

Chemicals of Concern

Iowa's First Field Research Area for Emerging Contaminants

Douglas Schnoebelen

Dana Kolpin

Larry Barber

Edward Furlong

Michael Meyer

Mary Skopec

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The Iowa Policy Project

318 2nd Avenue North, Mount Vernon, Iowa 52314

319-338-0773

www.iowapolicyproject.org

The Authors:

Douglas Schnoebelen is a research hydrologist for the U.S. Geological Survey (USGS) and an adjunct professor of Geology at the University of Iowa.

Dana Kolpin is a research hydrologist for the USGS.

Larry Barber is a research geologist for the USGS.

Edward Furlong is a research chemist for the USGS.

Michael Meyer is a supervisory geochemist for the USGS.

Mary Skopec is a research geologist for the Iowa Geological Survey of the Iowa Department of Natural Resources.

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Abstract

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Research has recently documented the prevalence of a wide variety of pharmaceuticals and other emerging contaminants (ECs) in streams across the United States. Wastewater treatment plants (WWTPs) have been found to be an important source and collection point of ECs to streams as many ECs are incompletely removed during treatment. To investigate the complex in-stream processes (e.g. dilution, sorption, degradation, dispersion, etc.) that can affect ECs following their input from a WWTP and determining if such input is having an effect on the aquatic ecosystem requires the integration of multi-disciplinary efforts at a carefully selected field site. Knowledge gained from previous research identified an 8-km reach of Fourmile Creek in central Iowa as an ideal research site to investigate such important research questions pertaining to ECs. Unique aspects of Fourmile Creek included: (1) a single source effluent-dominated stream, (2) background data document the input of a wide variety of ECs from WWTP discharge, (3) small basin size, (4) relatively simple flow system, (5) background data suggest that undefined processes are taking place reducing the level of some ECs during stream transport, (6) the WWTP uses treatment technology typical of many towns and cities, (7) the hydrogeologic setting is typical of many areas across the United States, (8) a low-head dam exists approximately 2 km upstream of the WWTP outfall allowing more accurate “above WWTP” and “below WWTP” comparisons in aquatic ecosystems, and (9) the WWTP is scheduled to close by around 2010 providing a unique opportunity to examine how a stream and aquatic biota react to the removal of the primary source of ECs and allowing a novel “before” and “after” assessment that has not been previously available in EC research. With the installation of a streamflow gaging station, dye-tracing tests conducted at varying flow conditions to determine water travel times, Lagrangian water-quality sampling at two flow (low vs. medium) and water temperature (warm vs. cold) regimes, sediment sampling, and basic fish community and fish health assessment, a unique framework has been built to investigate the important question of how ECs are transported through the environment and if the presence of such compounds are having a deleterious effect on aquatic ecosystems.

Emerging Contaminants: Chemicals of Concern

A wide variety of chemicals are used everyday in today's society (homes, industry, agriculture, etc.). Recent research has shown that compounds not previously considered as contaminants are present in the environment (Halling-Sorenson and others, 1998, Kolpin and others, 2002). These include a number of compounds such as human and veterinary prescription drugs, diagnostic agents, hormones, cosmetics, dyes, preservatives, detergents, and numerous other organic compounds. There are increasing concerns about the potential environmental effects that may inadvertently occur from such "emerging contaminants" (ECs). What are ECs? A useful working definition follows:

The term emerging contaminants can be broadly defined as any synthetic or naturally occurring chemical or any microorganism that is not commonly monitored in the environment, but has the potential to enter the environment and can cause suspected adverse ecological and/or human health effects. In some cases, release of emerging chemical or microbial contaminants to the environment has likely occurred for a long time, but may not have been recognized until new detection methods were developed. In other cases, synthesis of new chemicals or changes in use and disposal of existing chemicals can create new sources of emerging contaminants. (U.S. Geological Toxic Substances Hydrology, 2005).

Most ECs are not routinely monitored. Indeed, water-quality monitoring in the United States is largely driven by regulations of the Clean Water Act and Safe Drinking Water Act. Therefore, most monitoring programs are focused on compounds that are assigned standards by federal or state agencies (U.S. Environmental Protection Agency, 2003; U.S. Environmental Protection Agency, 2004). Over the last three decades, much of the water-quality monitoring work has focused almost exclusively on the conventional "priority pollutants," but this is only one piece of the larger environmental puzzle (Daughton and Ternes, 1999). Only recently have ECs begun to be examined in limited studies using newly developed laboratory analytical methods and techniques. Furthermore, the possibility that environmental contaminants may be complex mixtures that can interact synergistically or antagonistically has increased the need to understand ECs found in our waters.

In order to minimize ecologic effects from ECs, it is essential to understand how a contaminant moves and is altered in the environment. Investigations of processes influencing transport (e.g. sorption, dispersion, degradation, etc.) require a systematic evaluation of a variety of hydrologic, landscape and anthropogenic factors. The purpose of this paper is to provide a short synopsis of ECs as potential contaminants of concern and to highlight an 8-km reach of Fourmile Creek in central Iowa as an ideal research site to investigate the transport, fate, and effects from an urban source of ECs.

Analytical Development, Diverse Chemicals, and Complex Pathways

Recent advances in sample extraction and analytical instrumentation now permit the environmental measurement of ECs at unprecedented detection levels (e.g. Sedlak and others, 2000; Kolpin and others 2002; Cahill and others, 2004; Burkhardt and others, 2005). Quantification for many ECs was first reported in the parts per billion (microgram per liter) range, but now results are commonly reported in the parts per trillion (nanogram per liter) range. In comparison, many common pesticide compounds (atrazine, metolachlor, acetochlor, etc.) are routinely analyzed and reported in the parts per billion range and common inorganic substances (sodium, chloride, nitrate, etc.) are analyzed and reported in the parts per million (milligrams per liter) range. The low detection levels allow researchers to better define the range of compounds present in the environment and to properly gauge the importance of ECs. Improvements in

analytical methods allow policy makers to better determine potential health-based thresholds. (Focazio and others, 2004, p. 92).

The use of ECs for both human and veterinary purposes result in complex fate pathways through the environment (Figure 1). Research has shown that wastewater treatment plants (WWTPs) are an important collection point and source of ECs to the environment (Heberer, 2002; Carballa and others, 2004; Joss and others, 2005; Miao and others, 2004; Xia and others, 2005). WWTPs have multiple pathways to the environment including direct discharge of treated effluent to surface water bodies (Ashton and others, 2004; Glassmeyer and others, 2005), land application of treated effluent (Kinney and others, 2005), and land application of treated biosolids (Yang and Metcalf, 2005). Thus, as sinks for ECs, WWTPs are ideal locations to reduce the loading of these compounds to the environment. Early research comparing influent and effluent concentrations at select WWTPs in New York has shown that treatment technologies vary in their ability to reduce EC concentrations (Phillips et al., 2005).

Possible Effects of ECs: Endocrine Disruption and Antibiotic Resistance

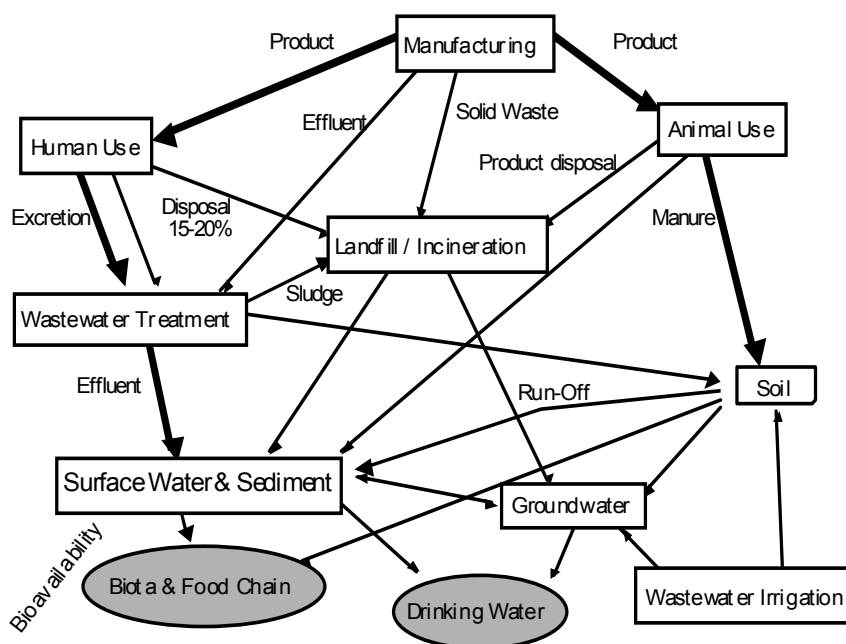
The potential toxicological behavior from the environmental occurrence of ECs and mixtures of ECs are largely unknown. In particular, the effects of ECs on aquatic organisms are difficult to measure because the concentrations of these compounds are generally low (nanogram per liter range) and over the life of the organism produce no acutely toxic effects. However, detrimental effects to organisms from ECs may be subtle and go unnoticed until some cumulative threshold is reached. In recent years the presence and effects of endocrine disrupting compounds (EDCs) in the environment has become an important issue (Keith, 1997). The endocrine system is the “key control system” of most organisms as hormones are secreted that interact with specific receptors on cells that enable functions to be controlled (Global Water Research Coalition, 2003). A working definition of an EDC as defined by the World Health Organization (WHO) is “an exogenous substance that alters the function of the endocrine system and consequently causes adverse health effects in an organism, or its progeny, or (sub)populations” (U.S. Environmental Protection Agency, web page <http://epa.gov/endocrine/Pubs/smithrep.html> accessed August, 2005). The presence of low concentrations of some chemicals in the environment (e.g. natural and synthetic hormones, alkylphenols, pesticides, solvents and pharmaceuticals) could affect or damage the function of the endocrine system (Global Water Research Coalition, 2003). For example, nonylphenol (a detergent degradation product), and AHTN (a polycyclic musk) have been shown to disrupt reproduction and growth in fish by affecting endocrine systems (Thorpe and others, 2001; Schreurs and others, 2004). A variety of ECs have been shown to bioaccumulate in fish tissue (Brooks and others, 2005; Kukrunthachalam and others, 2005). Data from laboratory experiments suggest that EDCs in the aquatic environment may impact the reproductive health of fish populations (Mills and Chichester, 2005). Linking EDCs to observed changes in fish populations, however, remains an open challenge (Mills and Chichester, 2005). Although less is known about potential effects to other aquatic species, early research suggest that effects to aquatic organisms are possible (Flaherty and Dodson, 2005; Oetken and others, 2005; Wilson and others, 2003). Indeed, ecological risk assessment for EDCs in the environment is in the infancy stage of research (Hartemann, 2004, p. 267).

Antibiotics are an important class of pharmaceuticals and their prevalence and use in the last 60 years has brought dramatic and often even “miraculous” progress in fighting bacterial infections in humans and animals. In livestock farming, sub-therapeutic doses of antibiotics are often used to promote more rapid animal growth (Alexy and others, 2004). Despite their widespread use, antibiotics have only recently received attention as environmental contaminants. However, the increase of resistant bacterial strains and the spread of bacterial resistance have become a worldwide concern (Kummerer, 2004b). Concerns also exist for antibiotic use and increasing antibiotic resistance in livestock confined feeding operations (Boxall and others, 2003; Osterberg and Wallinga, 2004). Many antibiotics are only partially metabolized after

administration to humans or animals (Hamscher and others, 2004). Concentrations of select antibiotics in animal manure have been reported at milligrams per liter levels (Hamscher and others, 2004, p. 140; Meyer, 2004).

Antibiotics can reach streams and ground water via a variety of mechanisms (Figure 1) and the potential for the aquatic environment to promote or maintain antibiotic resistance is largely unknown. Some chemicals, such as triclosan (an antimicrobial disinfectant found in many liquid soaps, dishwasher powders, and plastics), are suspected of increasing the antibiotic resistance of bacteria in the environment (McMurry and others, 1998), reducing algae diversity in streams (Wilson and others, 2003), and affecting natural ecosystem functions such as soil microbial activity (Thiele-Bruhn and Beck, 2005). In addition, research has shown effects of mixtures of antibiotics to aquatic organisms (Brain and others, 2005).

Figure 1. Potential sources, pathways, and sinks of emerging contaminants in the environments. Modified from Halling-Sorenson and others, 1998.



Evolution of Fourmile Creek as a Research Site for ECs

Following a national stream reconnaissance study (Kolpin and others, 2002), water samples were collected in 2001 upstream and downstream of select towns and cities in Iowa during low-, normal- and high-flow conditions to determine the contribution of urban centers to concentrations of ECs in streams under varying flow conditions (Kolpin and others, 2004). This study found that the number of ECs detected decreased as streamflow increased from low- (51 ECs detected) to normal- (28) to high-flow (24) conditions. Fourmile Creek near Ankeny, Iowa, was sampled for ECs for the first time during this study and results showed that a strong gradient in EC detections was observed during low-flow conditions between samples collected upstream of Ankeny (three ECs detected) compared to samples collected downstream (31 EC detected).

The initial EC results from Fourmile Creek (Kolpin and others, 2004), led to including this stream as part of collaborative research between the U.S. Geological Survey (USGS) and the U.S. Environmental Protection Agency to better understand the fate of ECs following their discharge from WWTPs (Glassmeyer and others, 2005). This research involved collecting four samples at each of 10 WWTPs across the nation: upstream of the WWTP, at the WWTP where effluent was being discharged into the stream, at a location in close proximity downstream of the WWTP, and at a location farther downstream from the WWTP. All samples were measured for 110 ECs and found between 28 and 50 ECs in treated wastewater effluent being discharged to streams (Glassmeyer et al., 2005). The similarity in chemical concentrations between WWTP effluent and proximal downstream sampling points clearly shows the contribution of WWTPs to EC concentrations in streams. Additional knowledge gained from Fourmile Creek during this study included:

- (1) the ECs detected in Fourmile Creek during the previous study (Kolpin and others, 2004) were primarily derived from the Ankeny WWTP (Figure 2),
- (2) there are significant reductions of the number of ECs detected and total EC concentrations through the 8.4 km study reach (Figure 2) by undefined natural processes (e.g. sorption, microbial degradation, photolysis, etc.),
- (3) ECs vary in their type of transport (conservative vs. nonconservative) through the study reach (Table 1),
- (4) at low-flow conditions, greater than 90 percent of the streamflow is derived from WWTP discharge (Glassmeyer and others, 2005).

Figure 2. Number of compounds and total concentration of analytes found in samples collected from Fourmile Creek near Ankeny, Iowa, 2002 (Glassmeyer and others, 2005). [WWTP, wastewater treatment plant km, kilometers].

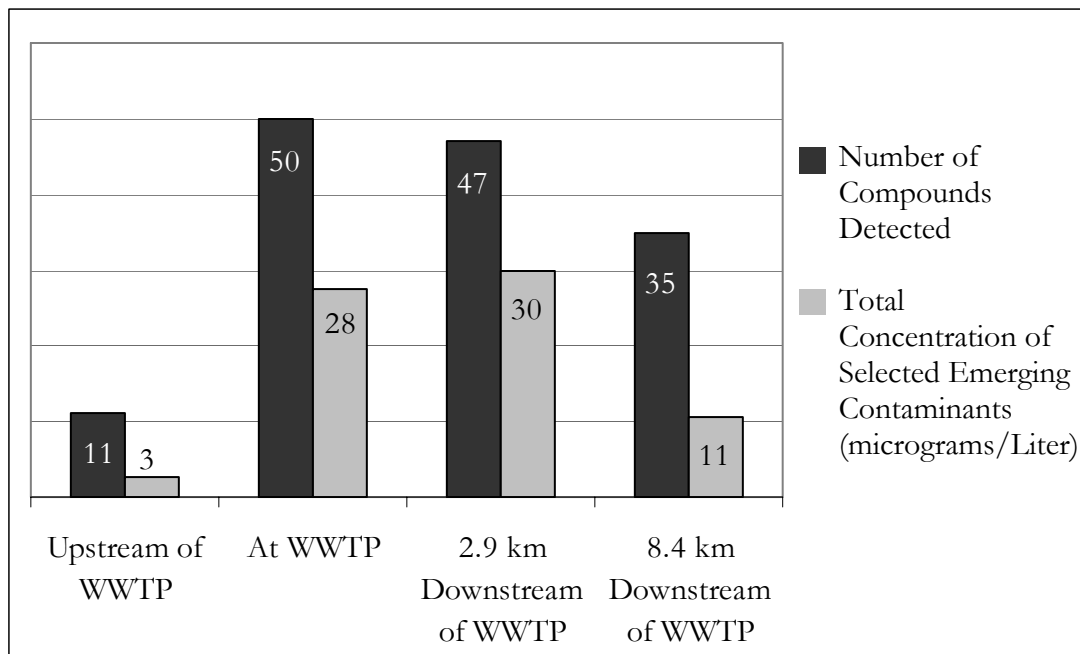


Table 1. Selected compounds detected, primary use, reporting level and concentrations – upstream, at source, and downstream – from samples collected at Fourmile Creek near Ankeny, Iowa, 2002. (Glassmeyer and others, 2005; written communication, June 2005, Susan Glassmeyer, U.S. Environmental Protection Agency). [$\mu\text{g/L}$, micrograms per liter; WWTP, wastewater treatment plant, km, kilometers; location of sites shown on figures 2 and 5].

Compound	Primary Use	Reporting Level ($\mu\text{g/L}$)	Concentration		
			upstream (site 1) of WWTP ($\mu\text{g/L}$)	at source (site 2) WWTP ($\mu\text{g/L}$)	Concentration 8.4 km downstream (site 5) of WWTP ($\mu\text{g/L}$)
Cimetidine	Antacid	0.012	undetected	0.123	0.107
Dehydronifedipine	Antianginal	0.015	undetected	0.202	0.018
Diltiazem	Antihypertensive	0.016	undetected	0.053	0.029
Diphenhydramine	Antihistamine	0.015	undetected	0.218	undetected
Sulfamethozole	Antibiotic	0.064	undetected	0.589	0.321
Tonalide (AHTN)	Fragrance, musk	0.500	undetected	2.300	0.700
Trimethoprim	Antibiotic	0.013	undetected	0.353	0.093

In 2003, the USGS EC Project (<http://toxics.usgs.gov/regional/emc/index.html>) was searching for a real-world setting to investigate the complex in-stream processes (e.g. dilution, sorption, degradation, dispersion, etc.) that can affect ECs following their discharge from a WWTP and determining if such input is having an effect on the aquatic ecosystem. Such research requires the integration of multi-disciplinary efforts at a carefully selected field site. Knowledge gained from previous research (Kolpin and others, 2004; Glassmeyer and others, 2005) and other unique aspects of Fourmile Creek lead to its selection as a field setting to help answer these important research questions. Critical aspects of Fourmile Creek included the following:

- (1) A single source effluent-dominated stream. The effluent discharge into Fourmile Creek can exceed 90 percent of the streamflow downstream of the WWTP outfall during low-flow conditions (Glassmeyer and others, 2005). A single source effluent-dominated stream allows for the examination of EC concentrations as water moves downstream without complications from additional inputs.
- (2) Background data document the input of a wide variety of ECs from WWTP discharge. Previous research found between 3 and 10 ECs present upstream of the WWTP and between 30-50 ECs downstream (Kolpin and others, 2004; Glassmeyer and others, 2005). Detectable concentrations are necessary to determine longitudinal patterns with downstream transport.
- (3) Small basin size (less than 160 km² size). A small basin size facilitates an increased understanding of the transport and fate of environmental contaminants as larger basins tend to have more complex interactions and flows that can obscure existing trends.
- (4) Relatively simple flow system. Little to no ground-water or surface-water inputs to stream flow exist in Fourmile Creek during normal flow conditions. Thus, any changes in EC concentrations observed with transport downstream can be attributed to in-stream processes taking place rather than from simple dilution of additional flow inputs from ground-water or surface-water sources.
- (5) Background data document that ECs vary in their type of transport. Within an 8 km stretch of Fourmile Creek, some compounds were found to behave relatively conservatively while others exhibited substantial reductions in concentration as water migrated downstream (Table 1). Thus, currently undefined processes are taking place within the stream that can affect EC concentrations.

(6) The WWTP uses a treatment technology (conventional activated-sludge) typical of many towns and cities across the United States. Thus, the source is representative of many similar sources in the United States.

(7) The hydrogeologic setting (low-gradient stream, glaciated deposits, rowcrop agriculture) is typical of the Midwest.

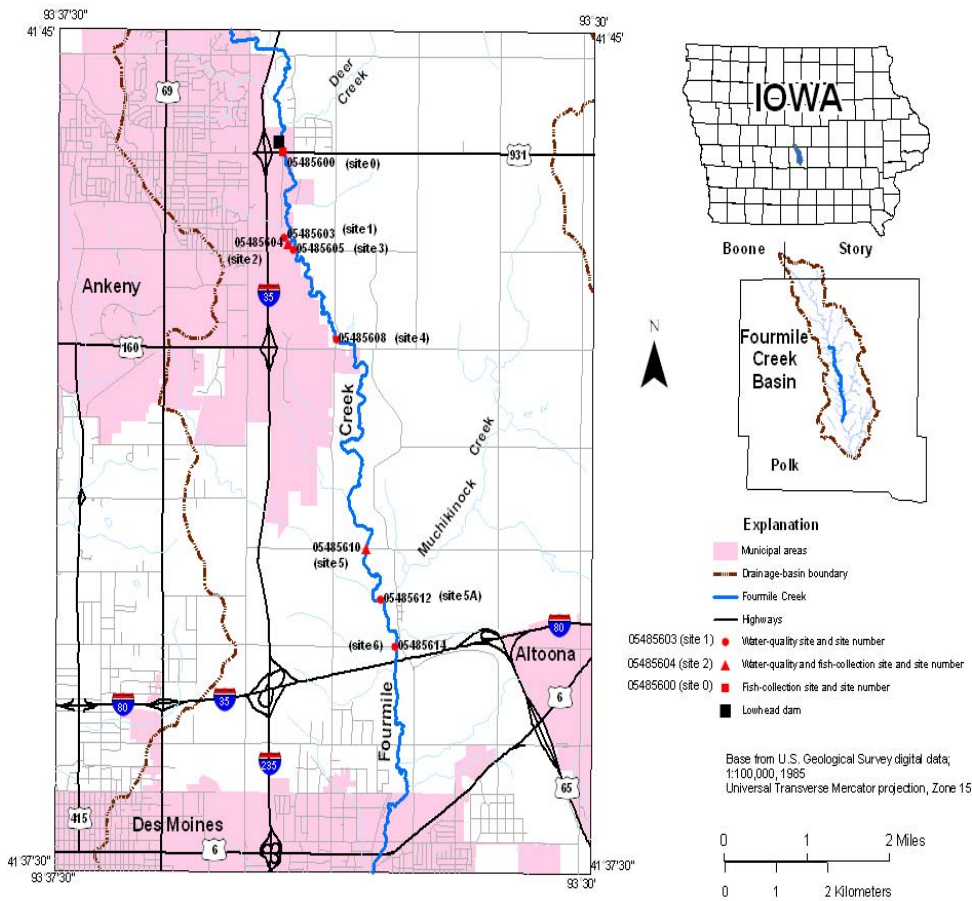
(8) A low-head dam exists approximately 2 km upstream of the WWTP outfall. The low-head dam provides a physical barrier to fish migration. Thus, “above WWTP” and “below WWTP” comparisons in fish community structure and fish health assessment can be made to more accurately determine potential effects from the input of ECs by the WWTP. Research has found a range of abnormalities in fish populations (vitellogenin induction in males and juvenile females, development of oocytes in testes, etc.) downstream of WWTPs (Mills and Chichester, 2005).

(9) A major change is anticipated to the primary source of ECs in the system. By around 2010, the WWTP is scheduled to close, with the waste currently treated at this plant being piped to a larger nearby WWTP. This closure provides a unique opportunity to examine how a stream and aquatic biota react to the removal of the primary source of ECs and allows a novel “before” and “after” assessment that has not been previously available in EC research.

Conducting Fate and Effects Research on Fourmile Creek

A first step in an investigation of chemical transport and fate is the collection of streamflow data that is critical to understanding the flow dynamics of a stream system (Barber and others, 2005). Streamflow—the amount of water (volume) passing through a stream cross-section in a given point in time — is important for understanding the types of flow (droughts to floods) and patterns of flow (diurnal to seasonal) that can occur at a site and within a drainage basin. Streamflow in a basin often displays a unique “pattern” over time based on numerous factors including: land use, vegetation, water inputs, soil types and slope. Streamflow is the primary transport mechanism for the movement of chemicals in a drainage basin once input into the stream. Knowledge of streamflow and chemical concentrations in a stream makes it possible to determine mass fluxes or chemicals loads in the stream under various flow conditions. Early tasks for the Fourmile Creek study site included the installation of a USGS gauging station (station number 05485606, site 3) downstream of the WWTP and a USGS staff plate (for measuring stream stage) at the first bridge upstream of the WWTP (station number 05485600, site 0) (Figure 3).

Figure 3. Location of the Fourmile Creek drainage basin in central Iowa and sampling sites along the Fourmile Creek stream reach.



Another critical component for research on chemical transport is determining how long it takes for water to flow through a stream reach under varying flow conditions. The most accurate method of determining travel times in a stream is by direct measurement using dye tracers. (Kilpatrick and Wilson, 1989; Jobson, 2000). Typically, dye tracing and time of travel involves the injection of a known volume of dye at an upstream location and the measurement of the dye plume at strategic locations downstream. Fluorescent, nontoxic dyes are used in dye tracing studies and the degree of fluorescence in the water sample can be determined with a fluorometer (Wilson, 1967). The concentration of dye in the sample is directly proportional to its fluorescence. A plot of concentration against time defines the dye-response curve, between sampling sites. The time of travel is measured by observing the time required for the movement of the “dye cloud,” as defined by the response curve, between sampling sites. Knowing travel times from dye tracing, it is then possible to extrapolate travel times to other flow conditions within the stream (Jobson, 2000). This allows investigators to more accurately predict travel times over a range of flow conditions. In addition, the dye tracing provides accurate time-of-travel data that can be used in various chemical transport models over a range of flow conditions. To accurately determine travel times between strategic locations along the study reach, dye-tracing tests were conducted at Fourmile Creek in August of 2003 and in March of 2005 (Figures 4 and 5).

Figure 4A. Injection of dye at Fourmile Creek near Ankeny, Iowa at the wastewater treatment plant outfall, March, 2005.



Figure 4B. Leading edge of the dye concentration at Fourmile Creek near Ankeny, Iowa, site 3, March 2005.



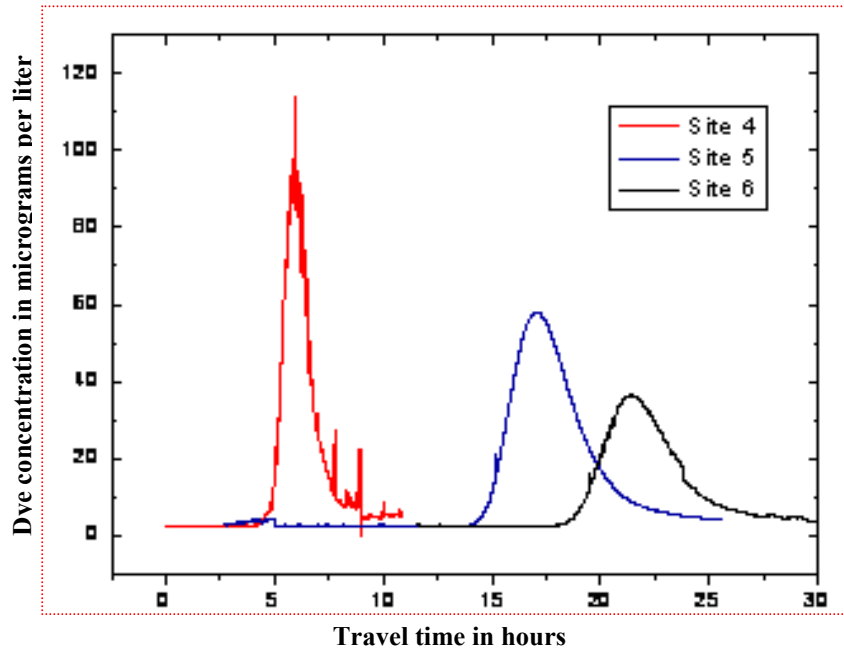
Figure 4C. Peak dye concentration at Fourmile Creek near Ankeny, Iowa, site3, March 2005.



Figure 4D. Trailing edge of the dye concentration at Fourmile Creek near Ankeny, Iowa, site 3, March 2005.



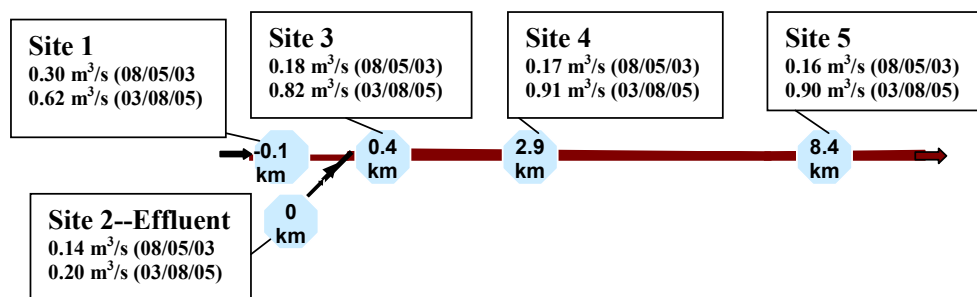
Figure 5. As the dye tracer moves downstream it undergoes dispersive mixing which results in spreading of the peak width and attenuation of peak concentrations without loss of mass.



Time-of-travel studies provide critical information for Lagrangian sampling of streams. Lagrangian transport examines the transport of any number of dissolved constituents that move with water in a stream. In a Lagrangian sample set, the same mass of water is tracked and sampled as the water migrates downstream. Once the time of travel information is established for a particular stream reach, the timing of the Lagrangian sample collection sets can readily be determined over a range of flow conditions. Lagrangian sample sets are more useful than traditional samples sets for constructing transport models of dissolved chemicals and suspended sediment (Meade and Stevens, 1990; Moody, 1993) and for identifying in-stream processes that affect stream chemistry (Hanor, 1988). For example, Lagrangian sample sets have been used to better understand the magnitude of subsequent in-stream transformations of nitrate (NO_3) in the Mississippi River (Battaglin and others, 2001) and the fate of atrazine in Roberts Creek (Kolpin and Kalkhoff, 1991; Kolpin and Kalkhoff, 1993).

To date, two sets of Lagrangian samples have been collected from Fourmile Creek representing low-flow/warm water conditions (August 2003) and medium flow/cold water conditions (March 2005) (Figure 6). The complete set of chemical analyses (representing measurements of 150 chemicals) are still in progress. By comparing the water-quality results between the Lagrangian samples collected in 2003 (warm water, low-flow conditions) to those in 2005 (cold water, medium-flow conditions), an increased understanding of processes (e.g. dilution, degradation, sorption, etc.) that are taking place within Fourmile Creek may be possible.

Figure 6. Schematic diagram of streamflow system at Fourmile Creek near Ankeny, Iowa and sampling sites during the Lagrangian sampling August 5, 2003 and March 8, 2005.



Since sediment has also been shown to be a reservoir for ECs (Furlong et al., 2004) both water and bed sediment samples were collected and measured for ECs during the two sampling events. To compliment the set of data from the Lagrangian water samples and streambed sediment samples, semipermeable membrane devices (SPMDs), for hydrophobic contaminants (Huckins and others, 2002; http://wwwaux.cerc.cr.usgs.gov/spmd/spmd_overview.htm) and polar organic chemical interactive sampler (POCIS) (<http://www.cerc.usgs.gov/pubs/center/pdfDocs/POCIS.pdf>) for hydrophilic contaminants (Alvarez and others, 2004; Jones-Lepp and others, 2004) were deployed for 28 days (based on work by Alvarez and others, 2004) at select sampling sites on Fourmile Creek in March of 2005. These passive samplers have the advantage of integrating large volumes of water for an extended period of time. Thus, these passive samplers can identify the presence of extremely low concentrations of contaminants (by accumulating them through time) or those contaminants that may only be present during episodic events that may be missed by instantaneous water samples. These time-weighted average concentrations are also fundamental to ecologic risk assessment for chemical stressors. To determine potential effects on fish from chemicals inputs by WWTPs, community structure (Moulton et al., 2002) and fish health assessment (Schmitt et al., 1999) were conducted at three sites in the stream reach (Figure 2). As conditions allow, fish health assessment will be conducted on two species: white sucker (*Catostomus commersoni*) and fathead minnow (*Pimephales promelas*).

Future Work

Future work on ECs will involve not only the occurrence of these compounds, but also their fate, transport and possible effects in the environment. Several large-scale studies in the United States by the USGS Toxic Substances Hydrology Program have already documented the occurrence of ECs in the environment (Kolpin and others 2002; Barnes and others, 2002; Furlong and others, 2004; Focazio and others, 2004; Barnes and others, 2005). These studies have shown that a wide variety of ECs are commonly detected in streams, streambed sediment and ground water as complex mixtures of compounds. Other studies have documented the occurrence of ECs globally (Kummerer, 2004a). Many of these same EC compounds have been detected in a study of Iowa's streams (Kolpin and others, 2004). Indeed, the data on ECs collected at Fourmile Creek near Ankeny, Iowa to date are consistent with similar national studies. However, the effects of long-term, low-level exposure to these mixtures of emerging contaminants on aquatic life and humans are currently unknown. Research on the effects of ECs in the environment is only in the beginning stages.

The field research site established at Fourmile Creek near Ankeny, Iowa, will continue to build a framework for better understanding of the transport, fate and effects of ECs in the environment. One goal of the field research site at Fourmile Creek is to move beyond documenting the occurrence of these compounds to

examine what happens to these compounds once they enter the environment and their potential effects to aquatic ecosystems. Work to date has proceeded with streamflow data, dye tracing/time of travel studies, Lagrangian water-quality sampling, and fish community and fish health assessment. The integration of chemical and biological research within the same stream reach will allow a greater understanding of the fate and effects of ECs.

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