan I	x	x	x
Endosulfan II	x	x	x
Endosulfan sulfate	x	x	x
Endrin	x	x	x
Endrin Ketone	x	x	x
Gamma-BHC	x	x	x
Gamma-chlordane	x	x	x
Heptachlor	x	x	x
Heptachlor Epoxide	x	x	x
Hexachlorobenzene	x	x	x
Methoxychlor	x	x	x
Mirex	x	x	x
Octachlorostyrene	x	x	x
Oxychlordane	x	x	x
Pentachloronitrobenzene	x	x	x
Perthane	x	x	x
Tecnazene	x	x	x
Trans-nonachlor	x	x	x
Organophosphorus			
Azinphos-methyl	x	x	x
Chlorpyrifos	x	x	x
Chlorpyrifos-oxon	x	x	x

Table A-5 (Continued)

Analyte	AXYS Method MLA-035 Rev 04 Plant A	AXYS Method MLA-035 Rev 04 Plants B- D	EPA Method 1699 Plants E - I
Diazinon	X	X	X
Diazinon-oxon	X	X	X
Disulfoton	X	X	X
Disulfoton sulfone	X	X	X
Ethyl-parathion	X	X	X
Fenitrothion	X	X	X
Fonofos	X	X	X
Malathion	X	X	X
Methamidophos	X	X	X
Methyl-chlorpyriphos	X	X	X
Methyl-parathion	X	X	X
Phorate	X	X	X
Phosmet	X	X	X
Pirimiphos-methyl	X	X	X
Pyrethroid			
Cis-permethrin			X
Cypermethrins	X	X	X
Permethrin	X	X	X
Trans-permethrin			X
Triazine			
Ametryn	X	X	X
Atrazine	X	X	X
Cyanazine	X	X	X
Desethyl atrazine	X	X	X
Hexazinone	X	X	X
Metribuzin	X	X	X
Simazine	X	X	X
Other Compounds (not included in EPA Method 1699)			
Alachlor	X	X	
Benzonitrile, 3,5-Dibromo-4-Hydroxy-	X		
Butralin	X	X	
Butylate	X	X	
Dimethenamid	X	X	
Dimethoate	X	X	
Ethalfuralin	X	X	
Ethion	X	X	
Fluazifop	X		
Flufenacet	X	X	
Flutriafol	X	X	
Linuron	X	X	
MCOA		X	

Table A-5 (Continued)

Analyte	AXYS Method MLA-035 Rev 04 Plant A	AXYS Method MLA-035 Rev 04 Plants B- D	EPA Method 1699 Plants E - I
Methoprene	x	x	
Metolachlor	x	x	
Pendamethalin	x	x	
Tebuconazol	x	x	
Terbufos	x	x	
Triallate	x	x	
Trifluralin	x	x	
Total	79	78	62

Appendix B

ANALYTICAL METHODS OVERVIEW

The analytical methods used to measure CECs in the Nine POTW Study were developed and revised throughout the course of this study. The version of the method used to analyze samples from each plant is listed in Table B-1.

Table B-1 Analytical Methods by POTW

Plant	PPCP Method	S/H Method	Pesticide Method	PBDE Method	APEs Method
A	Laboratory SOP	AXYS Method MLA-057 Rev 01 ^a	AXYS Method MLA-035 Rev. 04 ^a	Draft EPA Method 1614 (August 2003)	CRL-MS004 Rev #2
B	AXYS MLA-052 ^a	AXYS Method MLA-057 Rev 01 ^a	AXYS Method MLA-035 Rev. 04 ^a	Draft EPA Method 1614 (August 2003)	CRL-MS004 Rev #2
C	AXYS MLA-052 ^a	AXYS Method MLA-057 Rev 01 ^a	AXYS Method MLA-035 Rev. 04 ^a	Draft EPA Method 1614 (August 2003)	CRL-MS004 Rev #2
D	AXYS MLA-052 ^a	AXYS Method MLA-057 Rev 01 ^a	AXYS Method MLA-035 Rev. 04 ^a	Draft EPA Method 1614 (August 2003)	CRL-MS004 Rev #2
E	EPA Method 1694	EPA Method 1698	EPA Method 1699	EPA Method 1614 (August 2007)	CRL-MS004 Rev #2 and ASTM Method D 7065-06
F	EPA Method 1694	EPA Method 1698	EPA Method 1699	EPA Method 1614 (August 2007)	CRL-MS004 Rev #2 and ASTM Method D 7065-06
G	EPA Method 1694	EPA Method 1698	EPA Method 1699	EPA Method 1614 (August 2007)	CRL-MS004 Rev #2 and ASTM Method D 7065-06
H	EPA Method 1694	EPA Method 1698	EPA Method 1699	EPA Method 1614 (August 2007)	CRL-MS004 Rev #2 and ASTM Method D 7065-06
I	EPA Method 1694	EPA Method 1698	EPA Method 1699	EPA Method 1614 (August 2007)	CRL-MS004 Rev #2 and ASTM Method D 7065-06

^a AXYS Analytical Services, Sidney, British Columbia, Canada.

Samples collected during Stage 1 of the Study (September 2005 – September 2006) were analyzed with draft methods based on procedures developed at contract laboratories. EPA used the results of these analyses to develop more sensitive and selective analytical methods for three groups of CECs: PPCPs, steroids and hormones, and pesticides. Three new EPA methods resulted from this work (EPA 1694, 1698 and 1699). These methods were designed to screen for a large number of analytes in a broad range of POTW matrices including influent, effluent, and sewage sludge. After single-laboratory validation and peer review, three methods were published in December 2007, and EPA used the published methods to analyze samples collected in Stage 2 of the Nine POTW Study (November 2007 through September 2008).

The PBDE (flame retardants) analytical method (EPA Method 1614; EPA, 2007a) was developed before the Nine POTW Study began. Although EPA Method 1614 was multi-laboratory validated and published in August 2007; EPA made some additional refinements during this study to improve the detection of certain PBDEs.

These four methods (EPA 1614, 1694, 1698 and 1699) are available on the CWA methods website at <http://www.epa.gov/waterscience/methods/method/other.html>, and are listed in Table B-2.

Table B-2. EPA Analytical Methods for CECs

EPA Method Number	Analyte Group	Method Type	Method Status During Stage 1	Date Published
1614	PBDEs	HRGC/HRMS	Under development	August 2007
1694	PPCPs	HPLC/MS/MS	Under development	December 2007
1698	Steroids and Hormones	HRGC/HRMS	Under development	December 2007
1699	Pesticides	HRGC/HRMS	Under development	December 2007

HRGC/HRMS – High resolution gas chromatography/high resolution mass spectrometry.

HPLC/MS/MS – High performance liquid chromatography/tandem mass spectrometry.

In both stages of the Nine POTW Study, EPA analyzed samples of POTW influent and effluent for alkylphenols, APEs, and BPA using methods recently developed by the EPA Region 5 Chicago Regional Laboratory (CRL). During Stage 2, some samples were also analyzed using ASTM Method D 7065-06 (ASTM, 2006), which is based on the EPA Region 5 methods. Table B-3 lists these methods.

Table B-3. CRL Analytical Methods for Alkylphenol-Related Compounds

CRL Method Number	Analyte Group	Method Type	Date Published
MS004	Short chain alkylphenol ethoxylates and bisphenol A	GC/MS-SIM, high-volume injection	May 2007
ASTM D 7065-06	Short chain alkylphenol ethoxylates and bisphenol A	GC/MS-SIM, low-volume injection	December 2006

GC/MS-SIM – Selected Ion Monitoring Gas Chromatography/Mass Spectrometry.

Development of the analytical methods used for these five groups: PPCPs, steroids/hormones, pesticides, PBDEs and APEs are discussed below. Table B-1 lists the version of the method used to analyze samples collected during the Nine POTW Study.

PPCPs

A preliminary laboratory procedure (AXYS Method MLA-052) was used to analyze samples from Plant A. Plants B, C, and D were analyzed using a more refined laboratory procedure (AXYS Method MLA-052). An EPA method was under development at the Axys laboratory during the analysis of samples collected at Plants B, C, and D. After single-laboratory validation and peer review, the method was published as EPA Method 1694 in December 2007 (EPA, 2007b), and samples from Plants E, F, G, H, and I were analyzed using the published method.

EPA Method 1694 is designed to identify 73 PPCPs in POTW wastes. The 73 target analytes in EPA Method 1694 are divided into four groups (1 through 4). Each group represents an LC/MS/MS run. Groups 1, 2, and 3 are extracted under acidic (pH 2) conditions. Groups 1

and 2 are run in the positive electrospray ionization (ESI+) mode, but with differing run profiles. Group 3 is run in the negative electrospray ionization (ESI-) mode. Group 4 is extracted under basic pH 10 conditions and run in the ESI+ mode. Quantitation is achieved using isotope dilution and internal standard techniques.

EPA Method 1694 includes all analytes included in AXYS Method MLA-052 as well as an additional 33 analytes.

In this report, the PPCP analytes are organized by following groups:

1. Antibiotics (Analytical Group 1 antibiotics and Analytical Group 2);
2. Analytical Group 1, other than antibiotics;
3. Analytical Group 3; and
4. Analytical Group 4.

Steroids and Hormones

AXYS Method MLA-057 Rev 1 (a preliminary laboratory procedure) was used to analyze samples from Plants A, B, C, and D; the procedure consisted of low resolution gas chromatography mass spectrometry (GC/MS).

An EPA method was under development at the Axys laboratory during the analysis of samples collected at Plants A through D, and consisted of gas chromatography combined with high resolution mass spectrometry. After single-laboratory validation and peer review, the method was published as EPA Method 1698 (EPA, 2007c) in December 2007. Samples from Plants E, F, G, H, and I were analyzed using the published method.

EPA Method 1698 is designed to identify 27 steroids and hormones in POTW wastes. The method requires solvent extraction of the sample, followed by cleanup with a layered alumina/florisil column. Following cleanup, the target analytes are derivatized and analyzed by GC/HRMS. Quantitation is achieved with isotope dilution and internal standard techniques.

One analyte included in AXYS Method MLA-057 Rev 1 is not included in EPA Method 1698: alpha-zearalanol, an anabolic agent. EPA Method 1698 includes seven analytes not included in AXYS Method MLA-057 Rev. 1.

PBDEs

EPA analyzed samples collected during the Nine POTW Study for 8 PBDEs using EPA Method 1614 which is HRGC/HRMS. Prior to analysis of samples collected at Plants A, B, C, and D the method utilized a 30-meter GC column that, when heated, was suspected to degrade several high-molecular-weight congeners such as congener 209. Subsequent to analysis at Plants A, B, C and D, the method was modified to incorporate a temperature programmable injector (TPI) and a 15-meter short column to mitigate degradation of PBDE 209. After multi-laboratory validation and peer review, Method 1614 was revised and published in August 2007. Samples from Plants E, F, G, H, and I were analyzed using the 2007 version of 1614.

Pesticides

AXYS Method MLA-035 Rev. 04 (a preliminary laboratory procedure) was used to analyze for pesticides in Plants A, B, C, and D. An EPA method was under development at the Axys laboratory during the analysis of samples collected at Plants A, B, C, and D. Subsequent to analysis at Plants A, B, C, and D, organonitrogen pesticides were removed from the method by EPA because they demonstrated thermal instability and poor response under the conditions used in EPA Method 1699.

After single-laboratory validation and peer review, EPA published Method 1699 (EPA, 2007d) in December 2007. Samples from Plants E, F, G, H, and I were analyzed using the published method.

EPA Method 1699 was initially designed to identify 60 pesticides in the following chemical groups:

- 34 Organochlorine Pesticides;
- 17 Organophosphorus Pesticides;
- 7 Triazine Pesticides; and
- 2 Pyrethroid Pesticides.

Method 1699 requires solvent extraction of the sample, followed by cleanup. After cleanup, the target analytes are derivatized and analyzed by GC/HRMS. Quantitation is achieved with isotope dilution and internal standard techniques.

The AXYS Methods MLA-035 Rev. 04 and MLA-037 Rev. 03 analyte lists differed between Plant A and Plants B, C, and D. AXYS Method MLA-035 Rev. 04 includes 20 analytes not included in EPA Method 1699. EPA Method 1699 includes two analytes not included in AXYS Method MLA-035 Rev. 04.

Alkylphenols, APEs, and BPA

EPA analyzed samples for alkylphenols, APEs, and BPA using methods developed by the EPA Region 5 CRL. Certain samples were also analyzed using ASTM Method D 7065-06.

Appendix C

CECs ANALYTICAL RESULTS

This appendix presents the results of EPA's analysis of POTW influent and effluent samples collected during the Nine POTW Study; sludge sampling efforts were dropped after the first stage because the analytical methods needed more development. Tables C-1 through C-10 present the results of sample analysis for the following types of CECs:

- PPCPs;
- Sterols and hormones;
- Alkylphenols, APEs, and BPA;
- PBDEs; and
- Pesticides.

EPA developed Methods 1694, 1698, and 1699 to identify a large number and variety of analytes in three complex matrices, POTW influent, sludge and effluent. It is difficult to optimize analytical procedures for a large number of diverse analytes. Procedures that are optimal for one analyte may be less suitable for other analytes. As a result, many of the analytical results failed to meet method quality control (QC) specifications. For each analyte family, the percentage of the detected results (i.e., where the concentration was reported above the laboratory-reported detection limit) with no QC flags or other data interpretation issues were:

PPCPs	54 percent (441 detected results)
Sterols and hormones	58 percent (240 detected results)
Alkylphenols, APEs, and BPA	65 percent (31 detected results)
PBDEs	87 percent (156 detected results)
Pesticides	81 percent (238 detected results)

The majority of the sampling results were in one of three categories

1. Results for which an analyte was detected with no QC flags or other quality issues are reported as a measured concentration.
2. Results for which an analyte was detected with QC flags or other data interpretation issues are reported as detected (DET) with no numeric concentration. "DET" indicates that the analytes are present in the samples, but that the measured concentration was not considered reliable. Results are qualified because of QC issues including the following:
 - a. Poor spike recoveries including:
 - i. Labeled compound recovery (LCR),
 - ii. Matrix spike (MS) recovery,
 - iii. Initial precision and recovery (IPR), and
 - iv. Ongoing precision and recovery (OPR);
 - v. Continuous calibration verification (CCV);
 - b. Other matrix interference issues;
 - c. Calibration issues; and
 - d. Blank contamination.

3. Results for which an analyte was not detected at or above the laboratory-reported quantitation limit are reported as not quantified (NQ) with the laboratory-reported quantitation limit in parentheses.

Results of analysis of duplicate samples have been averaged for presentation in Tables C-1 through C-10. All results are presented in ng/L (parts per trillion (ppt)). The following designations and footnotes are used throughout the tables:

NA	Not analyzed.
NQ()	Not measured above the, laboratory-reported quantitation level with said level reported in parentheses.
DET	Detected but has associated QC flags
EXCLUDE	Excluded due to critical QC failure including: no/low recovery in matrix spikes and other laboratory spikes; matrix-specific effect on LCR; and no OPR performed.
<	For duplicate samples, an NQ result was averaged with a quantified result.
^a	Sample result set to NQ due to lab blank contamination (sample result less than five times the lab blank result). For duplicate samples, one or both sample results may have been set to NQ. A laboratory-reported detection limit may or may not have been provided.
^b	For duplicate samples, an NQ result was averaged with a DET result.
^c	Sample result set to NQ due to field blank contamination (sample result less than five times the field blank result). For duplicate samples, one or both sample results may have been set to NQ.

Table C-1. Occurrence of PPCPs in POTW Influent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Antibiotics									
4-Epianhydrochlortetracycline (EACTC)	NA	NA	NA	NA	NQ(519)	NQ(472)	NQ(2,860)	NQ(2,890)	NQ(560)
4-Epianhydrotetracycline (EATC)	NA	NA	NA	NA	NQ(130)	NQ(118)	NQ(1,340)	NQ(204)	NQ(140)
4-Epichlortetracycline (ECTC)	NA	NA	NA	NA	NQ(313)	NQ(272)	NQ(194)	NQ(204)	NQ(140)
4-Epioxytetracycline (EOTC)	NA	NA	NA	NA	NQ(51.9)	NQ(104)	NQ(288)	NQ(297)	NQ(189)
4-Epitetracycline (ETC)	NA	NA	NA	NA	DET	DET ^b	412	475	206
Anhydrochlortetracycline (ACTC)	NA	NA	NA	NA	NQ(462)	NQ(464)	NQ(768)	NQ(751)	NQ(140)
Anhydrotetracycline (ATC)	NA	NA	NA	NA	NQ(130)	NQ(118)	NQ(1,070)	NQ(757)	NQ(140)
Chlorotetracycline (CTC)	NQ ^a	434	NQ ^a	NQ(684)	NQ(51.9)	NQ(47.2)	NQ(77.7)	NQ(81.8)	NQ(56.0)
Demeclocycline	NA	NA	NA	NA	NQ(130)	NQ(118)	NQ(194)	NQ(204)	NQ(140)
Doxycycline	NQ ^a	2,970	NQ ^a	2,540	DET	NQ(47.2)	724	341	176
Isochlortetracycline (ICTC)	NA	NA	NA	NA	NQ(51.9)	NQ(47.2)	NQ(77.7)	NQ(81.8)	NQ(56.0)
Minocycline	NA	NA	NA	NA	DET	NQ(1,020)	NQ(3,090)	NQ(3,520)	NQ(2,270)
Oxytetracycline (OTC)	NQ(997)	NQ(169)	NQ ^a	NQ(326)	NQ(51.9)	NQ(47.2)	NQ(77.7)	NQ(81.8)	NQ(56.0)
Tetracycline (TC)	DET	NQ(329)	NQ ^a	234	DET	DET	368	490	231
Sulfachloropyridazine	NQ(59.2)	NQ(16.0)	EXCLUDE	NQ(130)	NQ(13.0)	NQ(11.8)	NQ(19.4)	NQ(20.4)	NQ(14.0)
Sulfadiazine	NA	NA	NA	NA	NQ(13.0)	31.1	NQ(19.4)	NQ(20.4)	NQ(14.0)
Sulfadimethoxine	DET	DET	EXCLUDE	NQ(24.0)	NQ(26.4)	NQ(9.09)	NQ(16.8)	NQ(23.7)	NQ(10.0)
Sulfamerazine	DET	12.5	EXCLUDE	NQ(65.0)	NQ(5.19)	12.6	NQ(7.77)	15.3	NQ(5.60)
Sulfamethazine	DET	DET	EXCLUDE	NQ(26.0)	8.94	NQ(4.72)	NQ(7.77)	NQ(79.0)	NQ(5.60)
Sulfamethizole	DET	NQ(3.00)	EXCLUDE	NQ(24.0)	NQ(5.50)	NQ(4.72)	NQ(7.77)	NQ(8.18)	NQ(5.60)
Sulfamethoxazole	DET	DET	EXCLUDE	1,500	DET	DET	DET	DET	2,620
Sulfanilamide	NA	NA	NA	NA	NQ(276)	NQ(118)	NQ(194)	NQ(204)	NQ(140)
Sulfathiazole	DET	NQ(6.00)	EXCLUDE	NQ(56.0)	NQ(13.0)	NQ(11.8)	NQ(19.4)	212	NQ(14.0)
Ciprofloxacin	DET	NQ(300)	DET	NQ ^a	1,440	803	591	15,100	1,530
Clarithromycin	NA	NA	NA	NA	748	384	DET	DET	292
Clinafloxacin	NA	NA	NA	NA	NQ(123)	NQ(47.2)	NQ(77.7)	NQ(334)	NQ(244)
Enrofloxacin	NQ ^a	NQ(649)	NQ(690)	NQ(331)	NQ(26.0)	NQ(23.6)	NQ(38.9)	NQ(40.9)	NQ(28.0)

Table C-1 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Erythromycin	NQ ^a	126	119	DET	529	586	2,330	280	202
Lomefloxacin	NA	NA	NA	NA	NQ(26.0)	NQ(23.6)	NQ(38.9)	NQ(40.9)	NQ(28.0)
Norfloxacin	NQ(33.8)	NQ(1,270)	NQ(1,350)	NQ(650)	NQ(130)	NQ(353)	NQ(194)	NQ(204)	NQ(140)
Ofloxacin	NA	NA	NA	NA	147	505	429	3,240	DET
Roxithromycin	NQ ^a	NQ(4.00)	NQ(4.00)	NQ(2.00)	NQ(5.50)	NQ(2.36)	NQ(3.89)	NQ(27.0)	NQ(2.80)
Sarafloxacin	NQ(51.2)	NQ(32.0)	NQ ^a	NQ(16.0)	NQ(119)	NQ(403)	NQ(777)	NQ(818)	NQ(560)
Tylosin	NQ(79.7)	NQ(16.0)	NQ(18.0)	NQ(17.0)	NQ(256)	NQ(47.2)	NQ(744)	NQ(2,480)	NQ(661)
Azithromycin	NA	NA	NA	NA	DET	DET	DET	DET	669
Carbadox	NQ(50.0)	NQ(3.00)	NQ(4.00)	NQ(3.00)	NQ(59.1)	NQ(25.7)	NQ(19.4)	NQ(180)	NQ(14.0)
Cefotaxime	NA	NA	NA	NA	NQ(1,240)	NQ(1,150)	NQ(786)	NQ(1,600)	NQ(858)
Cloxacillin	NA	NA	NA	NA	DET b	NQ(123)	NQ(38.9)	NQ(146)	NQ(28.0)
Flumequine	NA	NA	NA	NA	NQ(13.0)	NQ(11.8)	NQ(19.4)	NQ(20.4)	NQ(14.0)
Lincomycin	DET	DET	NQ(89.0)	19.1	NQ(102)	DET	DET	NQ(40.9)	NQ(28.0)
Ormetoprim	NA	NA	NA	NA	NQ(5.19)	NQ(4.72)	NQ(7.77)	NQ(8.18)	NQ(5.60)
Oxacillin	NA	NA	NA	NA	NQ(54.5)	NQ(50.1)	NQ(38.9)	NQ(40.9)	NQ(28.0)
Oxolinic Acid	NA	NA	NA	NA	NQ(21.1)	NQ(10.0)	NQ(7.77)	NQ(8.18)	NQ(5.60)
Penicillin G	NA	NA	NA	NA	NQ(26.0)	NQ(23.6)	NQ(38.9)	NQ(40.9)	NQ(28.0)
Penicillin V	NA	NA	NA	NA	NQ(109)	NQ(47.2)	DET	DET	NQ(56.0)
Trimethoprim	DET	195	498	DET	DET	DET	DET	DET	418
Virginiamycin	DET	NQ(8.00)	NQ(8.00)	NQ(8.00)	DET b	NQ(332)	NQ(205)	NQ(2,300)	NQ(148)
Analytical Group 1, other than antibiotics									
1,7-Dimethyl xanthine	DET	DET	DET	DET	62,600	19,400	DET	DET	4,860
Acetaminophen	DET	DET	DET	DET	79,300	54,400	129,000	340,000	40,200
Caffeine	DET	DET	DET	42,100	68,200	52,500	DET	DET	13,300
Carbamazepine	NA	NA	NA	NA	DET	DET	DET	DET	163
Codeine	NA	NQ(664)	NQ(706)	NQ(904)	DET	DET	DET	DET	345
Cotinine	DET	DET	DET	2,940	535	2,820	NQ(490)	DET	2,980
Dehydronifedipine	NA	NA	NA	NA	DET	DET	DET	DET	NQ(5.60)
Digoxigenin	NQ ^a	NQ(126)	NQ(134)	NQ(134)	NQ(553)	NQ(431)	NQ(605)	NQ(516)	NQ(215)
Digoxin	NQ ^a	NQ(1,330)	NQ(1,410)	NQ(1,410)	NQ(757)	NQ(630)	NQ(194)	NQ(2,530)	NQ(140)
Diltiazem	DET	DET	DET	DET	DET	DET	DET	DET	1,490
Diphenhydramine	NA	NA	NA	NA	DET	DET	NQ(7.77) ^a	NQ(40.9) ^a	1,440
Fluoxetine	DET	NQ(15.0)	DET	DET	24.7	58.7	NQ(38.9)	DET	56.1

C-4

Table C-1 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Miconazole	NA	NA	NA	NA	DET	DET ^b	DET	DET	114
Norgestimate	NA	NQ(16.0)	NQ(17.0)	NQ(8.00)	NQ(78.8)	NQ(104)	NQ(38.9)	NQ(40.9)	NQ(113)
Thiabendazole	NA	NA	NA	NA	NQ(13.0)	12.4	DET	DET	34.0
Analytical Group 3									
Gemfibrozil	DET	DET	DET	1,370	539	4,260	DET	DET	6,630
Ibuprofen	DET	DET	7,360	11,600	18,900	16,800	20,500	DET	13,400
Naproxen	NA	NA	NA	NA	11,300	14,800	DET	DET	18,800
Triclocarban	NA	NA	NA	NA	187	571	4,270	13,700	3,100
Triclosan	NA	NA	NA	NA	996	2,420	4,110	12,000	2,810
Warfarin	NQ(10.6)	DET	NQ(53.0)	DET	NQ(13.0)	NQ(11.8)	NQ(19.4)	DET	DET
Analytical Group 4									
Albuterol	DET	NQ(31.0)	NQ(121)	22.5	6.40	30.6	NQ(24.2)	75.6	68.9
Cimetidine	DET	461	452	227	73.1	330	120	11,700	1,100
Metformin	DET	NQ(326)	EXCLUDE	DET	17,400	29,400	36,500	248,000	11,100
Ranitidine	DET	496	EXCLUDE	DET	1,610	551	1,870	16,800	784

Table C-2. Occurrence of PPCPs in POTW Effluent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Antibiotics									
4-Epianhydrochlortetracycline (EACTC)	NA	NA	NA	NA	NQ(494)	NQ(555)	NQ(1,780)	NQ(1,800)	NQ(502)
4-Epianhydrotetracycline (EATC)	NA	NA	NA	NA	NQ(124)	NQ(342)	NQ(470)	NQ(483)	NQ(125)
4-Epichlortetracycline (ECTC)	NA	NA	NA	NA	NQ(124)	NQ(296)	NQ(125)	NQ(125)	NQ(125)
4-Epioxytetracycline (EOTC)	NA	NA	NA	NA	NQ(49.4)	NQ(120)	NQ(190)	NQ(49.8)	NQ(267)
4-Epitetracycline (ETC)	NA	NA	NA	NA	NQ(49.4)	NQ(206)	NQ(50.0)	NQ(168)	NQ(226)
Anhydrochlortetracycline (ACTC)	NA	NA	NA	NA	NQ(124)	NQ(586)	NQ(465)	NQ(477)	NQ(125)
Anhydrotetracycline (ATC)	NA	NA	NA	NA	NQ(124)	NQ(313)	NQ(510)	NQ(515)	NQ(125)
Chlortetracycline (CTC)	NQ(34.4)	<462 ^a	NQ ^a	NQ(673)	NQ(49.4)	NQ(55.5)	NQ(50.0)	NQ(169)	NQ(50.2)
Demeclocycline	NA	NA	NA	NA	NQ(124)	NQ(139)	NQ(125)	NQ(125)	NQ(125)
Doxycycline	NQ(372) ^a	<284 ^a	NQ ^a	NQ(641)	NQ(49.4)	NQ(55.5)	NQ(50.0)	NQ(49.8)	NQ(50.2)
Isochlortetracycline (ICTC)	NA	NA	NA	NA	NQ(49.4)	NQ(55.5)	NQ(50.0)	NQ(49.8)	NQ(50.2)
Minocycline	NA	NA	NA	NA	NQ(494)	NQ(1,940)	NQ(2,620)	NQ(2,180)	NQ(2,080)
Oxytetracycline (OTC)	NQ(460)	NQ(160)	NQ ^a	NQ(320)	NQ(49.4)	NQ(55.5)	NQ(50.0)	NQ(49.8)	NQ(50.2)
Tetracycline (TC)	NQ(104)	568	NQ ^a	NQ(320)	NQ(49.4)	NQ(55.5)	NQ(50.0)	NQ(49.8)	NQ(50.2)
Sulfachloropyridazine	NQ(5.86)	NQ(14.5)	EXCLUDE	NQ(16.0)	NQ(12.4)	NQ(13.9)	NQ(12.5)	NQ(12.5)	NQ(12.5)
Sulfadiazine	NA	NA	NA	NA	NQ(12.4)	<14.2	NQ(12.5)	NQ(12.5)	NQ(12.5)
Sulfadimethoxine	NQ(0.885)	DET	EXCLUDE	NQ(3.00)	NQ(2.47)	NQ(2.78)	NQ(2.50)	NQ(2.49)	NQ(2.51)
Sulfamerazine	NQ(0.245)	NQ(7.50)	EXCLUDE	NQ(8.00)	NQ(4.94)	NQ(5.55)	NQ(5.00)	NQ(4.98)	NQ(5.02)
Sulfamethazine	NQ(10.6)	DET ^b	EXCLUDE	NQ(3.00)	NQ(4.94)	NQ(5.55)	NQ(5.00)	NQ(19.9)	NQ(5.02)
Sulfamethizole	NQ(1.31)	NQ(3.00)	EXCLUDE	NQ(3.00)	NQ(4.94)	NQ(5.55)	33.5	NQ(4.98)	NQ(5.02)
Sulfamethoxazole	DET	DET	EXCLUDE	9.54	276	1,490	DET	DET	NQ(5.02)
Sulfanilamide	NA	NA	NA	NA	NQ(124)	NQ(139)	NQ(125)	NQ(125)	NQ(125)
Sulfathiazole	NQ(1.52)	NQ(6.00)	EXCLUDE	NQ(7.00)	NQ(12.4)	NQ(13.9)	NQ(12.5)	NQ(12.5)	NQ(12.5)
Ciprofloxacin	NQ ^a	NQ(284)	DET	NQ(301)	36.7	161	NQ(219)	53.0	NQ(20.0)
Clarithromycin	NA	NA	NA	NA	89.7	23.9	DET	NQ(12.5)	NQ(12.5)
Clinafloxacin	NA	NA	NA	NA	NQ(49.4)	NQ(55.5)	NQ(345)	NQ(49.8)	NQ(50.2)
Enrofloxacin	NQ ^a	NQ(614)	NQ(627)	NQ(326)	NQ(24.7)	NQ(27.8)	NQ(100)	NQ(24.9)	NQ(25.1)

Table C-2 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Erythromycin	NQ(4.99) ^a	114	115	NQ(9.00)	167	91.8	418	NQ(2.49)	NQ(2.51)
Lomefloxacin	NA	NA	NA	NA	NQ(24.7)	NQ(27.8)	NQ(25.0)	NQ(24.9)	NQ(25.1)
Norfloxacin	NQ(1.21)	NQ(1,210)	NQ(1,230)	NQ(640)	NQ(124)	NQ(139)	NQ(1,170)	NQ(125)	NQ(125)
Ofloxacin	NA	NA	NA	NA	NQ(12.4)	162	NQ(12.5)	NQ(12.5)	NQ(12.5)
Roxithromycin	NQ(0.203) ^a	NQ(4.00)	NQ(4.00)	NQ(2.00)	NQ(2.47)	NQ(2.78)	NQ(2.50)	NQ(2.49)	NQ(2.51)
Sarafloxacin	NQ(9.80)	NQ(31.0)	NQ ^a	NQ(16.0)	NQ(113)	NQ(290)	NQ(1,060)	NQ(498)	NQ(502)
Tylosin	45.1	NQ(15.5)	NQ(16.0)	NQ(17.0)	NQ(49.4)	NQ(55.5)	NQ(307)	NQ(317)	NQ(50.2)
Azithromycin	NA	NA	NA	NA	DET	DET	NQ(12.5)	NQ(12.5)	NQ(12.5)
Carbadox	NQ(13.5)	NQ(3.00)	NQ(3.00)	NQ(3.00)	NQ(12.4)	NQ(13.9)	NQ(12.5)	NQ(12.5)	NQ(12.5)
Cefotaxime	NA	NA	NA	NA	DET ^b	NQ(846)	NQ(361)	NQ(450)	NQ(698)
Cloxacillin	NA	NA	NA	NA	NQ(93.9)	NQ(100)	NQ(25.0)	NQ(24.9)	NQ(25.1)
Flumequine	NA	NA	NA	NA	NQ(12.4)	NQ(13.9)	NQ(12.5)	NQ(12.5)	NQ(12.5)
Lincomycin	DET	NQ(22.0)	NQ(91.5)	NQ(11.0)	NQ(24.7)	DET	NQ(25.0)	NQ(24.9)	NQ(25.1)
Ormetoprim	NA	NA	NA	NA	NQ(4.94)	NQ(5.55)	NQ(5.00)	NQ(4.98)	NQ(5.02)
Oxacillin	NA	NA	NA	NA	NQ(24.7)	NQ(27.8)	NQ(25.0)	NQ(24.9)	NQ(25.1)
Oxolinic Acid	NA	NA	NA	NA	NQ(10.6)	NQ(5.55)	NQ(5.00)	NQ(4.98)	NQ(5.02)
Penicillin G	NA	NA	NA	NA	NQ(24.7)	NQ(27.8)	NQ(25.0)	NQ(24.9)	NQ(25.1)
Penicillin V	NA	NA	NA	NA	NQ(49.4)	NQ(55.5)	NQ(50.0)	NQ(49.8)	NQ(50.2)
Trimethoprim	NQ(1.14)	NQ(15.0)	385	NQ(16.0)	NQ(41.1)	DET	293	NQ(12.5)	NQ(12.5)
Virginiamycin	NQ(1.11)	NQ(7.50)	NQ(8.00)	NQ(8.00)	NQ(109)	NQ(187)	NQ(25.0)	NQ(85.4)	NQ(25.1)
Analytical Group 1, other than antibiotics									
1,7-Dimethyl xanthine	NQ(13.1)	NQ(1,320)	DET ^b	NQ(700)	NQ(1,240)	NQ(1,390)	NQ(1,250)	NQ(1,250)	NQ(1,250)
Acetaminophen	NQ(27.3)	NQ(32.5)	NQ(129)	NQ(343)	NQ(494)	NQ(555)	NQ(500)	NQ(498)	NQ(502)
Caffeine	NQ(79.4)	NQ(303)	NQ(309)	NQ(322)	NQ(124)	NQ(139)	NQ ^c	NQ ^c	NQ(125)
Carbamazepine	NA	NA	NA	NA	DET	DET	598	487	NQ(12.5)
Codeine	NA	NQ(628)	NQ(642)	NQ(890)	NQ(24.7)	DET	NQ(25.0)	NQ(24.9)	NQ(25.1)
Cotinine	NQ(1.41)	DET	NQ(722)	NQ(72.0)	46.0	NQ(127)	NQ(83.7)	NQ(61.5)	20.7
Dehydronifedipine	NA	NA	NA	NA	NQ(4.94)	DET	NQ(5.00)	44.7	16.5
Digoxigenin	NQ(1,080)	NQ(120)	NQ(122)	NQ(132)	NQ(186)	NQ(406)	NQ(50.0)	NQ(49.8)	NQ(50.2)
Digoxin	NQ ^a	NQ(1,260)	NQ(1,280)	NQ(1,390)	NQ(124)	NQ(139)	NQ(125)	NQ(125)	NQ(125)
Diltiazem	DET	NQ(2.00)	DET	NQ(2.00)	DET	DET	NQ(5.00)	NQ(4.98)	NQ(5.02)
Diphenhydramine	NA	NA	NA	NA	DET	DET	NQ(5.00) ^a	NQ(4.98) ^a	NQ(5.02)
Fluoxetine	NQ(4.26)	DET ^b	DET	NQ(8.00)	14.7	24.7	NQ(25.0)	DET	NQ(25.1)

C-7

Table C-2 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Miconazole	NA	NA	NA	NA	NQ(12.4)	NQ(13.9)	NQ(12.5)	NQ(12.5)	NQ(12.5)
Norgestimate	NA	NQ(15.0)	NQ(15.0)	NQ(8.00)	NQ(98.0)	NQ(104)	NQ(25.0)	NQ(24.9)	NQ(91.2)
Thiabendazole	NA	NA	NA	NA	<12.8	19.8	DET	DET	NQ(12.5)
Analytical Group 3									
Gemfibrozil	DET	DET	DET	NQ(35.0)	18.9	259	DET	DET	NQ(12.5)
Ibuprofen	NQ(34.6)	NQ(87.0)	NQ(690)	NQ(358)	NQ(124)	NQ(139)	NQ(125)	NQ(125)	NQ(125)
Naproxen	NA	NA	NA	NA	75.3	NQ(27.8)	NQ(25.0)	NQ(24.9)	NQ(25.1)
Triclocarban	NA	NA	NA	NA	154	45.4	40.6	76.4	NQ(25.1)
Triclosan	NA	NA	NA	NA	NQ(494)	NQ(555)	NQ(500)	NQ(498)	NQ(502)
Warfarin	NQ(0.797)	NQ(1.00)	NQ(6.00)	NQ(7.00)	NQ(12.4)	NQ(13.9)	NQ(12.5)	NQ(12.5)	NQ(12.5)
Analytical Group 4									
Albuterol	DET	NQ(30.0)	NQ(125)	NQ(31.0)	NQ(2.83)	DET	NQ(5.01)	NQ(4.92)	NQ(5.03)
Cimetidine	NQ(0.0453)	NQ(41.0)	374	NQ(21.0)	NQ(5.66)	DET	NQ(5.01)	NQ(4.92)	NQ(5.03)
Metformin	DET	DET	EXCLUDE	DET	5,420	1,250	3,650	826	NQ(252)
Ranitidine	NQ ^a	NQ(7.00)	EXCLUDE	NQ(8.00)	NQ(5.66)	DET	7.22	NQ(4.92)	NQ(5.03)

Table C-3. Occurrence of Sterols and Hormones in POTW Influent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Sterols									
Beta Sitosterol	DET	271,000	DET	239,000	DET	DET	DET	DET	DET
Beta Stigmastanol	NA	NA	NA	NA	34,000	27,300	DET	DET	46,000
Campesterol	NA	NA	NA	NA	DET	46,600	DET	DET	DET
Cholestanol	DET	39,700	18,400	35,800	DET	45,700	DET	DET	DET
Cholesterol	DET	636,000	590,000	745,000	DET	DET	DET	DET	DET
Coprostanol	DET	496,000	470,000	366,000	DET	DET	DET	DET	DET
Desmosterol	DET	2,320	1,330	1,370	2,870	2,210	DET	7,030	11,100
Epicoprostanol	DET	20,100	9,370	19,900	5,780	21,400	DET	DET	DET
Ergosterol	DET	DET	NQ(5,230)	EXCLUDE	4,490	2,820	DET	DET	DET
Stigmasterol	DET	17,400	14,800	14,900	DET	37,200	DET	DET	DET
Hormones									
17 Alpha Estradiol	DET	NQ(253)	NQ(256)	NQ(314)	NQ(10.9)	NQ(10.9)	DET	NQ(11.9)	NQ(44.4)
17 Alpha Ethinyl Estradiol	NQ(351)	NQ(253)	NQ(256)	NQ(628)	NQ(13.0)	NQ(13.0)	NQ(5.07) ^a	NQ(11.9)	NQ(3.93) ^a
17 Alpha-Dihydroequilin	DET	NQ(285)	NQ(289)	NQ(354)	DET ^b	NQ(59.3)	NQ(33.2)	NQ(75.1)	NQ(91.0)
17 Beta Estradiol	DET	NQ(283)	NQ(286)	NQ(351)	NQ(10.9)	NQ(10.9)	DET	DET	NQ(38.9)
Alpha-Zearalanol	NQ(347)	DET	DET	NQ(317)	NA	NA	NA	NA	NA
Androstenedione	NA	NA	NA	NA	510	380	864	NQ(378)	NQ(1,640)
Androsterone	NA	NA	NA	NA	DET	DET	2,910	661	DET
Beta Estradiol 3-Benzoate	DET	NQ(263)	NQ(266)	NQ(653)	DET	DET	NQ(5.07)	NQ(11.9)	NQ(3.93)
Desogestrel	NQ(3,510)	NA	NA	NA	NQ(75.0)	NQ(71.5)	NQ(91.1)	NQ(125)	NQ(459)
Equilenin	NA	NA	NA	NA	NQ(5.22)	NQ(5.24)	NQ(5.07)	NQ(11.9)	NQ(13.6)
Equilin	NQ(150)	NQ(251)	NQ(254)	NQ(311)	<12.7	NQ(10.7)	NQ(17.2)	28.9	NQ(101)
Estriol	NA	NA	NA	NA	DET	DET	212	1,000	DET
Estrone	DET	NQ(256)	NQ(259)	NQ(317)	DET	54.1	63.3	98.9	NQ(89.2)
Mestranol	NQ(417)	NQ(266)	DET	NQ(329)	NQ(48.9)	NQ(12.3)	NQ(5.07)	NQ(11.9)	NQ(3.93)
Norethindrone	<810	DET	NQ(80.5)	NQ(396)	NQ(11.2)	DET ^b	NQ(5.07)	NQ(125)	NQ(28.3)

Table C-3 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Norgestrel	NQ(509)	DET	DET	NQ(395)	NQ(606)	NQ(21.8)	NQ(36.3)	NQ(95.2)	NQ(62.1)
Progesterone	NA	NA	NA	NA	NQ(212)	118	NQ(191)	NQ(489)	NQ(349)
Testosterone	DET	DET	DET	NQ(659)	917	831	DET	DET	2,650

Note - To analyze influent samples, EPA's contract laboratory split the sample extract into two portions for analysis of sterols and hormones. The contract laboratory used a small portion for sterol analysis and the remaining portion for hormone analysis. The extract is split in this manner to accommodate the anticipated high sterol levels in samples and preserve the laboratory-reported detection limits of the hormone analyses. The laboratory did not split the effluent samples in this manner because sterols concentrations were anticipated to be much lower in treated effluent.

Table C-4. Occurrence of Sterols and Hormones in POTW Effluent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Sterols									
Beta Sitosterol	NQ(636) ^a	NQ ^a	DET	375	<988 c	<663 c	NQ(9.64) ^a	NQ(9.60) ^a	NQ(5.77) ^a
Beta Stigmastanol	NA	NA	NA	NA	117	NQ(16.6)	NQ(9.64) ^a	NQ(9.60) ^a	19.6
Campesterol	NA	NA	NA	NA	148	37.6	NQ(16.1) ^a	NQ(16.0) ^a	NQ(9.62)
Cholestanol	72.1	155	486	NQ(641)	524	40.6	123	197	NQ(9.62) ^a
Cholesterol	NQ(916) ^a	2,220	15,800	458	2,080	DET	DET	NQ(16.0) ^a	NQ(9.62) ^a
Coprostanol	163	699	DET	NQ(695)	758	114	1,320	111	103
Desmosterol	DET	<190	1,160	NQ(594)	41.7	NQ(31.2)	NQ(32.1)	NQ(32.0)	NQ(19.2)
Epicoprostanol	NQ(6.84)	NQ(252)	<195	NQ(300)	38.5	8.55	DET	4.41	49.4
Ergosterol	DET	DET	DET	EXCLUDE	NQ(30.9)	NQ(30.5)	DET	NQ(80.0)	NQ(48.1)
Stigmasterol	DET	1,300	406	414	<401 c	<287 c	NQ(3.21) ^a	NQ(3.20) ^a	NQ(9.05) ^a
Hormones									
17 Alpha Estradiol	NQ(2.08)	NQ(257)	NQ(300)	NQ(306)	NQ(5.89)	NQ(5.82)	NQ(3.21)	NQ(3.20)	NQ(1.92)
17 Alpha Ethinyl Estradiol	NQ(3.95)	NQ(257)	NQ(300)	NQ(612)	NQ(7.03)	NQ(6.94)	NQ(3.21) ^a	NQ(3.20) ^a	NQ(1.92)
17 Alpha-Dihydroequilin	NQ(7.99)	NQ(289)	NQ(337)	NQ(344)	NQ(19.8)	NQ(20.8)	NQ(11.4)	NQ(3.20)	NQ(8.96)
17 Beta Estradiol	NQ(1.55)	NQ(287)	NQ(334)	NQ(341)	NQ(5.89)	NQ(5.82)	NQ(3.21) ^a	NQ(3.20) ^a	NQ(1.92)
Alpha-Zearalanol	NQ(6.83)	NQ(259)	NQ(303)	NQ(309)	NA	NA	NA	NA	NA
Androstenedione	NA	NA	NA	NA	NQ(31.8)	NQ(31.3)	NQ(64.1)	NQ(97.1)	NQ(95.3)
Androsterone	NA	NA	NA	NA	NQ(6.44)	NQ(6.35)	NQ(14.1)	NQ(23.3)	NQ(11.3)
Beta Estradiol 3-Benzoate	NQ(1.63)	NQ(267)	NQ(311)	NQ(635)	NQ(6.01)	NQ(5.93)	NQ(3.21)	NQ(3.20)	NQ(11.9)
Desogestrel	NQ(44.5)	NA	NA	NA	NQ(6.58)	NQ(16.0)	NQ(16.7)	NQ(20.9)	NQ(12.7)
Equilenin	NA	NA	NA	NA	NQ(2.84)	NQ(2.80)	NQ(3.21)	NQ(3.20)	NQ(1.92)
Equilin	NQ(7.85)	NQ(254)	NQ(297)	NQ(303)	NQ(5.78)	NQ(5.71)	NQ(3.21)	NQ(3.20)	NQ(11.8)
Estriol	NA	NA	NA	NA	NQ(19.0)	NQ(14.4)	NQ(33.1)	NQ(21.9)	NQ(1.92)
Estrone	NQ(1.18)	NQ(259)	NQ(303)	NQ(309)	NQ(6.58)	NQ(6.49)	NQ(3.21) ^a	NQ(3.20)	NQ(14.2)
Mestranol	NQ(1.77)	NQ(269)	NQ(314)	NQ(321)	NQ(6.63)	NQ(6.55)	NQ(3.21)	NQ(3.20)	NQ(1.92)
Norethindrone	NQ(13.7)	NQ(80.6)	NQ(94.0)	NQ(386)	NQ(6.12)	NQ(6.05)	NQ(3.21)	NQ(3.20)	NQ(7.55)

Table C-4 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Norgestrel	NQ(7.65)	NQ(80.6)	NQ(94.0)	NQ(384)	NQ(11.6)	NQ(11.4)	NQ(6.43)	NQ(6.40)	NQ(30.4)
Progesterone	NA	NA	NA	NA	NQ(86.0)	NQ(29.8)	NQ(71.1)	NQ(16.0)	NQ(99.6)
Testosterone	NQ(8.80) ^a	NQ(269)	NQ(314)	NQ(641)	NQ(33.3)	NQ(47.8)	NQ(36.5)	NQ(70.0)	NQ(40.1)

Note - To analyze influent samples, EPA's contract laboratory split the sample extract into two portions for analysis of sterols and hormones. The contract laboratory used a small portion for sterol analysis and the remaining portion for hormone analysis. The extract is split in this manner to accommodate the anticipated high sterol levels in samples and preserve the laboratory-reported detection limits of the hormone analyses. The laboratory did not split the effluent samples in this manner because sterols concentrations are anticipated to be much lower in treated effluent.

Table C-5. Occurrence of Alkylphenols, APEs, and BPA in POTW Influent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
BPA	NQ(5,000)	EXCLUDE	NQ(610)	NQ(1,200)	EXCLUDE	EXCLUDE	NQ(330)	NQ(340)	NQ(320)
NP2EO	NQ(100,000)	NQ(53,000)	DET	200,000	NQ(1,900)	NQ(1,950)	NQ(2,000)	NQ(2,000)	NQ(1,900)
NP1EO	NQ(50,000)	NQ(19,000)	DET	100,000	NQ(1,250)	NQ(1,300)	NQ(1,300)	NQ(1,400)	NQ(1,300)
OP	NQ(5,000)	NQ(2,000)	NQ(400)	13,000	DET	DET	NQ(220)	NQ(230)	3,500
NP	39,700	79,000	44,000	78,000	DET	DET	5,700	24,000	63,000

Table C-6. Occurrence of Alkylphenols, APEs, and BPA in POTW Effluent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
BPA	EXCLUDE	EXCLUDE	NQ(350)	NQ(380)	NQ(100)	NQ(100)	NQ ^c	NQ ^c	NQ(300)
NP2EO	EXCLUDE	NQ(1,100)	NQ(2,100)	NQ(2,200)	NQ(1,100)	NQ(1,100)	NQ(1,100)	NQ(1,100)	NQ(1,800)
NP1EO	NQ(1,000)	1,100	NQ(1,400)	NQ(1,500)	NQ(370)	NQ(370)	NQ(370)	NQ(370)	NQ(1,200)
OP	NQ(100)	NQ(40.0)	NQ(230)	NQ(250)	NQ(40.0)	NQ(40.0)	NQ(40.0)	NQ(40.0)	NQ(200)
NP	NQ(500)	NQ(250)	NQ(1,050)	NQ(1,100)	NQ(250)	<520 ^c	NQ(250)	NQ(250)	NQ(900)

Table C-7. Occurrence of PBDEs in POTW Influent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
PBDE-28+PBDE-33	0.683	2.17	1.33	1.78	1.11	1.48	0.710	4.08	2.96
PBDE-47	DET	97.1	78.1	103	DET	DET	34.0	200	151
PBDE-99	DET	91.1	71.3	96.2	46.6	90.5	18.7	155	148
PBDE-100	8.41	20.7	16.6	23.1	10.2	19.3	4.26	35.8	33.1
PBDE-153	DET	10.6	7.24	10.4	DET	DET	1.57	15.8	14.6
PBDE-154	2.54	8.81	6.08	8.46	DET	DET	DET	11.8	11.9
PBDE-183	0.746	1.52	1.11	2.16	1.45	1.55	0.485	2.08	1.31
PBDE-209	EXCLUDE	211	256	225	260	241	149	119	142

Table C-8. Occurrence of PBDEs in POTW Effluent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
PBDE-28+PBDE-33	NQ(0.0538)	0.0652	0.0897	NQ(0.0593)	0.0574	0.0904	NQ(0.153)	NQ(0.140)	NQ(0.141)
PBDE-47	NQ ^a	1.64	3.37	0.967	DET	0.954	1.49	1.31	1.98
PBDE-99	NQ ^a	1.31	3.01	0.781	1.01	0.456	0.787	0.527	1.63
PBDE-100	NQ(0.0538)	0.265	0.676	0.199	0.221	0.131	0.180	NQ(0.140)	0.347
PBDE-153	NQ(0.0538)	0.138	0.297	0.0810	0.0763	DET	NQ(0.153)	NQ(0.140)	0.142
PBDE-154	NQ(0.0538)	0.111	0.241	0.0743	0.0649	0.0332	NQ(0.153)	NQ(0.140)	NQ(0.141)
PBDE-183	NQ(0.108)	NQ(0.118)	NQ(0.125)	NQ(0.118)	0.0506	0.0705	NQ(0.305)	NQ(0.280)	NQ(0.281)
PBDE-209	NQ(2.15)	NQ(2.36)	DET ^b	NQ(2.37) ^a	NQ(7.65) ^a	<12.0 ^a	NQ(6.10)	NQ(5.61)	DET

Table C-9. Occurrence of Pesticides in POTW Influent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Organochlorine									
2,4'-DDD	DET	1.83	NQ(6.30)	NQ(6.32)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
2,4'-DDE	NQ(1.07)	NQ(3.43)	NQ(6.30)	NQ(3.16)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
2,4'-DDT	NQ(1.29)	NQ(3.43)	1.00	NQ(3.16)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
4,4'-DDD	DET	2.66	NQ(6.30)	NQ(6.32)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
4,4'-DDE	DET	2.35	4.00	3.01	<1.50	2.06	NQ(1.44)	4.58	2.76
4,4'-DDT	DET	NQ(7.63)	2.00	NQ(3.16)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
Aldrin	<0.293	NQ(6.87)	NQ(6.30)	NQ(31.6)	NQ(4.05)	NQ(3.93)	NQ(4.32)	NQ(4.13)	NQ(3.73)
Alpha-BHC	0.640	NQ(6.87)	NQ(6.30)	NQ(6.32)	NQ(2.89)	NQ(2.79)	NQ(2.88)	NQ(2.75)	NQ(2.49)
Alpha-chlordane	<5.06	1.89	DET	12.3	<1.76	3.53	NQ(1.44)	4.32	6.64
Beta-BHC	<0.494	NQ(6.87)	NQ(6.30)	NQ(6.32)	NQ(2.89)	NQ(2.79)	NQ(2.88)	NQ(2.75)	NQ(2.49)
Captan	EXCLUDE	NQ(21.5)	EXCLUDE	NQ(39.5)	EXCLUDE	EXCLUDE	NQ(24.0)	NQ(22.9)	NQ(20.7)
Chlorothalonil	EXCLUDE	1.04	NQ(3.94)	NQ(7.90)	EXCLUDE	EXCLUDE	NQ(4.80)	NQ(4.59)	NQ(4.15)
Cis-Nonachlor	<0.625	0.307	NQ(3.15)	DET	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
Dacthal	NQ(0.200)	NQ(2.15)	NQ(1.97)	NQ(3.95)	NQ(0.900)	NQ(0.873)	NQ(0.959)	NQ(2.29)	NQ(0.830)
Delta-BHC	NQ(0.474)	NQ(6.87)	NQ(6.30)	NQ(31.6)	NQ(2.89)	NQ(2.79)	NQ(2.88)	NQ(2.75)	NQ(2.49)
Dieldrin	DET	1.44	1.00	3.08	NQ(1.44)	6.98	3.61	1.96	7.09
Endosulfan I	DET	NQ(3.43)	NQ ^a	NQ ^a	NQ(4.50)	NQ(4.36)	NQ(4.80)	NQ(4.59)	NQ(4.15)
Endosulfan II	NQ ^a	NQ ^a	NQ ^a	NQ(31.6)	NQ(4.50)	NQ(4.36)	NQ(4.80)	NQ(4.59)	NQ(4.15)
Endosulfan sulfate	NQ(1.20)	NQ(3.43)	NQ(3.15)	NQ(31.6)	NQ(1.80)	NQ(1.74)	NQ(1.92)	NQ(1.83)	NQ(1.66)
Endrin	NQ(0.518)	NQ(3.43)	NQ(0.00)	NQ(3.16)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(1.38)	NQ(1.24)
Endrin Ketone	NQ(1.14)	NQ(3.43)	NQ(3.15)	NQ(12.6)	NQ(1.80)	NQ(1.74)	NQ(1.92)	NQ(1.83)	NQ(1.66)
Gamma-BHC	1.45	7.14	1.00	1.95	NQ(2.89)	NQ(2.79)	NQ(2.88)	NQ(2.75)	NQ(2.49)
Gamma-chlordane	DET	2.60	1.00	16.3	<2.26	6.61	NQ(2.40)	6.99	11.8
Heptachlor	DET	NQ(3.43)	NQ(3.15)	NQ(6.32)	NQ(1.44)	NQ(1.40)	NQ(1.44)	NQ(3.44)	NQ(1.24)
Heptachlor Epoxide	<0.463	0.291	NQ(0.00)	1.46	NQ(1.80)	NQ(1.74)	NQ(1.92)	NQ(1.83)	1.91
Hexachlorobenzene	EXCLUDE	0.425	NQ(0.00)	1.37	NQ(1.80)	NQ(1.74)	NQ(1.92)	NQ(1.83)	NQ(1.66)
Methoxychlor	NQ(3.59)	NQ(6.87)	DET	NQ(15.8)	NQ(14.4)	NQ(9.44)	NQ(8.33)	NQ(23.4)	NQ(9.05)
Mirex	NQ(0.183)	NQ(3.43)	NQ(3.15)	NQ(63.2)	NQ(4.50)	NQ(4.36)	NQ(4.80)	NQ(4.59)	NQ(4.15)

Table C-9 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Octachlorostyrene	NQ(0.272)	NQ(1.20)	NQ(1.10)	NQ(11.1)	NQ(1.80)	NQ(1.74)	NQ(1.92)	NQ(1.83)	NQ(1.66)
Oxychlorthane	NQ(0.630)	NQ(6.87)	NQ(6.30)	NQ(6.32)	NQ(2.89)	NQ(2.79)	NQ(2.88)	NQ(2.75)	NQ(2.49)
Pentachloronitrobenzene	NQ(0.363)	NQ(17.2)	NQ(7.87)	NQ(79.0)	NQ(3.61)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Perthane	NQ(20.4)	NQ(34.3)	DET	NQ(126)	DET	30.4	NQ(84.2)	NQ(66.0)	NQ(79.2)
Tecnazene	NQ(0.333)	NQ(8.58)	NQ(7.87)	NQ(39.5)	NQ(3.60)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Trans-Nonachlor	<3.46	1.50	1.00	7.86	NQ(1.80)	2.01	NQ(1.92)	2.96	4.66
Organophosphorus									
Azinphos-methyl	NQ(38.8)	NQ(10.7)	NQ(19.7)	NQ(98.7)	NQ(35.6)	NQ(8.72)	NQ(9.59)	NQ(54.6)	NQ(8.30)
Chlorpyriphos	DET	19.3	17.0	NQ(79.0)	NQ(3.61)	13.0	NQ(3.84)	262	64.9
Chlorpyriphos-oxon	NQ(2.07)	NQ(8.58)	NQ(7.87)	DET	NQ(3.61)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Diazinon	NQ(4.67)	8.89	DET	41.0	NQ(3.61)	71.9	NQ(3.84)	6.31	20.9
Diazinon oxon	NQ(1.63)	NQ(8.58)	NQ(7.87)	NQ(158)	NQ(3.60)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Disulfoton	NQ(26.2)	NQ(42.5)	NQ(39.0)	NQ(78.2)	NQ(18.0)	NQ(17.5)	NQ(19.2)	NQ(18.3)	NQ(16.6)
Disulfoton sulfone	NQ(0.699)	NQ(0.687)	NQ(0.630)	DET	NQ(1.35)	NQ(1.31)	NQ(1.44)	NQ(1.38)	NQ(1.24)
Ethyl-parathion	NQ(7.66)	NQ(8.58)	NQ(7.87)	NQ(15.8)	NQ(3.60)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Fenitrothion	NQ(3.46)	NQ(8.58)	NQ(7.87)	NQ(31.6)	NQ(3.61)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Fonofos	NQ(1.26)	NQ(8.58)	NQ(7.87)	NQ(39.5)	NQ(3.61)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Malathion	NQ(70.7)	NQ(114)	NQ(104)	DET	NQ(46.8)	NQ(45.4)	NQ(48.0)	NQ(45.9)	474
Methamidophos	NQ(62.3)	NQ(60.3)	NQ(15.7)	DET	NQ(45.0)	NQ(65.2)	NQ(48.0)	NQ(45.9)	NQ(41.5)
Methyl-chlorpyriphos	DET	NQ(10.7)	NQ(9.84)	NQ(19.7)	NQ(4.68)	NQ(4.54)	NQ(4.80)	NQ(4.59)	NQ(4.15)
Methyl-parathion	NQ(38.3)	NQ(25.8)	NQ(23.6)	94.6	NQ(10.8)	NQ(10.5)	NQ(9.59)	NQ(9.17)	NQ(8.30)
Phorate	NQ(5.40)	NQ(17.2)	NQ(15.7)	NQ(31.6)	NQ(9.00)	NQ(8.72)	NQ(9.59)	NQ(9.17)	NQ(8.30)
Phosmet	NQ(8.36)	NQ(21.5)	NQ(39.4)	NQ(79.0)	NQ(21.6)	NQ(8.73)	NQ(9.59)	NQ(9.17)	NQ(8.30)
Pirimiphos-methyl	NQ(0.736)	NQ(8.58)	NQ(7.87)	NQ(15.8)	NQ(3.61)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Pyrethroid									
Cis-Permethrin	NA	NA	NA	NA	25.3	155	NQ(9.59)	44.5	306
Cypermethrins	<23.2	26.4	52.0	70.5	NQ(9.00)	DET	NQ(9.59)	DET	66.8
Permethrin	DET	146	176	349	59.0	DET	NQ(19.1)	104	689
Trans-Permethrin	NA	NA	NA	NA	33.8	238	9.26	59.3	383

Table C-9 (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Triazine									
Ametryn	NQ(1.85)	NQ(8.58)	NQ(7.87)	NQ(15.8)	NQ(3.61)	NQ(3.49)	NQ(3.84)	NQ(3.67)	NQ(3.32)
Atrazine	DET	87.7	DET	DET	35.9	58.9	DET	NQ(13.2)	37.0
Cyanazine	NQ(11.1)	NQ(8.58)	NQ(7.87)	NQ(31.6)	NQ(15.7)	NQ(17.4)	NQ(3.84)	NQ(3.67)	NQ(11.1)
Desethyl atrazine	DET	27.3	31.0	4.96	58.0	7.84	2.65	NQ(1.83)	6.11
Hexazinone	NQ(6.16)	NQ(10.7)	NQ(19.7)	NQ(79.0)	NQ(4.68)	7.13	NQ(4.80)	NQ(11.5)	NQ(4.15)
Metribuzin	NQ(3.43)	NQ(2.15)	NQ(1.97)	NQ(7.90)	NQ(2.70)	NQ(2.62)	NQ(2.88)	NQ(2.75)	NQ(2.49)
Simazine	DET	2.67	DET	NQ(15.8)	6.65	NQ(3.49)	NQ(3.84)	NQ(9.17)	NQ(3.32)

Table C-10. Occurrence of Pesticides in POTW Effluent (ng/L)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Organochlorine									
2,4'-DDD	NQ(0.404)	NQ(3.55)	NQ(3.17)	NQ(3.16)	NQ(0.745)	NQ(0.699)	NQ(0.657)	NQ(0.691)	NQ(0.614)
2,4'-DDE	NQ(0.575)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.745)	NQ(1.48)	NQ(0.657)	NQ(0.691)	NQ(0.614)
2,4'-DDT	NQ(0.452)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.745)	NQ(1.48)	NQ(0.657)	NQ(0.691)	NQ(0.614)
4,4'-DDD	NQ(0.220)	NQ(3.55)	NQ(3.17)	NQ(3.16)	NQ(0.745)	NQ(1.48)	NQ(0.657)	NQ(0.691)	NQ(1.23)
4,4'-DDE	NQ(0.743)	NQ(3.55)	NQ(3.17)	NQ(3.16)	NQ(0.745)	NQ(1.48)	NQ(0.657)	NQ(0.691)	NQ(0.614)
4,4'-DDT	NQ(0.468)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.745)	NQ(1.48)	NQ(0.657)	NQ(0.691)	NQ(0.614)
Aldrin	NQ(0.115)	NQ(7.09)	NQ(6.33)	NQ(3.16)	NQ(2.09)	NQ(1.97)	NQ(1.97)	NQ(2.07)	NQ(1.84)
Alpha-BHC	NQ(0.154)	NQ(7.09)	NQ(6.33)	NQ(3.16)	NQ(1.49)	NQ(1.40)	NQ(1.31)	NQ(1.38)	NQ(1.23)
Alpha-chlordane	NQ(0.174)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.745)	NQ(0.699)	NQ(0.657)	NQ(0.691)	NQ(0.614)
Beta-BHC	NQ(0.213)	NQ(7.09)	NQ(6.33)	NQ(3.16)	NQ(1.49)	NQ(1.40)	NQ(1.31)	NQ(1.38)	NQ(1.23)
Captan	NQ(9.46)	NQ(22.2)	EXCLUDE	NQ(19.7)	EXCLUDE	EXCLUDE	NQ(10.9)	NQ(11.5)	NQ(20.5)
Chlorothalonil	NQ(0.0867)	NQ(4.43)	NQ(3.95)	NQ(3.95)	EXCLUDE	EXCLUDE	NQ(2.19)	NQ(2.30)	NQ(2.05)
Cis-Nonachlor	NQ(0.216)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.745)	NQ(1.48)	NQ(0.657)	NQ(0.691)	NQ(0.614)
Dacthal	NQ(0.0867)	NQ(2.22)	NQ(1.98)	NQ(1.97)	NQ(0.466)	NQ(0.437)	NQ(0.438)	NQ(0.460)	NQ(0.409)
Delta-BHC	NQ(0.233)	NQ(7.09)	NQ(6.33)	NQ(3.16)	NQ(1.49)	NQ(1.40)	NQ(1.31)	NQ(1.38)	NQ(1.23)
Dieldrin	NQ(0.102)	0.273	NQ(0.00)	0.589	NQ(0.745)	1.64	0.888	NQ(0.691)	1.40
Endosulfan I	2.06	NQ ^a	NQ ^a	NQ ^a	NQ(2.33)	NQ(2.18)	NQ(2.19)	NQ(2.30)	NQ(2.05)
Endosulfan II	NQ(0.429) ^a	NQ ^a	NQ(3.17)	NQ(3.16)	NQ(2.33)	NQ(2.18)	NQ(2.19)	NQ(2.30)	NQ(2.05)
Endosulfan sulfate	2.86	NQ(3.55)	NQ(3.17)	NQ(3.16)	NQ(0.931)	NQ(0.873)	NQ(0.876)	NQ(0.921)	NQ(0.819)
Endrin	NQ(0.229)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.745)	NQ(0.699)	NQ(0.657)	NQ(0.691)	NQ(0.614)
Endrin Ketone	NQ(0.369)	NQ(3.55)	NQ(4.73)	NQ(6.32)	NQ(0.931)	NQ(0.873)	NQ(0.876)	NQ(0.921)	NQ(0.819)
Gamma-BHC	NQ(0.244)	2.45	1.00	3.84	NQ(1.49)	NQ(1.40)	NQ(1.31)	NQ(1.38)	NQ(1.23)
Gamma-chlordane	NQ(0.186)	NQ(3.55)	NQ(3.17)	0.863	NQ(1.16)	NQ(1.09)	NQ(1.09)	NQ(1.15)	NQ(1.02)
Heptachlor	NQ(0.0347)	NQ(3.55)	NQ(3.17)	NQ(3.16)	NQ(0.745)	NQ(0.699)	NQ(0.657)	NQ(0.691)	NQ(0.614)
Heptachlor Epoxide	NQ(0.143)	NQ(3.55)	NQ(3.17)	0.471	NQ(0.931)	NQ(0.873)	NQ(0.876)	NQ(0.921)	NQ(0.819)
Hexachlorobenzene	EXCLUDE	NQ(3.55)	NQ(3.17)	NQ ^a	NQ(0.931)	NQ(0.873)	NQ(0.876)	NQ(0.921)	NQ(0.819)
Methoxychlor	DET	DET	NQ(6.27)	0.416	NQ(1.63)	DET ^b	NQ(0.657)	NQ(0.691)	NQ ^c
Mirex	NQ(0.0657)	NQ(3.55)	NQ(3.17)	NQ(6.32)	NQ(2.33)	NQ(2.18)	NQ(2.19)	NQ(2.30)	NQ(2.05)

Table C-10. (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Octachlorostyrene	NQ(0.0662)	NQ(1.24)	NQ(1.11)	NQ(1.11)	NQ(0.931)	NQ(0.873)	NQ(0.876)	NQ(0.921)	NQ(0.819)
Oxychlorthane	NQ(0.184)	NQ(7.09)	NQ(6.33)	NQ(3.16)	NQ(1.49)	NQ(2.96)	NQ(1.31)	NQ(1.38)	NQ(1.23)
Pentachloronitrobenzene	NQ(0.0867)	NQ(8.86)	NQ(7.91)	NQ(7.90)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Perthane	NQ(0.403)	NQ(35.5)	NQ(31.7)	NQ(63.2)	NQ(8.78)	NQ(2.18)	NQ(2.19)	NQ(2.30)	NQ(2.05)
Tecnazene	NQ(0.0867)	NQ(4.43)	NQ(3.95)	NQ(3.95)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Trans-Nonachlor	NQ(0.173)	NQ(3.55)	NQ(3.17)	NQ(1.58)	NQ(0.931)	NQ(0.873)	NQ(0.876)	NQ(0.921)	NQ(0.819)
Organophosphorus									
Azinphos-methyl	NQ(6.45)	NQ(11.1)	NQ(9.88)	NQ(9.87)	NQ(4.65)	NQ(4.37)	NQ(4.38)	NQ(4.60)	NQ(4.09)
Chlorpyriphos	NQ(0.817)	NQ(8.86)	NQ(7.91)	NQ(7.90)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	EXCLUDE
Chlorpyriphos-oxon	NQ(2.04)	NQ(8.86)	NQ(7.91)	NQ(15.8)	NQ(1.86)	NQ(1.75)	NQ(1.75)	15.9	2.59
Diazinon	NQ(0.610)	NQ(8.86)	3.50	3.84	<11.9	DET	2.86	NQ(1.84)	NQ(1.64)
Diazinon oxon	NQ(0.479)	6.67	NQ(11.8)	NQ(15.8)	NQ(1.86)	NQ(1.75)	NQ(1.75)	4.49	NQ(1.64)
Disulfoton	NQ(8.84)	NQ(43.9)	NQ(58.6)	NQ(39.1)	NQ(9.31)	NQ(18.5)	NQ(8.76)	NQ(9.21)	NQ(8.19)
Disulfoton sulfone	NQ(0.345)	NQ(0.709)	NQ(0.944)	NQ(0.632)	NQ(0.698)	NQ(0.655)	NQ(0.657)	NQ(0.691)	NQ(1.23)
Ethyl-parathion	NQ(1.48)	NQ(8.86)	NQ(7.91)	NQ(7.90)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Fenitrothion	NQ(1.66)	NQ(8.86)	NQ(7.91)	NQ(15.8)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Fonofos	NQ(0.207)	NQ(8.86)	NQ(7.91)	NQ(3.95)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Malathion	NQ(27.2)	NQ(118)	NQ(105)	NQ(105)	NQ(24.2)	NQ(48.1)	NQ(21.9)	NQ(23.0)	NQ(20.5)
Methamidophos	NQ(58.6)	NQ(8.86)	NQ(7.91)	DET	NQ(34.8)	NQ(21.8)	NQ(21.9)	NQ(97.4)	NQ(20.5)
Methyl-chlorpyriphos	NQ(0.0899)	NQ(11.1)	NQ(9.88)	NQ(9.87)	NQ(2.42)	NQ(2.27)	NQ(2.19)	NQ(2.30)	NQ(2.05)
Methyl-parathion	NQ(21.8)	NQ(26.6)	NQ(23.7)	NQ(23.7)	NQ(5.59)	NQ(11.1)	NQ(4.38)	NQ(4.60)	NQ(4.09)
Phorate	NQ(1.59)	NQ(8.86)	NQ(7.91)	NQ(15.8)	NQ(4.65)	NQ(4.37)	NQ(4.38)	NQ(4.60)	NQ(4.09)
Phosmet	NQ(1.26)	NQ(22.2)	NQ(19.8)	NQ(39.5)	NQ(4.66)	NQ(9.27)	NQ(4.38)	NQ(4.60)	NQ(4.09)
Pirimiphos-methyl	NQ(0.123)	NQ(8.86)	NQ(7.91)	NQ(7.90)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Pyrethroid									
Cis-Permethrin	NA	NA	NA	NA	NQ(4.65)	NQ(4.37)	NQ(4.38)	NQ(4.60)	NQ(4.09)
Cypermethrins	NQ(2.82)	NQ(22.2)	NQ(19.8)	NQ(19.7)	NQ(4.66)	NQ(9.27)	NQ(4.38)	NQ(4.60)	NQ(4.09)
Permethrin	NQ(0.775)	<3.34	6.00	NQ(3.95)	NQ(9.26)	NQ(8.69)	NQ(8.71)	NQ(9.16)	NQ(6.14)
Trans-Permethrin	NA	NA	NA	NA	NQ(2.33)	NQ(2.18)	NQ(2.19)	NQ(2.30)	NQ(2.05)

Table C-10. (Continued)

Analyte	Plant A	Plant B	Plant C	Plant D	Plant E	Plant F	Plant G	Plant H	Plant I
Triazine									
Ametryn	NQ(0.463)	NQ(8.86)	NQ(7.91)	NQ(7.90)	NQ(1.86)	NQ(1.75)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Atrazine	DET	60.1	DET	DET	32.9	53.8	DET	DET	26.3
Cyanazine	NQ(2.24)	NQ(8.86)	DET ^b	NQ(15.8)	NQ(4.11)	NQ(4.54)	NQ(1.75)	NQ(1.84)	NQ(1.64)
Desethyl atrazine	DET	25.0	27.5	6.13	57.2	7.44	1.63	NQ(0.921)	5.83
Hexazinone	NQ(1.18)	NQ(11.1)	NQ(9.88)	NQ(39.5)	4.09	DET	NQ(2.19)	NQ(2.30)	NQ(2.05)
Metribuzin	NQ(1.17)	NQ(2.22)	DET ^b	NQ(3.95)	NQ(1.40)	1.90	NQ(1.31)	NQ(1.38)	NQ(1.23)
Simazine	2.64	2.55	5.00	NQ(7.90)	5.05	NQ(1.75)	NQ(1.75)	5.28	NQ(1.64)