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Dissolved Pesticides, Dissolved Organic Carbon, and Water-Quality Characteristics in Selected Idaho Streams, April–December 2010

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U.S. Department of the Interior
U.S. Geological Survey

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Conversion Factors

SI to Inch/Pound

Multiply	By	To obtain
	Length	
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
	Area	
square kilometer (km^2)	0.3861	square mile (mi^2)
	Volume	
liter (L)	1.057	quart (qt)
liter (L)	0.2642	gallon (gal)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound, avoirdupois (lb)
microgram (µg)	3.527×10^{-8}	ounce, avoirdupois (oz)
nanogram (ng)	3.527×10^{-11}	ounce, avoirdupois (oz)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

°F=(1.8×°C)+32

Vertical coordinate information is referenced to the North American Vertical Datum of 1988 (NAVD 88).

Horizontal coordinate information is referenced to the North American Datum of 1983 (NAD 83).

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius (µS/cm at 25°C).

Concentrations of chemical constituents in water are given in milligrams per liter (mg/L), micrograms per liter (µg/L), or nanograms per liter (ng/L).

Unit Abbreviations

°C	Degrees Celsius
°C/min	Degrees Celsius per minute
cm^3	cubic centimeters
dd	decimal degrees
g	gram
L	liter
m	meter
mg	milligrams
mg/L	milligrams per liter
min	minute
mL	milliliter
mm	millimeter
ng/L	nanograms per liter
ng/µL	nanograms per microliter
NTU	Nephelometric turbidity unit
µg/kg	micrograms per kilogram
µL	microliter
µm	micrometer
µS/cm	microsiemens per centimeter at 25 degrees Celsius

Acronyms

DCM	dichloromethane
DOC	dissolved organic carbon
GC-EIMS	gas chromatograph/election ionization mass spectrometer
GC-ITMS	gas chromatograph/ion trap mass spectrometer
GF/F	glass fiber filter
H_2SO_4	sulfuric acid
i.d.	inner diameter
KHP	potassium hydrogen phthalate
LOD	limit of detection
MDL	method detection limit
N_2	nitrogen gas
Na_2SO_4	sodium sulfate

ND	not detected
NWIS	National Water-Quality Information System
QA/QC	quality assurance/quality control
SPE	solid-phase extraction
SD	standard deviation
SIM	selective ion monitoring
SIS	selective ion storage
USDA	U.S. Department of Agriculture
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey

Dissolved Pesticides, Dissolved Organic Carbon, and Water-Quality Characteristics in Selected Idaho Streams, April–December 2010

By Timothy J. Reilly[1], Kelly L. Smalling[1], Emma R. Wilson[2], and William A. Battaglin[1]

Abstract

Water-quality samples were collected from April through December 2010 from four streams in Idaho and analyzed for a suite of pesticides, including fungicides, by the U.S. Geological Survey. Water samples were collected from two agricultural and two nonagricultural (control) streams approximately biweekly from the beginning of the growing season (April) through the end of the calendar year (December). Samples were analyzed for 90 pesticides using gas chromatography/mass spectrometry. Twenty-three pesticides, including 8 fungicides, 10 herbicides, 3 insecticides, and 2 pesticide degradates, were detected in 45 water samples. The most frequently detected compounds in the two agricultural streams and their detection frequencies were metolachlor, 96 percent; azoxystrobin, 79 percent; boscalid, 79 percent; atrazine, 46 percent; pendimethalin, 33 percent; and trifluralin, 33 percent. Dissolved-pesticide concentrations ranged from below instrumental limits of detection (0.5–1.0 nanograms per liter) to 771 nanograms per liter (hexazinone). The total number of pesticides detected in any given water sample ranged from 0 to 11. Only three pesticides (atrazine, fipronil, and simazine) were detected in samples from the control streams during the sampling period.

Introduction

Fungicides are used to prevent the outbreak of persistent, historically significant plant diseases like late blight (caused by *Phytophthora infestans* and responsible for the Irish Potato Famine of 1846) and newer plant diseases, such as Asian Soy Rust. Both late blight and Asian Soy Rust are potentially devastating if not controlled (Leadbeater and Gisi, 2009). Of the more than 67,000 pesticide products currently registered for use in the United States, more than 3,600 are used to combat fungal diseases (U.S. Environmental Protection Agency, 2011). Even with the use of chemical-based crop-protection measures, fungal pathogens were responsible for 7 to 24 percent of losses in yields to commodity crops, such as potatoes, worldwide during 2001–03 (Oercke, 2006). Pesticide manufacturers are continually developing new fungicides to find more effective treatments and to outpace the rate at which pathogens acquire resistance to these chemicals. For example, *Phytophthora infestans* rapidly developed strains resistant to widely used phenylamide fungicides, leading to its classification as a high-risk pathogen and necessitating co-application of other fungicides to provide the necessary crop protection (Brent and Hollomon, 2007). Depending on the pathogen genome and the mode of fungicidal action (single versus multiple pathways), pathogens can develop resistance to newly introduced fungicides within a few years of exposure. For example, field and laboratory studies identified *Alternaria alernata* strains (responsible for *Alternaria* late blight of pistachio crops) resistant to boscalid within 2 years of registration and to azoxystrobin within 3 to 4 years of continuous application (Ma and Others, 2003; Avenot and Michailides, 2007).

Relatively little is known about the fate and effects of fungicides at environmentally relevant concentrations in the aquatic environment. Recent studies have documented the presence of some fungicides in runoff from greenhouse production and commercial foliage plant nurseries (Roseth and Haarstad, 2010; Wilson and Riiska, 2010), soil and water associated with banana production (Geissen and others, 2010), streams and bed sediment (Battaglin and others, 2010; Smalling and Orlando, 2011), and the atmosphere (Schummer and others, 2010). Even less is known about the presence in the environment of recently registered fungicides. For example, the presence of boscalid (first registered for use in the United States in 2003) has been documented in only four studies; it has been found in streams and groundwater (Smalling and Orlando, 2011; Reilly and others, in press) using field experiments to determine soil dissipation and residuals (Chen and Zhang, 2010), and in the atmosphere (Schummer and others, 2010).

The potential for chronic exposure of nontarget fungi to fungicides at environmentally relevant concentrations exists but has not been extensively evaluated. Unlike most other pesticides, multiple fungicides typically are applied as

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[1] U.S. Geological Survey

[2] Boise State University

a prophylactic crop protectant more than 10 times per season (depending on conditions and crop type) but at lower application rates than herbicides or insecticides. This difference in usage increases the likelihood of chronic exposure of aquatic ecosystems to low concentrations of fungicides. The effects of fungicides at environmentally relevant concentrations on aquatic organisms and communities are poorly understood. However, recent studies have documented the potential effects of fungicides on amphibians and macroinvertebrate communities (Schäfer and others, 2011; Belden and others, 2010). The genotoxic, teratogenic, and endocrine-disrupting properties of several fungicides have been established by field, mesocosm, *in vivo* (zebrafish and human lymphocytes), and *in vitro* studies (Bony and others, 2008; Şişman and Türkez, 2010; Orton and others, 2011; Taxvig and others, 2008).

Aside from studies conducted by manufacturers during the registration process, data on the effects of fungicides on aquatic organisms and nontarget fungi are limited. Environmental concentrations of fungicides are typically less than 100 nanograms per liter (ng/L) (Battaglin and others, 2010; Smalling and Orlando, 2011). On the basis of current literature, the effect on aquatic communities of fungicides at these concentration levels is likely to be sublethal or an indirect disruption of community structure by altering fungal, phytoplankton, and zooplankton populations. Nondisease (nontarget) fungi are known to be sensitive to antifungal substances (including fungicides), and changes to their community structure could affect key food-web dynamics, as well as critical nutrient and carbon-cycling pathways (Barron, 2003; Gleason and Marano, 2010).

A study was conducted by the U.S. Geological Survey (USGS) Toxic Substances Hydrology (Toxics) Program to measure water-quality characteristics and the concentrations of currently used pesticides (especially fungicides) and dissolved organic carbon in selected streams in Idaho. In 2010, field sampling focused on two streams with agriculture in the surrounding areas and with unknown insect and gut fungi populations and two streams with little, if any, agriculture and that contain populations of insects with robust gut fungi populations. Water-quality results from this study can be used to focus future efforts of the USGS Toxics Program and other researchers to better understand the effects of pesticides (particularly fungicides) on native fungi and the insects that they inhabit.

Purpose and Scope

This report describes the methods and procedures used to determine pesticide concentrations in 45 water samples collected from 4 stream sites in Idaho in April to December 2010. Water samples were analyzed for a suite of 90 pesticides; concentrations of 23 pesticides are listed in a table. Concentrations of dissolved organic carbon and values of water-quality characteristics are also presented.

Description of Sampling Sites and Watershed Characteristics

Two of the four streams selected for this study drain agricultural land (hereafter referred to as agricultural streams)—Sand Run Gulch @ Hwy 95 Xing nr Parma ID (station 13210360) and Ditch nr Wanstad Rd nr Parma ID (station 13213008)—and two streams (hereafter referred to as control streams) drain nonagricultural land. The two streams that drain nonagricultural land—Cottonwood Creek nr Boise, ID (station 433711116110700) and Dry Creek nr Bogus Basin Rd nr Boise, ID (station 434006116112100)—contain insect larvae (including black flies) with robust populations of gut fungi. Sampling locations are shown in figure 1, and watershed characteristics are listed in table 1.

Table 1. Description of four surface-water sampling sites and associated watershed characteristics in Idaho.

[Horizontal datum is North American Datum of 1983. Dominant crops and percentage of watershed in agriculture from U.S. Department of Agriculture (2011). NA, not applicable; <, less than]

USGS station number	USGS station name	Latitude	Longitude	Watershed area (acres)	Percentage of watershed in agriculture	Dominant crops (Percentage of agricultural acreage)
13210360	Sand Run Gulch at Highway 95 crossing near Parma, ID	43.764	-116.912	50,877	37.1	Alfalfa (25.0), corn (15.9), winter wheat (14.3)
13213008	Ditch near Wanstad Road near Parma, ID	43.803	-116.957	760	90.5	Winter wheat (27.5), hay (18.1), corn (17.7)
433711116110700	Cottonwood Creek near Boise, ID	43.620	-116.185	10,613	0.1	NA
434006116112100	Dry Creek near Bogus Basin Road near Boise, ID	43.668	-116.189	6,171	<0.1	NA

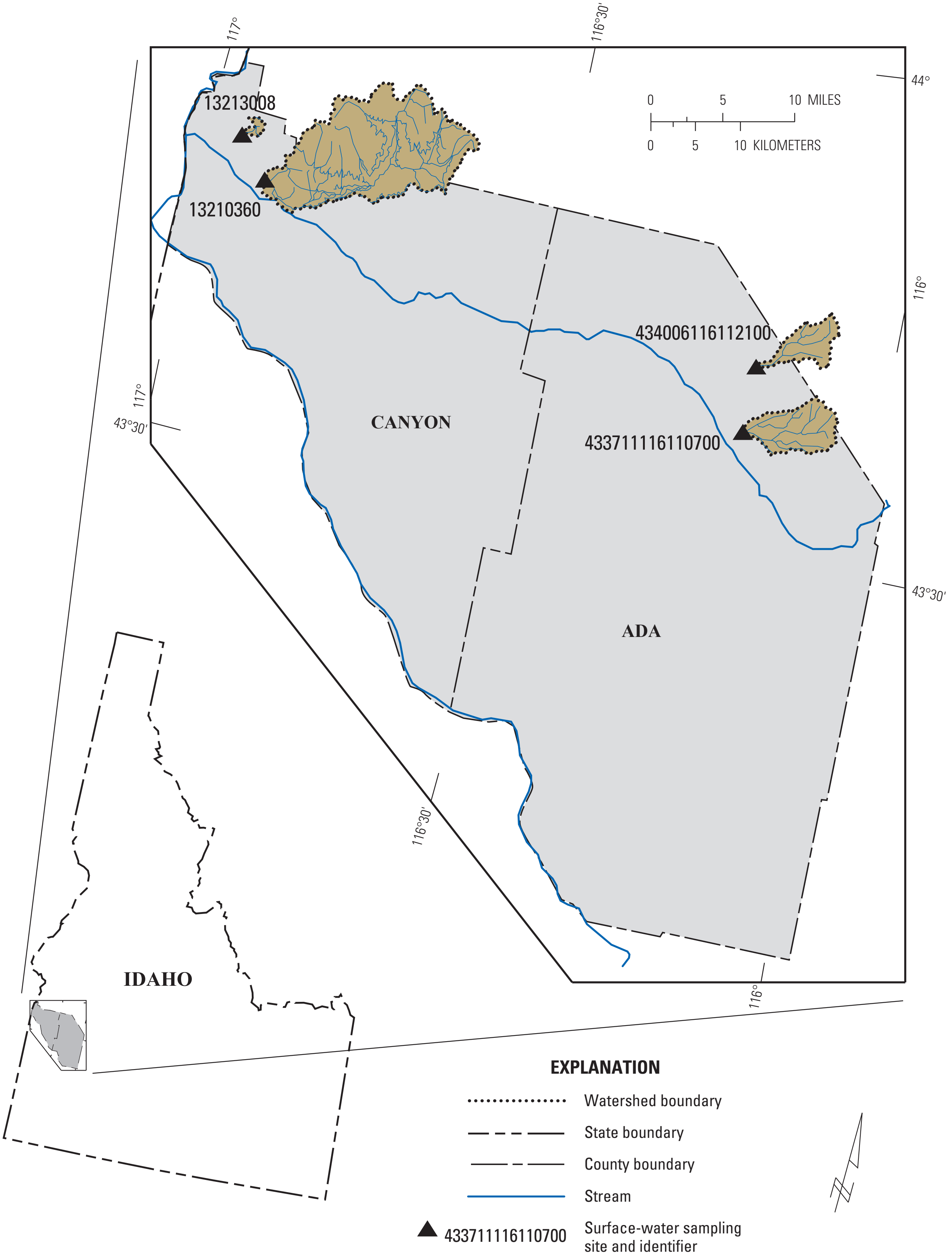

Figure 1. Location of four surface-water sampling sites and associated watersheds in Idaho.

Methods of Study

Watershed Delineation and Land-Use Characterization

Watersheds were delineated using ArcGIS Desktop GIS software (ESRI, Redlands, California). Initial watershed boundaries were taken from a preexisting dataset of national watershed boundaries (Natural Resources Conservation Service, 2009). The boundaries were then modified to match specific sampling sites by digitizing the boundaries on-screen using high-resolution aerial photographs, digital topographic maps, and detailed hydrography as a backdrop. The 2010 Idaho cropland data layer (U.S. Department of Agriculture, 2011; figs. 2 and 3) was used to calculate the crop statistics shown in table 1. This data layer has a spatial resolution of approximately 30 meters (m). In most cases the land-use categories shown represent several defined classifications in the cropland data layer. For example, the forest category in the figures is composed of deciduous forest, evergreen forest, and forest land categories from the cropland data layer.

Sampling Methods

Twelve water samples were collected from each of the agricultural streams, and 10 and 11 samples were collected from Dry Creek and Cottonwood Creek, respectively. Samples were collected approximately biweekly starting in April for the two agricultural streams and Cottonwood Creek and starting in June for Dry Creek. The period of sample collection spans the typical growing season in Idaho. Sample collection did not target runoff events or other specific hydrologic conditions.

Water-quality sampling was conducted following the same procedures at all sites. Samples were collected by immersing pre-cleaned, amber glass bottles—1 liter (L) for pesticides and 125 milliliters (mL) for dissolved organic carbon (DOC)—once at each site to a depth of not less than 0.1 m below the water surface. A 1-L polyurethane bottle was rinsed three times with streamwater, then filled for determination of basic water-quality characteristics (specific conductance in microseimens per centimeter at 25 degrees Celsius (µS/cm), pH in standard units, and turbidity in nepholometric turbidity units (NTU)). At the time of sample collection, water temperature was measured with an alcohol-filled thermometer. Samples were then packed on ice and shipped overnight to the USGS Organic Chemistry Laboratory in Sacramento, California, for extraction and analysis.

Analysis of Dissolved Pesticides

All water samples were filtered in the laboratory using 0.7-micrometer (µm) glass fiber filters (GF/F) (Whatman, Florham Park, New Jersey) to remove suspended material. Filtered water samples were analyzed for a suite of 90 pesticides by extracting 1 L of sample water onto OasisTM hydrophilic-lipophilic-balanced (HLB) solid-phase extraction (SPE) cartridges (6 cubic centimeters (cm³), 500 milligrams (mg), 60 µm; Waters Corporation, Milford, Massachusetts). All samples were spiked with ring-$^{13}C_3$-atrazine and diazinon-diethyl-d_{10} (Cambridge Isotopes, Andover, Massachusetts) as recovery surrogates prior to cartridge extraction. Following extraction, the SPE cartridges were dried with carbon dioxide, eluted with 12 mL of ethyl acetate, and reduced under nitrogen. After extraction, approximately 1 gram (g) of sodium sulfate (Na_2SO_4) was added to the sample bottles to remove any residual water, and the bottles were rinsed three times with dichloromethane (DCM). The bottle rinses were reduced to 1 mL under nitrogen and combined with the ethyl acetate fraction. The entire sample (bottle rinse plus SPE eluate) was reduced to a final volume of 200 µL (microliters) for analysis. Acenaphthene-d_{10} and pyrene-d_{10} (used as internal standards) were added to all samples prior to instrumental analysis. All sample extracts were analyzed for herbicides and insecticides by gas chromatography/mass spectrometry. Additional details are given in Hladik and others (2008).

Water-sample extracts (1-µL injection volume) were analyzed for herbicides and insecticides using a Varian Saturn 2000 (Walnut Creek, California) gas chromatograph (GC)–ion trap mass spectrometer (ITMS). Analyte separation on the GC was achieved using a 30 m x 0.25 millimeter (mm) inner diameter (i.d.), 0.25-µm DB-5ms fused silica column (Agilent Technologies, Folsom, California) with helium as the carrier gas. The temperature of the splitless injector was held constant at 275 degrees Celsius (°C). The temperature program was 80ºC (hold 0.5 minute (min)), increase to 120ºC at 10ºC/min, increase to 200ºC at 3ºC/min (hold 5 min), increase to 219ºC at 3ºC/min, and finally increase to 300ºC at 10ºC/min (hold 10 min). The transfer line and ion trap temperatures were 280ºC and 220ºC, respectively. The ITMS was operated in electron ionization (EI) mode with an emission current of 15 microamperes (µA) and no offset when run in full-scan mode, and an emission current of 45 µA with a multiplier offset of 300 volts when using selective ion storage (SIS) windows. Data were collected in full scan and SIS modes. Complete details of the analytical method are described in Hladik and others (2008) and Smalling and Orlando (2011).

Sample extracts were analyzed for fungicides using an Agilent 5975 GC/electron ionization mass spectrometer (EIMS) (Folsom, California). Analyte separation was achieved using a 30-m x 0.25-mm i.d., 0.25-µm DB-5ms fused silica column (Agilent Technologies, Folsom, Calif.) with helium as the carrier gas. The temperature of the splitless injector was held constant at 275ºC. The temperature program was 80ºC (hold 0.5 min), increase to 180ºC at 10ºC/min, increase to 220ºC at 5ºC/min (hold 1 min), increase to 280ºC at 4ºC/min (hold 1 min), and finally increase to 300ºC at 10ºC/min (hold 10 min). The transfer-line, quadrupole, and source temperatures were 280ºC, 150ºC, and 230ºC, respectively. Data for all fungicides were collected in selective ion monitoring (SIM) mode with each compound having one quantifier ion and one or two qualifier ions.

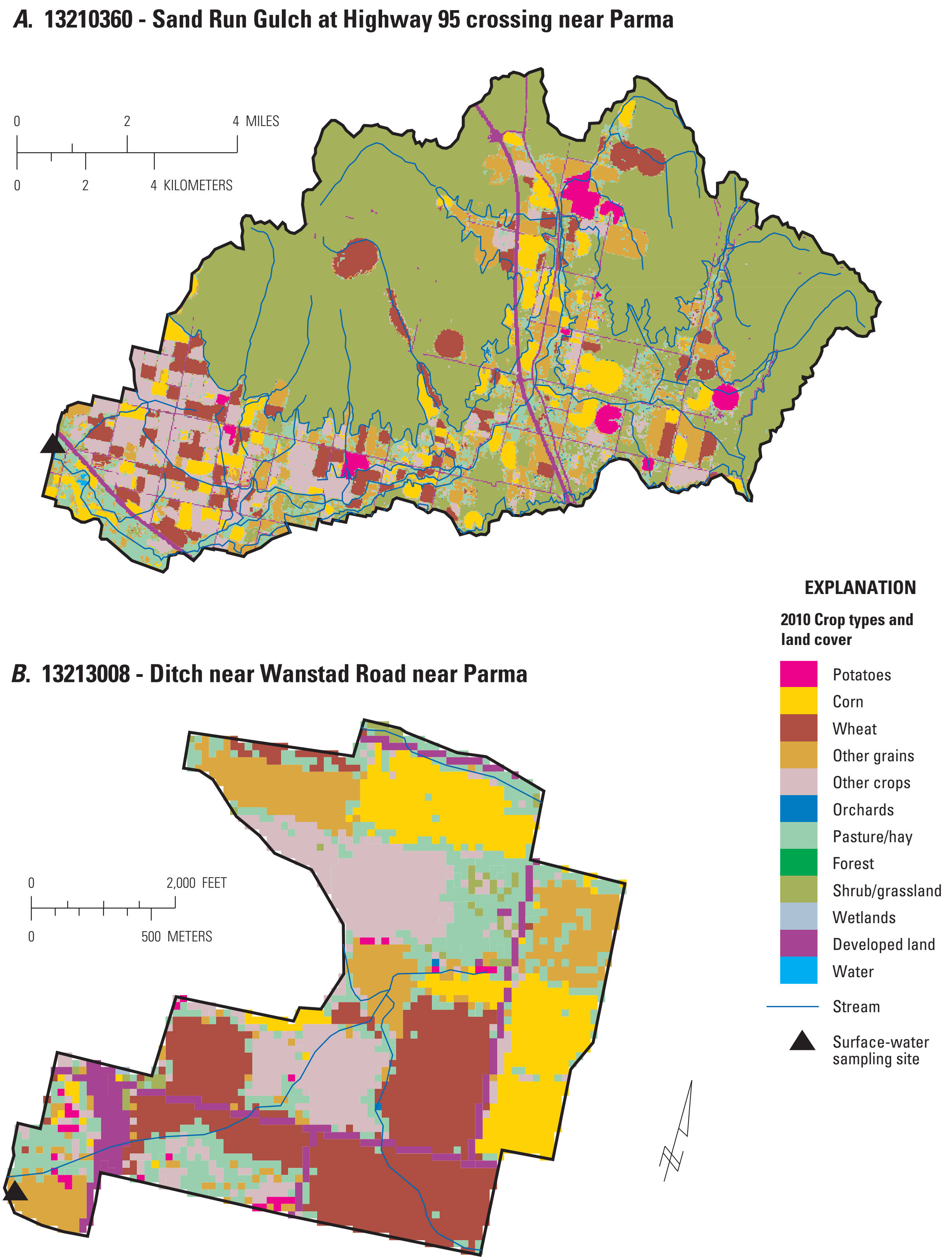

Figure 2. Crop and land-cover types for (A) Sand Run Gulch at Highway 95 crossing near Parma (USGS station number 13210360) and (B) Ditch near Wanstad Road near Parma (USGS station number 13213008) in Idaho.

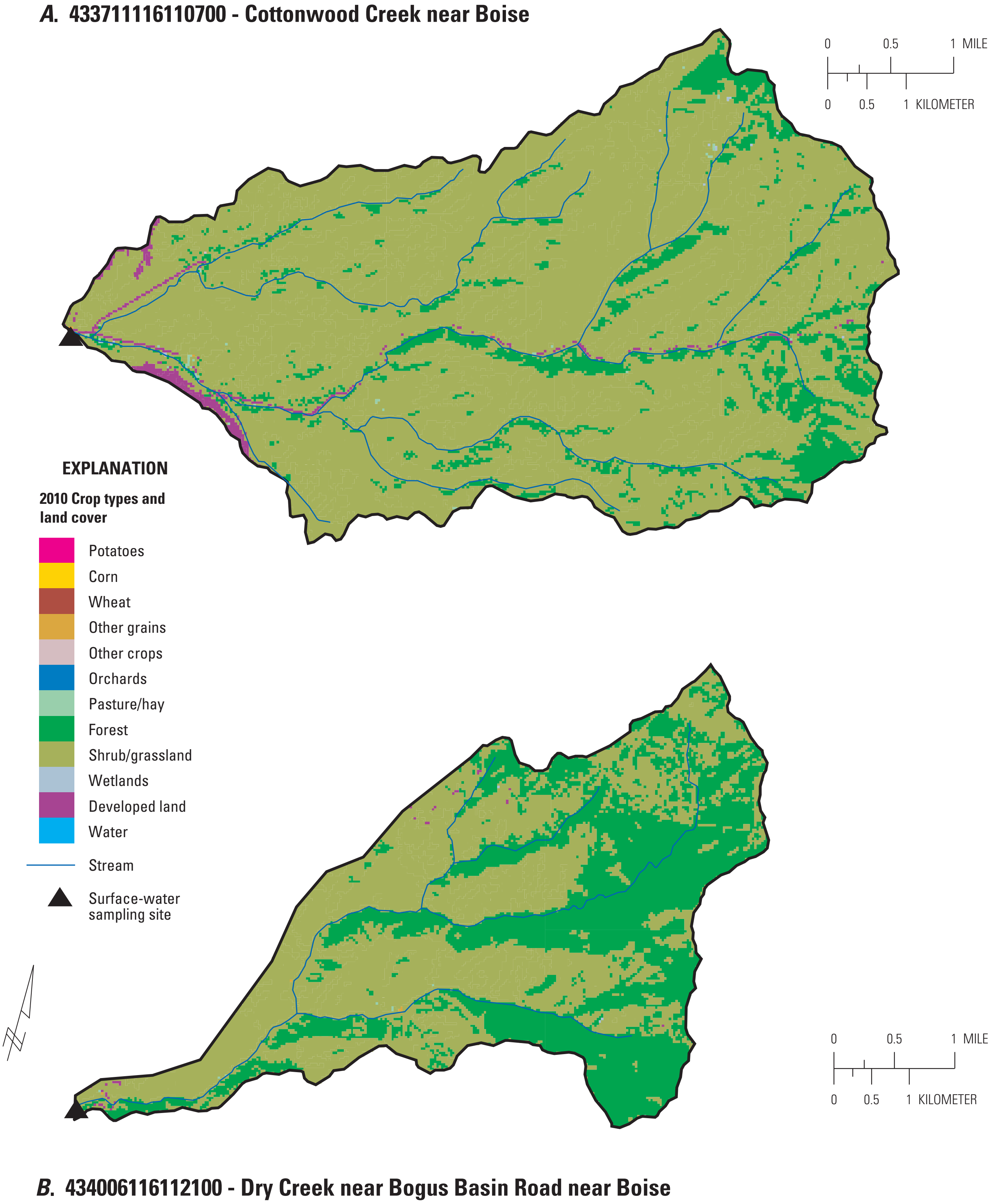

Figure 3. Crop and land-cover types for sampling sites (A) Cottonwood Creek near Boise (USGS station number 433711116110700) and (B) Dry Creek near Bogus Basin Road near Boise (USGS station number 434006116112100) in Idaho.

Instrument Calibration

Calibration of both instruments (GC-ITMS and GC-EIMS) was achieved using calibration standards that spanned the linear range of instrument response. An eight-point calibration standard was developed with concentrations ranging from 0.025 to 5.0 nanograms per microliter (ng/µL). Calibration curves were considered acceptable if the R^2 for each individual compound was greater than 0.995. The response of the instruments was monitored every six to eight samples with mid-level check standards. The instruments were considered to be stable if recovery of the check standards fell within the range of 80 to 115 percent of the nominal standard concentration. If environmental sample concentrations fell outside the linear range of the instrument, the samples were diluted appropriately and reanalyzed.

Quality Assurance/Quality Control

Dissolved-pesticide concentrations in water were validated against a comprehensive set of performance-based quality assurance/quality control (QA/QC) criteria, including laboratory blanks, matrix spikes, and surrogate recovery. All environmental and QC data were reviewed by project staff, and results for the target constituents analyzed under the same conditions were of acceptable quality.

Eight laboratory blanks (tap water) were processed to test the cleanliness of laboratory procedures. No pesticides were detected in any of the blank samples. Ring-$^{13}C_3$-atrazine and diazinon-diethyl-d_{10} were used as recovery surrogates to assess the efficiency of sample extraction. Sample data were excluded if recovery was less than 70 percent. Recovery of surrogates for all samples analyzed (including QC samples) ranged from 73 to 118 percent with a mean (± standard deviation) for ring-$^{13}C_3$-atrazine and diazinon-diethyl-d_{10} of 89 ± 9 percent and 93 ± 12 percent, respectively. Six samples were spiked in the laboratory with a suite of 90 pesticides, and the percent recovery ranged from 78 to 110 percent with a median of 92 percent. This test measured the efficiency of the extraction method, as well as the stability and holding times of all compounds during transport to the laboratory.

Method Detection Limits

Surface-water method detection limits (MDLs) were validated using the U.S. Environmental Protection Agency (USEPA) procedure described in 40 CFR Part 136 (U.S. Environmental Protection Agency, 1992). Water samples used to determine MDLs for insecticides and herbicides were collected in 2005 from the Sacramento River at Miller Park (Hladik and others, 2008), and water samples for fungicide MDLs were collected in 2008 from the American River near the California State University Campus. The MDLs were calculated for each compound using the following equation:

$$MDL = S \times t\,(n\text{-}1,\ 1\text{-}\alpha = 0.99)\ , \qquad (1)$$

where

MDL = method detection limit, in nanograms per liter;
S = standard deviation of replicate samples;
n = number of replicates; and
t = value of Student’s t statistic at 6 degrees of freedom and 99-percent confidence level.

MDLs for all compounds in water ranged from 0.9 to 10.5 ng/L, and instrumental limits of detection (LOD) ranged from 0.5 to 1.0 ng/L (table 2). Analytes detected at concentrations greater than the instrumental LOD but less than the MDL are reported as estimates.

Analysis of Dissolved Organic Carbon

All water samples were filtered in the laboratory using 0.7-µm GF/Fs (Whatman, Florham Park, New Jersey.) to remove suspended material. Filtered water samples were analyzed for dissolved organic carbon (DOC) by high-temperature catalytic combustion using a Shimadzu TOC-V_{CNS} total organic carbon analyzer (Shimadzu Scientific Instruments, Columbia, Maryland) according to a modified version of USEPA method 415.3 (Potter and Wimsatt, 2009). The instrument was calibrated using potassium hydrogen phthalate (KHP) standards prepared in organic-free water with concentrations ranging from 0.0 to 4.0 milligrams per liter (mg/L). All standards, blanks, and samples were acidified prior to analysis with approximately four drops of concentrated sulfuric acid (H_2SO_4) to lower the pH to less than 2.0. The accuracy and precision of the measurements were within 5 percent, as indicated by internal standards (KHP and caffeine), laboratory replicates, and matrix spikes (Bird and others, 2003; Potter and Wimsatt, 2009). The quantitative limit of detection for DOC was 0.3 mg/L.

Dissolved Pesticides in Streams

Twenty-three pesticides or pesticide degradates were detected in 45 water samples collected from four streams in Idaho (table 3). Maximum pesticide concentrations ranged from less than the instrumental limit of detection to 771 ng/L (for hexazinone). The maximum number of pesticides detected in a single water sample was 11. Twenty-three pesticides, including 8 fungicides, 10 herbicides, 3 insecticides, and 2 pesticide degradates, were detected in 24 water samples from the agricultural streams. The most frequently detected compounds and the detection frequencies in the two agricultural streams are metolachlor, 96 percent; azoxystrobin, 79 percent; boscalid, 79 percent; atrazine, 46 percent; pendimethalin, 33 percent; and trifluralin, 33 percent (table 4).

Table 2. Dissolved pesticides with types, method detection limits, and instrumental limits of detection.

[D, degradate; H, herbicide; I, insecticide; F, fungicide; S, synergist; ng/L, nanograms per liter]

Compound	Pesticide type	Method detection limit (ng/L)	Instrumenal limit of detection (ng/L)
3,4-Dichloroaniline (3,4 DCA)	D	8.3	1.0
3,5-Dichloroaniline (3,5 DCA)	D	7.6	1.0
Alachlor	H	1.7	1.0
Allethrin	I	6.0	1.0
Atrazine	H	2.3	0.5
Azoxystrobin	F	3.1	0.5
Bifenthrin	I	4.7	0.5
Boscalid	F	2.8	0.5
Butylate	H	1.8	1.0
Carbaryl	I	6.5	1.0
Carbofuran	I	3.1	1.0
Chlorothalonil	F	4.1	0.5
Chlorpyrifos	I	2.1	0.5
Clomazone	H	2.5	1.0
Cycloate	H	1.1	1.0
Cyfluthrin	I	5.2	1.0
Cypermethrin	I	5.6	1.0
Cyproconazole	F	4.7	0.5
Cyprodinil	F	7.4	1.0
DCPA (Dacthal)	H	2.0	0.5
Deltamethrin	I	3.5	1.0
Diazinon	I	0.9	1.0
Difenoconazole	F	10.5	1.0
(E)-Dimethomorph	F	6.0	1.0
EPTC (Eptam)	H	1.5	0.5
Esfenvalerate	I	3.9	0.5
Ethalfluralin	H	3.0	0.5
Etofenprox	I	2.2	1.0
Famoxadone	F	2.5	1.0
Fenarimol	F	6.5	1.0
Fenbuconazole	F	5.2	1.0
Fenhexamid	F	7.6	1.0
Fenpropathrin	I	4.1	1.0
Fipronil	I	2.9	0.5
Desulfinylfipronil	D	1.6	0.5
Fipronil sulfide	D	1.8	0.5
Fipronil sulfone	D	3.5	0.5
Fluazinam	F	4.4	1.0
Fludioxonil	F	7.3	1.0
Fluoxastrobin	F	4.2	1.0
Flusilazole	F	5.1	1.0
Flutriafol	F	4.5	1.0
Hexazinone	H	8.4	1.0
Imazalil	F	10.5	1.0
Iprodione	F	4.4	1.0

Table 2. Dissolved pesticides with types, method detection limits, and instrumental limits of detection.—Continued

[D, degradate; H, herbicide; I, insecticide; F, fungicide; S, synergist; ng/L, nanograms per liter]

Compound	Pesticide type	Method detection limit (ng/L)	Instrumenal limit of detection (ng/L)
Kresoxim-methyl	F	4.0	1.0
Lambda-Cyhalothrin	I	2.0	0.5
Malathion	I	3.7	1.0
Metconazole	F	5.2	0.5
Methidathion	I	7.2	1.0
Methoprene	I	6.4	1.0
Methyl parathion	I	3.4	1.0
Metolachlor	H	1.5	1.0
Molinate	H	3.2	1.0
Myclobutanil	F	6.0	0.5
Napropamide	H	8.2	1.0
Oxyfluorfen	H	3.1	1.0
p,p' DDD	D	4.1	0.5
p,p' DDE	D	3.6	0.5
p,p' DDT	I	4.0	0.5
Pebulate	H	2.3	0.5
Pendimethalin	H	2.3	0.5
Pentachloroanisole (PCA)	D	4.7	0.5
Pentachloronitrobenzene (PCNB)	F	3.1	0.5
Permethrin	I	3.4	0.5
Phenothrin (Sumithrin)	I	5.1	1.0
Phosmet	I	4.4	1.0
Piperonyl Butoxide	S	2.3	1.0
Prometon	H	2.5	1.0
Prometryn	H	1.8	1.0
Propanil	H	10.1	1.0
Propiconazole	F	5.0	0.5
Propyzamide	H	5.0	1.0
Pyraclostrobin	F	2.9	0.5
Pyrimethanil	F	4.1	0.5
Resmethrin	I	5.7	1.0
Simazine	H	5.0	1.0
Tebuconazole	F	3.7	0.5
Tefluthrin	I	4.2	1.0
Tetraconazole	F	5.6	0.5
Tetramethrin	I	2.9	1.0
Tau-Fluvalinate	I	5.3	1.0
Thiobencarb	H	1.9	0.5
Triadimefon	F	8.9	1.0
Triadimenol	F	8.0	1.0
Trifloxystrobin	F	4.7	0.5
Triflumizole	F	6.1	1.0
Trifluralin	H	2.1	0.5
Triticonazole	F	6.9	1.0
Zoxamide	F	3.5	1.0

Table 3. Dissolved pesticide concentrations measured in surface-water samples collected from sampling sites on four streams in Idaho, April–December 2010.

[Values are in nanograms per liter. Values in parenthesis ( ) are below method detection limits but above instrumental limits of detection and are estimates. The following compounds were analyzed but were not detected in any samples; 3,4-DCA, allethrin, bifenthrin, butylate, carbaryl, carbofuran, cycloate, cyfluthrin, cyhlothrin, cypermethrin, cyproconazole, cyprodinil, DCPA, deltamethrin, difenoconazole, dimethomorph, esfenvalerate, etofenprox, famoxadone, fenarimol, fenbuconazole, fenhexamide, fenpropathrin, fipronil desulfynil, fipronil sulfide, fipronil sulfone, fluazinam, fludioxinil, fluoxastrobin, flusilazole, flutriafol, iprodione, kresoxim-methyl, malathion, metconazole, methidathion, methoprene, methyl parathion, molinate, myclobutanil, napropamide, oxyfluorfen, p,p'-DDE, p,p'-DDT, PCA, PCNB, pebulate, permethrin, phenothrin, phosmet, piperonyl butoxide, prometon, prometryn, propanil, propyzamide, pyraclostrobin, resmethrin, tau-fluvalinate, tebuconazole, tefluthrin, tetramethrin, thiobencarb, triadimefon, triadimenol, trifloxystrobin, triticonazole and zoxamide. USGS, U.S. Geological Survey; ND, not detected]

USGS station number	USGS station name	Date	Time	3,5-DCA	Alachlor	Atrazine	Azoxystrobin	Boscalid
13213008	Ditch near Wantad Road near Parma, ID	4/13/2010	1400	8.84	ND	ND	3.37	36.6
		5/3/2010	1530	ND	14.1	4.65	4.04	13.3
		5/24/2010	1345	ND	ND	ND	4.20	7.00
		6/16/2010	1145	ND	ND	15.2	3.37	15.6
		7/6/2010	1340	ND	ND	ND	9.23	20.9
		7/26/2010	950	ND	ND	ND	31.0	11.6
		8/16/2010	1115	ND	ND	ND	40.4	21.0
		9/9/2010	1135	ND	ND	ND	3.43	11.7
		9/29/2010	907	ND	ND	5.00	ND	ND
		10/18/2010	1130	ND	ND	ND	(3.0)	4.60
		11/8/2010	1050	ND	ND	2.60	3.60	9.40
		11/29/2010	950	ND	ND	3.40	(2.2)	5.20
13210360	Sand Run Gulch at Highway 95 crossing near Parma, ID	4/13/2010	1430	ND	ND	9.60	(1.8)	8.20
		5/3/2010	1320	ND	ND	5.80	(2.2)	10.2
		5/24/2010	1430	ND	ND	ND	(1.4)	4.42
		6/16/2010	1145	ND	ND	28.1	ND	ND
		7/6/2010	1340	ND	ND	7.51	6.29	10.8
		7/26/2010	950	ND	ND	ND	11.4	16.0
		8/16/2010	1200	ND	ND	ND	20.2	37.8
		9/9/2010	1135	ND	ND	4.31	12.5	14.9
		9/29/2010	930	ND	ND	ND	ND	ND
		10/18/2010	945	ND	ND	ND	(2.6)	5.41
		11/8/2010	920	ND	ND	ND	ND	ND
		11/29/2010	1100	ND	ND	6.60	ND	ND
433711116110700	Cottonwood Creek near Boise, ID	4/21/2010	1525	ND	ND	ND	ND	ND
		5/13/2010	1100	ND	ND	ND	ND	ND
		6/2/2010	1300	ND	ND	ND	ND	ND
		6/22/2010	1300	ND	ND	ND	ND	ND
		7/12/2010	1200	ND	ND	ND	ND	ND
		8/2/2010	930	ND	ND	ND	ND	ND
		8/23/2010	900	ND	ND	ND	ND	ND
		9/15/2010	940	ND	ND	ND	ND	ND
		10/4/2010	1230	ND	ND	ND	ND	ND
		11/12/2010	1230	ND	ND	ND	ND	ND
		12/6/2010	1330	ND	ND	ND	ND	ND
434006116112100	Dry Creek near Bogus Basin Road near Boise, ID	6/2/2010	945	ND	ND	ND	ND	ND
		6/22/2010	1300	ND	ND	ND	ND	ND
		7/12/2010	1200	ND	ND	ND	ND	ND
		8/2/2010	930	ND	ND	ND	ND	ND
		8/23/2010	900	ND	ND	21.4	ND	ND
		9/15/2010	940	ND	ND	ND	ND	ND
		10/4/2010	1430	ND	ND	ND	ND	ND
		10/25/2010	845	ND	ND	ND	ND	ND
		11/12/2010	1030	ND	ND	ND	ND	ND
		12/6/2010	950	ND	ND	ND	ND	ND

Table 3. Dissolved pesticide concentrations measured in surface-water samples collected from sampling sites on four streams in Idaho, April–December 2010.—Continued

[Values are in nanograms per liter. Values in parenthesis ( ) are below method detection limits but above instrumental limits of detection and are estimates. The following compounds were analyzed but were not detected in any samples; 3,4-DCA, allethrin, bifenthrin, butylate, carbaryl, carbofuran, cycloate, cyfluthrin, cyhlothrin, cypermethrin, cyproconazole, cyprodinil, DCPA, deltamethrin, difenoconazole, dimethomorph, esfenvalerate, etofenprox, famoxadone, fenarimol, fenbuconazole, fenhexamide, fenpropathrin, fipronil desulfynil, fipronil sulfide, fipronil sulfone, fluazinam, fludioxinil, fluoxastrobin, flusilazole, flutriafol, iprodione, kresoxim-methyl, malathion, metconazole, methidathion, methoprene, methyl parathion, molinate, myclobutanil, napropamide, oxyfluorfen, p,p'-DDE, p,p'-DDT, PCA, PCNB, pebulate, permethrin, phenothrin, phosmet, piperonyl butoxide, prometon, prometryn, propanil, propyzamide, pyraclostrobin, resmethrin, tau-fluvalinate, tebuconazole, tefluthrin, tetramethrin, thiobencarb, triadimefon, triadimenol, trifloxystrobin, triticonazole and zoxamide. USGS, U.S. Geological Survey; ND, not detected]

USGS station number	Chlorothalonil	Chlorpyrifos	Clomazone	Diazinon	EPTC	Ethalfluralin	Fipronil	Hexazinone	Imazalil
13213008	ND	ND	ND	ND	ND	ND	ND	ND	176
	ND	ND	ND	ND	ND	13.5	ND	66.9	ND
	ND	5.00	ND	10.6	25.2	14.4	ND	46.2	ND
	(3.6)	ND	35.8	ND	21.1	ND	ND	ND	ND
	ND	ND	ND	ND	ND	5.49	ND	ND	ND
	ND	ND	ND	ND	ND	5.71	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
13210360	ND	ND	ND	ND	ND	ND	ND	771	205
	ND	ND	ND	ND	ND	9.0	ND	96.6	ND
	ND	ND	ND	ND	23.3	ND	ND	137	ND
	ND	ND	ND	ND	22.9	ND	ND	ND	ND
	ND	ND	ND	ND	9.54	5.89	ND	16.9	ND
	ND	ND	ND	ND	ND	3.20	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
433711116110700	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	14.4	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
434006116112100	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND

Table 3. Dissolved pesticide concentrations measured in surface-water samples collected from sampling sites on four streams in Idaho, April–December 2010.—Continued

[Values are in nanograms per liter. Values in parenthesis ( ) are below method detection limits but above instrumental limits of detection and are estimates. The following compounds were analyzed but were not detected in any samples; 3,4-DCA, allethrin, bifenthrin, butylate, carbaryl, carbofuran, cycloate, cyfluthrin, cyhlothrin, cypermethrin, cyproconazole, cyprodinil, DCPA, deltamethrin, difenoconazole, dimethomorph, esfenvalerate, etofenprox, famoxadone, fenarimol, fenbuconazole, fenhexamide, fenpropathrin, fipronil desulfynil, fipronil sulfide, fipronil sulfone, fluazinam, fludioxinil, fluoxastrobin, flusilazole, flutriafol, iprodione, kresoxim-methyl, malathion, metconazole, methidathion, methoprene, methyl parathion, molinate, myclobutanil, napropamide, oxyfluorfen, p,p'-DDE, p,p'-DDT, PCA, PCNB, pebulate, permethrin, phenothrin, phosmet, piperonyl butoxide, prometon, prometryn, propanil, propyzamide, pyraclostrobin, resmethrin, tau-fluvalinate, tebuconazole, tefluthrin, tetramethrin, thiobencarb, triadimefon, triadimenol, trifloxystrobin, triticonazole and zoxamide. USGS, U.S. Geological Survey; ND, not detected]

USGS station number	Metolachlor	p,p′-DDD	Pendimethalin	Propiconazole	Pyrimethanil	Simazine	Tetraconazole	Trifluralin	Triflumizole
13213008	28.8	ND	ND	ND	ND	ND	ND	(1.9)	ND
	259	17.0	51.3	ND	ND	13.9	ND	ND	ND
	114	ND	154	ND	ND	15.6	ND	2.2	ND
	113	ND	47.4	ND	ND	ND	ND	5.5	ND
	566	ND	31.9	ND	ND	ND	ND	40.9	ND
	84.1	ND	ND	ND	ND	ND	ND	3.9	ND
	321	ND	ND	ND	ND	ND	ND	5.2	ND
	71.3	ND	ND	ND	ND	ND	ND	ND	ND
	40.4	ND	ND	ND	ND	ND	ND	ND	ND
	36.4	ND	ND	ND	ND	ND	ND	ND	68.8
	37.0	ND	ND	ND	ND	ND	ND	ND	ND
	20.4	ND	ND	ND	ND	ND	ND	ND	ND
13210360	21.6	ND	ND	ND	ND	10.6	ND	ND	ND
	122	ND	34.2	ND	ND	ND	ND	ND	ND
	66.9	ND	28.5	ND	ND	ND	ND	ND	ND
	171	ND	38.9	ND	ND	ND	ND	ND	ND
	137	ND	45.9	ND	ND	ND	ND	(1.2)	ND
	78.4	ND	ND	ND	ND	ND	ND	(2.0)	ND
	16.2	ND	ND	ND	ND	ND	ND	ND	ND
	19.6	ND	ND	ND	ND	ND	ND	ND	ND
	11.0	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	(4.8)	5.2	ND	(4.8)	ND	150
	5.40	ND	ND	ND	ND	ND	ND	ND	ND
	2.80	ND	ND	ND	ND	ND	ND	ND	ND
433711116110700	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
434006116112100	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	142	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND
	ND	ND	ND	ND	ND	ND	ND	ND	ND

Table 4. Dissolved pesticides with type; detection frequency; and median, minimum, and maximum concentrations measured in surface-water samples collected from Sand Run Gulch at Highway 95 crossing near Parma, Idaho (USGS station number 13210360) and Ditch near Wanstad Road near Parma, Idaho (USGS station number 13213008), in Idaho, April–December, 2010.

[Values are in nanograms per liter. Values in parentheses ( ) are below method detection limits but above instrumental limits of detection and are estimates. ND, not detected]

Dissolved pesticide	Pesticide type	Detection frequency (in percent)	Concentration		
			Median	Minimum	Maximum
Metolachlor	Herbicide	96	66.9	2.80	566
Azoxystrobin	Fungicide	79	3.60	(1.4)	40.4
Boscalid	Fungicide	79	11.6	4.42	37.8
Atrazine	Herbicide	46	5.80	2.60	28.1
Pendimethalin	Herbicide	33	42.4	28.5	154
Trifluralin	Herbicide	33	3.04	(1.2)	40.9
Ethalfluralin	Herbicide	29	5.89	3.20	14.4
Hexazinone	Herbicide	25	81.7	16.9	771
EPTC	Herbicide	21	22.9	9.54	25.2
Simazine	Herbicide	13	13.9	10.6	15.6
Imazalil	Fungicide	8	191	176	205
Triflumizole	Fungicide	8	109	68.80	150
3,5-DCA	Degradate	4	8.84	8.84	8.84
Alachlor	Herbicide	4	14.1	14.1	14.1
Chlorothalonil	Fungicide	4	(3.6)	(3.6)	(3.6)
Chlorpyrifos	Insecticide	4	5.0	5.0	5.0
Clomazone	Herbicide	4	35.8	35.8	35.8
Diazinon	Insecticide	4	10.6	10.6	10.6
p,p'-DDD	Degradate	4	17.0	17.0	17.0
Propiconazole	Fungicide	4	(4.8)	(4.8)	(4.8)
Pyrimethanil	Fungicide	4	5.19	5.19	5.19
Tetraconazole	Fungicide	4	(4.8)	(4.8)	(4.8)
Fipronil	Insecticide	4	14.4	14.4	14.4

Dissolved Organic Carbon and Water-Quality Characteristics in Streams

Dissolved organic carbon (DOC) concentrations in samples collected during this study ranged from 1.65 to 4.32 mg/L. Results of the analysis of DOC and water-quality characteristics (temperature, specific conductance, and turbidity) for each sampling event are shown in table 5.

Table 5. Dissolved organic carbon concentrations and water-quality characteristics measured in surface-water samples collected from four streams in Idaho, April–December 2010.

[USGS, U.S. Geological Survey; DOC, dissolved organic carbon; mg/L, milligrams per liter; °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25 degrees Celsius; NTU, nephelometric turbidity units; --, no data]

USGS station number	USGS station name	Date	Time	DOC (mg/L)	Water temperature (°C)	Specific conductance (µS/cm)	Turbidity (NTU)
13213008	Ditch near Wanstad Road near Parma, ID	4/13/2010	1400	2.52	--	695	44.7
		5/3/2010	1530	3.19	12.0	323	88.1
		5/24/2010	1345	3.37	13.5	237	50.1
		6/16/2010	1145	2.68	14.5	250	75.1
		7/6/2010	1340	3.10	17.0	292	87.1
		7/26/2010	950	3.06	--	464	66.0
		8/16/2010	1115	3.91	18.0	443	32.6
		9/9/2010	1135	2.97	16.0	300	71.8
		9/29/2010	907	2.76	18.0	370	14.5
		10/18/2010	1130	4.23	--	540	24.0
		11/8/2010	1050	1.9	--	951	4.7
		11/29/2010	950	1.65	8.0	947	0.5
13210360	Sand Run Gulch at Highway 95 crossing near Parma, ID	4/13/2010	1430	2.69	11.5	854	28.4
		5/3/2010	1320	3.13	11.0	465	12.4
		5/24/2010	1430	3.51	13.5	370	17.6
		6/16/2010	1145	3.40	15.0	405	28.2
		7/6/2010	1340	3.69	16.0	420	44.9
		7/26/2010	950	3.79	19.0	289	134.0
		8/16/2010	1200	3.22	18.0	307	91.0
		9/9/2010	1135	3.47	16.0	451	11.5
		9/29/2010	930	2.98	16.0	494	15.7
		10/18/2010	945	2.01	--	614	108.0
		11/8/2010	920	2.67	9.5	669	7.8
		11/29/2010	1100	2.78	4.0	713	61.3

Table 5. Dissolved organic carbon concentrations and water-quality characteristics measured in surface-water samples collected from four streams in Idaho, April–December 2010.—Continued

[USGS, U.S. Geological Survey; DOC, dissolved organic carbon; mg/L, milligrams per liter; °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25 degrees Celsius; NTU, nephelometric turbidity units; --, no data]

USGS station number	USGS station name	Date	Time	DOC (mg/L)	Water temperature (°C)	Specific conductance (µS/cm)	Turbidity (NTU)
433711116110700	Cottonwood Creek near Boise, ID	4/21/2010	1525	3.71	--	121	16.7
		5/13/2010	1100	2.78	8	169	5.8
		6/2/2010	1300	3.93	11	136	12.3
		6/22/2010	1300	2.82	--	132	5.0
		7/12/2010	1200	3.05	14	211	5.6
		8/2/2010	930	2.97	12	339	12.4
		8/23/2010	900	2.87	12.7	350	10.2
		9/15/2010	940	2.98	15	378	1.1
		10/4/2010	1230	3.07	13	--	--
		11/12/2010	1230	3.26	6	401	0.6
		12/6/2010	1330	4.32	7	374	0.6
434006116112100	Dry Creek near Bogus Basin Road near Boise, ID	6/2/2010	945	3.91	8.4	112	9.4
		6/22/2010	1300	3.00	--	120	5.0
		7/12/2010	1200	2.61	13	141	5.3
		8/2/2010	930	2.35	16	152	5.0
		8/23/2010	900	2.58	13.3	155	4.2
		9/15/2010	940	2.06	12	168	2.4
		10/4/2010	1430	2.36	14	--	--
		10/25/2010	845	3.91	7	166	2.5
		11/12/2010	1030	1.67	2.5	167	1.1
		12/6/2010	950	2.61	2	161	0.3

Summary

The U.S. Geological Survey, as part of the Toxic Substances Hydrology Program, conducted a study to measure the concentrations of currently used pesticides (especially fungicides) in two agricultural streams known from a previous study to contain fungicides and two control streams draining nonagricultural areas in Idaho. Dissolved organic carbon concentrations and water-quality characteristics also were measured.

Water samples were collected from the four streams from April through December 2010 and analyzed for a suite of pesticides (including fungicides). Twenty-three pesticides, including 8 fungicides, 10 herbicides, 3 insecticides, and 2 pesticide degradates, were detected in 45 water samples. The most frequently detected compounds and the detection frequencies in the two agricultural streams are metolachlor, 96 percent; azoxystrobin, 79 percent; boscalid, 79 percent; atrazine, 46 percent; pendimethalin, 33 percent; and trifluralin, 33 percent. Concentrations of dissolved pesticides ranged from below instrumental limits of detection (0.5–1.0 nanograms per liter (ng/L)) to 771 ng/L (for hexazinone). Three pesticides (atrazine, fipronil, and simazine) were each detected once in the samples from the control streams.

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For additional information, write to:

Director
U.S. Geological Survey
New Jersey Water Science Center
Mountain View Office Park
810 Bear Tavern Rd., Suite 206
West Trenton, NJ 08628

or visit our Web site at:
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