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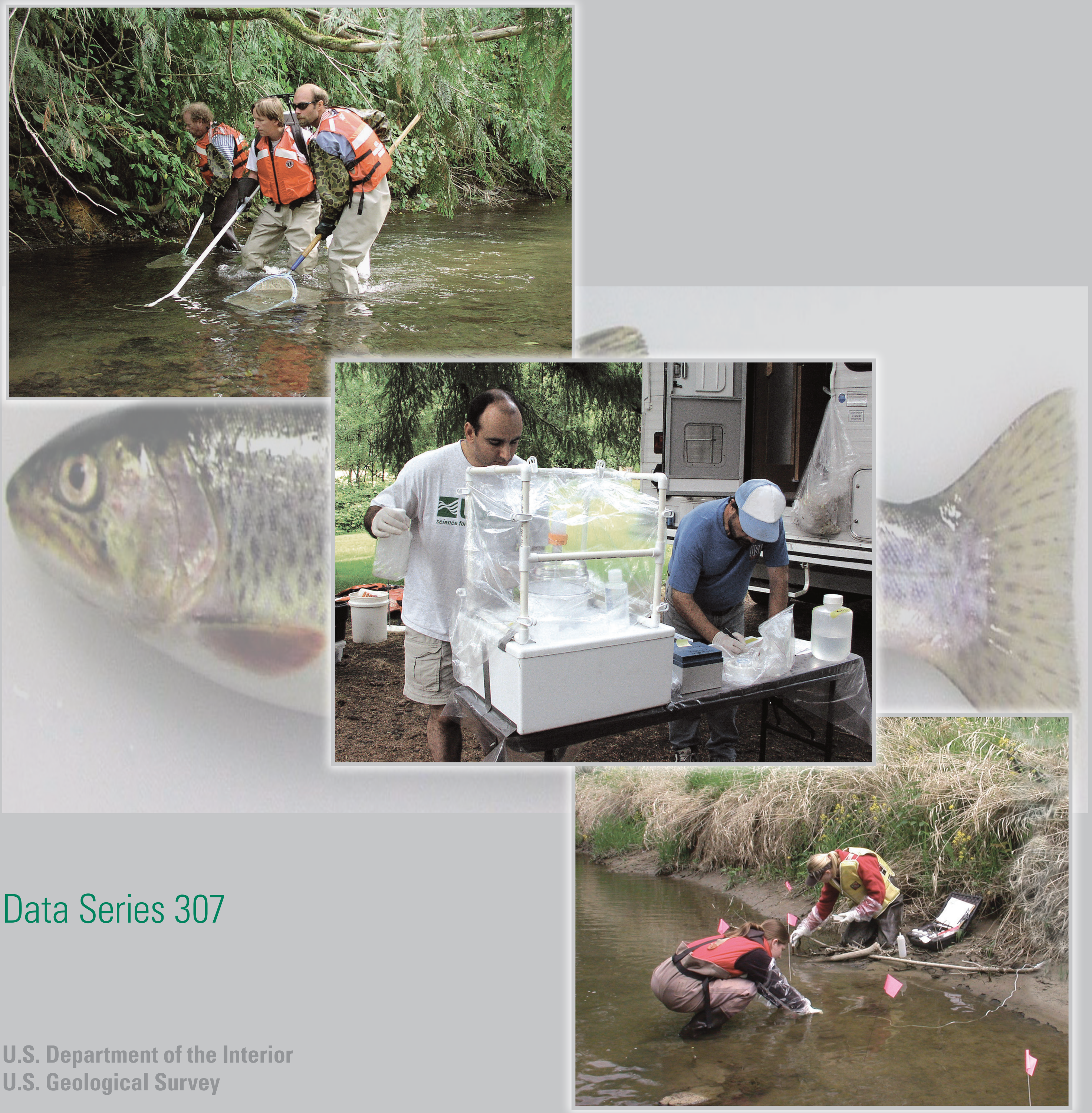
Data on Mercury in Water, Bed Sediment, and Fish from Streams Across the United States, 1998–2005



Data Series 307

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Cover photographs:
Top left, fish collection by electrofishing
Middle, stream-water processing
Bottom right, streambed sediment sampling
Background, cutthroat trout (*Oncorhynchus clarkii*)

All photographs by Dennis A. Wentz, U.S. Geological Survey

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U.S. Department of the Interior
KEN SALAZAR, Secretary

U.S. Geological Survey
Suzette M. Kimball, Acting Director

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FOREWORD

The U.S. Geological Survey (USGS) is committed to providing the Nation with reliable scientific information that helps to enhance and protect the overall quality of life and that facilitates effective management of water, biological, energy, and mineral resources (*http://www.usgs.gov/*). Information on the Nation's water resources is critical to ensuring long-term availability of water that is safe for drinking and recreation and is suitable for industry, irrigation, and fish and wildlife. Population growth and increasing demands for water make the availability of that water, measured in terms of quantity and quality, even more essential to the long-term sustainability of our communities and ecosystems.

The USGS implemented the National Water-Quality Assessment (NAWQA) Program in 1991 to support national, regional, State, and local information needs and decisions related to water-quality management and policy (*http://water.usgs.gov/nawqa*). The NAWQA Program is designed to answer: What is the quality of our Nation's streams and ground water? How are conditions changing over time? How do natural features and human activities affect the quality of streams and ground water, and where are those effects most pronounced? By combining information on water chemistry, physical characteristics, stream habitat, and aquatic life, the NAWQA Program aims to provide science-based insights for current and emerging water issues and priorities. From 1991 to 2001, the NAWQA Program completed interdisciplinary assessments and established a baseline understanding of water-quality conditions in 51 of the Nation's river basins and aquifers, referred to as Study Units (*http://water.usgs.gov/nawqa/studyu.html*).

National and regional assessments are ongoing in the second decade (2001–2012) of the NAWQA Program as 42 of the 51 Study Units are selectively reassessed. These assessments extend the findings in the Study Units by determining water-quality status and trends at sites that have been consistently monitored for more than a decade and filling critical gaps in characterizing the quality of surface water and ground water. For example, increased emphasis has been placed on assessing the quality of source water and finished water associated with many of the Nation's largest community water systems. During the second decade, NAWQA is addressing five national priority topics that build an understanding of how natural features and human activities affect water quality, and establish links between sources of contaminants, the transport of those contaminants through the hydrologic system, and the potential effects of contaminants on humans and aquatic ecosystems. Included are studies on the fate of agricultural chemicals, effects of urbanization on stream ecosystems, bioaccumulation of mercury in stream ecosystems, effects of nutrient enrichment on aquatic ecosystems, and transport of contaminants to public-supply wells. In addition, national syntheses of information on pesticides, volatile organic compounds (VOCs), nutrients, trace elements, and aquatic ecology are continuing.

The USGS aims to disseminate credible, timely, and relevant science information to address practical and effective water-resource management and strategies that protect and restore water quality. We hope this NAWQA publication will provide you with insights and information to meet your needs and will foster increased citizen awareness and involvement in the protection and restoration of our Nation's waters.

The USGS recognizes that a national assessment by a single program cannot address all water-resource issues of interest. External coordination at all levels is critical for cost-effective management, regulation, and conservation of our Nation's water resources. The NAWQA Program, therefore, depends on advice and information from other agencies—Federal, State, regional, interstate, Tribal, and local—as well as nongovernmental organizations, industry, academia, and other stakeholder groups. Your assistance and suggestions are greatly appreciated.

Matthew C. Larsen
Associate Director for Water

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Conversion Factors

Multiply	By	To obtain
Length		
nanometer (nm)	3.937 10^{-8}	inch (in.)
micrometer (µm)	3.937 10^{-5}	inch (in.)
millimeter (mm)	0.03937	inch (in.)
centimeter (cm)	0.3937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
foot (ft)	0.3048	meter (m)
Area		
square kilometer (km^2)	0.3861	square mile (mi^2)
Volume		
liter (L)	33.82	ounce, fluid (fl. oz)
milliliter (mL)	0.03382	ounce, fluid (fl. oz)
Flow rate		
cubic foot per second (ft^3/s)	0.02832	cubic meter per second (m^3/s)
Mass		
gram (g)	0.03527	ounce, avoirdupois (oz)
milligram (mg)	3.527 10^{-5}	ounce, avoirdupois (oz)
microgram (µg)	3.527 10^{-8}	ounce, avoirdupois (oz)
nanogram (ng)	3.527 10^{-11}	ounce, avoirdupois (oz)
Yield		
kilogram per hectare (kg/ha)	0.893	pound per acre (lb/acre)

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

$$°F = (1.8 \times °C) + 32$$

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).

Altitude, as used in this report, refers to distance above the vertical datum.

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 either in milligrams per liter (mg/L, or part per million), or nanograms per liter (ng/L, or part per trillion). Concentrations of chemical constituents in solid media (sediment and fish) are given either in micrograms per gram (µg/g, or part per million) or nanograms per gram (ng/g, or part per billion).

Abbreviations and Acronyms

AVS	acid-volatile sulfide
BrCl	bromine monochloride
CERC	Columbia Environmental Research Center
CVAFS	cold vapor atomic fluorescence spectroscopy
DOC	dissolved organic carbon
DOLT	dogfish (*Squalus acanthias*) liver
DORM	dogfish (*Squalus acanthias*) muscle
FMeHg	methylmercury in filtered water
FTHg	total mercury in filtered water
GIRAS	Geographic Information Retrieval and Analysis System
GIS	Geographic Information System
>	greater than
HCl	hydrochloric acid
Hg	mercury
ITIS	Integrated Taxonomic Information System
<	less than
LOI	loss on ignition
LULC	Land Use and Land Cover
MAS/MILS	Mineral Availability System/Mineral Industry Location System
MDL	method detection limit
MeHg	methylmercury
MRDS	Mineral Resources Data System
N	normal (moles of equivalents per liter)
NADP	National Atmospheric Deposition Program
NAWQA	National Water-Quality Assessment
NFM	National Field Manual
NIST	National Institute of Standards and Technology
NLCD	National Land Cover Data
NRCC	National Research Council Canada
NRP OCTL	National Research Program Organic Carbon Transformations Laboratory
NWIS	National Water Information System
NWQL	National Water Quality Laboratory
PETG	polyethylene terephthalate copolyester, glycol-modified
PMeHg	methylmercury in particulate matter
PRISM	Parameter-elevation Regressions on Independent Slopes Model
PTHg	total mercury in particulate matter
QC	quality control
RSD	relative standard deviation
SD	standard deviation
SGL	Sulfur Geochemistry Laboratory
SMeHg	methylmercury in bed sediment
$SnCl_2$	stannous chloride
STATSGO	State Soil Geographic
STHg	total mercury in bed sediment
SUVA	specific ultraviolet absorbance
TEAM	Trace Elements Analysis Model
TERL	Trace Element Research Laboratory (at Texas A&M University, College Station, Texas)
THg	total mercury
$\mu g/m^2$	microgram per square meter
UMeHg	methylmercury in unfiltered water
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
UTHg	total mercury in unfiltered water
WMRL	Wisconsin Mercury Research Laboratory

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Abstract

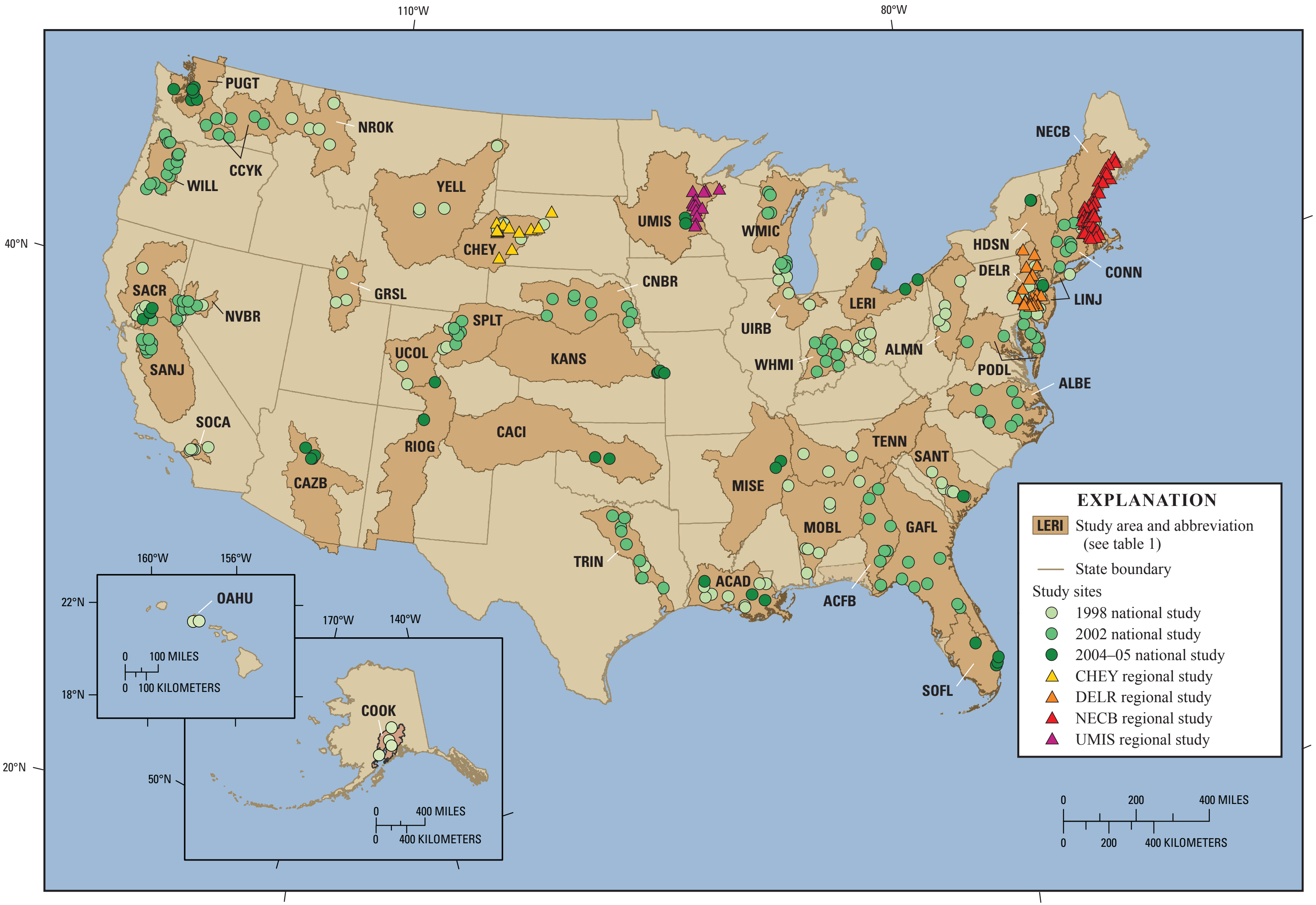
The U.S. Geological Survey (USGS) National Water-Quality Assessment (NAWQA) and Toxic Substances Hydrology Programs conducted the National Mercury Pilot Study in 1998 to examine relations of mercury (Hg) in water, bed sediment and fish in streams across the United States, including Alaska and Hawaii. Water and bed-sediment samples were analyzed for total Hg (THg), methylmercury (MeHg), and other constituents; fish were analyzed for THg. Similar sampling was conducted at additional streams across the country in 2002 and 2004–05. This report summarizes sample collection and processing protocols, analytical methods, environmental data, and quality-assurance data for stream water, bed sediment, and fish for these national studies. To extend the geographic coverage of the data, this report also includes four regional USGS Hg studies conducted during 1998–2001 and 2004. The environmental data for these national and regional Hg studies are provided in an electronic format.

Introduction

In 1998, the U.S. Geological Survey (USGS) National Water-Quality Assessment (NAWQA) and Toxic Substances Hydrology Programs conducted the National Mercury Pilot Study with the goal of examining relations of mercury (Hg) in water, bed sediment, and fish in streams across the Nation, including Alaska and Hawaii (Krabbenhoft and others, 1999; Brumbaugh and others, 2001). At each stream site, investigators conducted a one-time sampling of water, bed sediment, and fish for the analysis of total Hg (THg), methylmercury (MeHg), and other constituents. This Pilot study was the first known study in which nationwide multimedia sampling occurred jointly with low-level Hg speciation analysis (Krabbenhoft and others, 1999). The USGS conducted similar national and regional studies at additional streams across the Nation during 1998–2001, 2002, and 2004–05 (fig. 1, table 1). For the remainder of this report, results from the 1998 National Mercury Pilot Study are described together with results from the national and regional studies conducted during 1998–2005.

This report describes sample collection and processing procedures, analytical methods, environmental data, and quality-assurance data for water, bed sediment, and fish samples collected from 266 stream sites in 42 NAWQA study areas during national studies conducted in 1998, 2002, and 2004–05, and for samples collected from 101 stream sites during regional USGS studies in four study areas conducted in 1998–2001 and 2004. Water-quality, bed-sediment, and fish data, along with quality-assurance data and ancillary site data, are available for download on the Appendix Data page at *http://pubs.usgs.gov/ds/307/*. Analytical data for water include filtered THg (FTHg), filtered MeHg (FMeHg), particulate THg (PTHg), particulate MeHg (PMeHg), dissolved organic carbon (DOC) concentration, specific fractions and specific ultraviolet absorbance (SUVA) of DOC, sulfate and suspended-sediment concentrations, and suspended-sediment grain size. Bed-sediment data include THg, MeHg, loss on ignition (LOI, a measure of organic carbon content of sediment), acid-volatile sulfide (AVS), and grain size. Fish data include THg, type of cut (whole fish, skin-on fillet, skin-off fillet), weight and length measurements, and fish age. Data on streamflow, field measurements (water temperature, pH, specific conductance, and dissolved oxygen concentration), and ancillary site information are provided.

In all, 367 streams were sampled for national and regional studies combined. Fish Hg concentrations (715 samples) are available for 291 streams; concentrations of Hg in water and bed sediment are available for 342 streams. All three media (water, bed sediment, and fish) were sampled at 267 streams. Scudder and others (2009) describe the occurrence and spatial distribution of the Hg data in relation to regional and national gradients of Hg source strength and land-use/land-cover types; they also discuss associations among the Hg data and other measures of stream-water and bed-sediment chemistry.



Base composited from National Atlas of the United States boundaries of the United States, 1:2,000,000, 2005; Hitt and Nakagaki National Water-Quality Assessment (NAWQA) study-unit investigations in the conterminous United States 1991-2001, 1:2,000,000, 2006; Hitt and Nakagaki National Water-Quality Assessment (NAWQA) study-unit investigations in the conterminous United States 2001-2012, 1:2,000,000, 2006. Continental United States in Albers Conical Equal Area Projection, referenced to North American Datum of 1983. Alaska in Alaska Albers, referenced to North American Datum of 1983. Hawaii in Hawaii State Plane zone 1 FIPS zone 5101, referenced to North American Datum of 1983.

Figure 1. Streams sampled for mercury, 1998–2005. (Regional studies are CHEY, Cheyenne–Belle Fourche River Basins, 1998–99; DELR, Delaware River Basin, 1999–2001; NECB, New England Coastal Basins, 1999–2000; and UMIS, Upper Mississippi River Basin, 2004).

Table 1. U.S. Geological Survey National Water-Quality Assessment Program study area abbreviations, study area names, and years sampled, 1998–2005.

[--, not applicable]

Study area	Study area name	Year of national mercury study	Year of regional mercury study
ACAD	Acadian-Pontchartrain Drainages	1998, 2004–05	--
ACFB	Apalachicola-Chattahoochee-Flint River Basin	2002	--
ALBE	Albemarle-Pamlico Drainage Basin	2002	--
ALMN	Allegheny and Monongahela River Basins	1998	--
CACI	Canadian-Cimarron River Basins	2004–05	--
CAZB	Central Arizona Basins	2004–05	--
CCYK	Central Columbia Plateau–Yakima River Basin	2002	--
CHEY	Cheyenne–Belle Fourche River Basins	1998	1998–99
CNBR	Central Nebraska Basins	2002	--
CONN	Connecticut, Housatonic, and Thames River Basins	2002	--
COOK	Cook Inlet Basin	1998	--
DELR	Delaware River Basin	1998	1999–2001
GAFL	Georgia-Florida Coastal Plain	2002	--
GRSL	Great Salt Lake Basins	1998	--
HDSN	Hudson River Basin	2004–05	--
KANS	Kansas River Basin	2004–05	--
LERI	Lake Erie–Lake Saint Clair Drainages	2004–05	--
LINJ	Long Island–New Jersey Coastal Drainages	1998, 2004–05	--
MISE	Mississippi Embayment	2004–05	--
MOBL	Mobile River Basin	1998	--
NECB	New England Coastal Basins	1998	1999–2000
NROK	Northern Rockies Intermontane Basins	1998	--
NVBR	Nevada Basin and Range	1998, 2002	--
OAHU	Island of Oahu	1998	--
PODL	Potomac River Basin and Delmarva Peninsula	2002	--
PUGT	Puget Sound Basin	2004–05	--
RIOG	Rio Grande Valley	2004–05	--
SACR	Sacramento River Basin	1998, 2004–05	--
SANJ	San Joaquin–Tulare Basins	2002	--
SANT	Santee River Basin and Coastal Drainages	1998, 2004–05	--
SOCA	Southern California Coastal Drainages	1998	--
SOFL	Southern Florida	2004–05	--
SPLT	South Platte River Basin	2002	--
TENN	Tennessee River Basin	1998	--
TRIN	Trinity River Basin	1998, 2002	--
UCOL	Upper Colorado River Basin	1998	--
UIRB	Upper Illinois River Basin	1998	--
UMIS	Upper Mississippi River Basin	2004–05	2004
WHMI	White, Great and Little Miami River Basins	1998, 2002	--
WILL	Willamette Basin	2002	--
WMIC	Western Lake Michigan Drainages	2002	--
YELL	Yellowstone River Basin	1998	--

Data Collection

Site Selection

The streams included in the 1998 National Mercury Pilot Study represent a diversity of environmental settings across the Nation. Sites were chosen to represent a range of values for a number of environmental variables, including wetland density and other land-use/land-cover characteristics, pH, sulfate, total organic carbon and DOC, and Hg loading (source type, strength, and extent). Streams for the 2002 and 2004–05 studies were selected to include the same range in environmental variables as the 1998 National Mercury Pilot Study, as well as to extend geographic coverage, increase representation of key land-use and land-cover categories, and increase the range of DOC values (both concentration and SUVA). Additional factors considered in site selection were the availability of historical water-quality and ecological data and the presence of top predator fish. Because largemouth bass (*Micropterus salmoides*) is a widely distributed top predator fish, this species and other black bass species (*Micropterus* spp.) were emphasized as a first preference for collection.

Data for Hg in water, bed sediment, and fish from 101 streams sampled during USGS regional Hg studies conducted from 1998 through 2001 and during 2004 in four NAWQA study areas are included in this report to extend the geographic coverage of data. The inclusion of these studies was based on availability of Hg data for water, bed sediment, and fish, with supporting field properties (water temperature, pH, specific conductance, dissolved oxygen concentration) and streamflow. The studies are defined as CHEY (Cheyenne–Belle Fourche River Basins) regional study (S.K. Sando, USGS, unpub. data, 2005), DELR (Delaware River Basin) regional study (Brightbill and others, 2003), NECB (New England Coastal Basins) regional study (Chalmers and Krabbenhoft, 2001), and UMIS (Upper Mississippi River Basin) regional study (Christensen and others, 2006). For the first three regional studies, many streams were sampled more than once. Only one sample per site is included in the data tables to maintain consistency with the national studies and to avoid overweighing analysis and interpretation in favor of sites with more than one sample. For a site with multiple samples, the sample included in the data tables had the greatest number of constituents measured, was sampled during low flow in August, and(or) was sampled during the same week in August as most other samples for the regional study. Additional data for the DELR and NECB regional studies are available in Brightbill and others (2003) at *http://nh.water.usgs.gov/projects/nawqa/sw_merc.htm*, and on the USGS National Water Information System (NWIS) Web at *http://waterdata.usgs.gov/usa/nwis/qw*.

Sample Collection and Processing

Water samples compiled for this database were collected and processed for analysis of FTHg, FMeHg, PTHg, PMeHg, DOC, sulfate, and suspended sediment (concentration and grain size). Bed-sediment samples were collected and processed for analysis of THg and MeHg, LOI, AVS, and grain size. Fish samples were collected and processed for analysis of THg only; otoliths, scales, or spines were collected from fish for age determination. The types of samples collected during each study are summarized in tables 2–4. Field properties (water temperature, pH, specific conductance, dissolved oxygen concentration) were measured when water and bed-sediment samples were collected (Wilde, variously dated). At most sites, streamflow also was measured (Rantz and others, 1982).

Stream-Water Sample Collection

USGS clean-sampling procedures (sometimes called trace element part-per-billion or ppb protocols) and ultra trace-level clean techniques (for Hg) were used to collect water samples. Detailed descriptions of these procedures and techniques are provided in the USGS National Field Manual (NFM) (U.S. Geological Survey, variously dated [chapters 1–4]); Fitzgerald and Watras (1989); U.S. Environmental Protection Agency (1996a); Olson and DeWild (1999); and Lewis and Brigham (2004). USGS scientists were trained in clean sampling techniques prior to sample collection and processing. Because low-level Hg samples are susceptible to contamination from many sources, specific measures were required for collecting Hg samples to prevent contamination (Lewis and Brigham, 2004). Field personnel wore shoulder-length polyethylene gloves under powder-free, wrist-length nitrile or latex gloves. Particle-shedding clothing (for example, fleece jackets, wool sweaters, or soiled field clothing) was avoided; outerwear, if needed, was clean, nylon-shell (or comparable material) clothing. Field personnel were instructed to open sample containers while holding them upwind from themselves or with the container inside a sample-processing chamber (Lane and others, 2003).

Stream-water samples were collected during low-flow conditions between late spring and early fall, depending on the general geographic location. For streams known to be well mixed, dip samples for Hg, DOC, sulfate, and suspended-sediment analyses were obtained from the approximate centroid of flow. For streams not known to be well mixed, an isokinetic, depth-integrated sample or a multiple-vertical composite sample was collected (Wilde and others, 1999; Lewis and Brigham, 2004). At wadeable sites, the in-channel collection site was approached from downstream, and sample containers were extended upstream into the current to avoid disturbance of sample water by field personnel. At nonwadeable sites, samples were collected from the bow of a boat moving upstream at low speed; sample bottles and

Table 2. Summary of samples collected and methods used for the determination of field properties and streamflow, and laboratory analysis of mercury, organic carbon, major ions, and suspended sediment in stream water, 1998–2005.

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; --, not applicable; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; 1998, National Mercury Pilot Study conducted in 1998; 2004–05, national study of mercury conducted in 2004–05; NWQL, National Water Quality Laboratory; 2002, national study of mercury conducted in 2002. See text for methods used in sample collection and processing]

Constituent	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Laboratory	Reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004		
Field properties and streamflow										
Water temperature	Temp	yes	yes	yes	yes	yes	yes	yes	--	Wilde, F.D., ed., variously dated
pH	pH	yes	yes	yes	yes	yes	yes	yes	--	Wilde, F.D., ed., variously dated
Specific conductance	SC	yes	yes	yes	yes	yes	yes	yes	--	Wilde, F.D., ed., variously dated
Dissolved oxygen	DO	yes	yes	yes	yes	yes	yes	yes	--	Rantz, S.E., and others, 1982
Streamflow	Q_inst	yes	yes	yes	yes	yes	yes	yes	--	Wilde, F.D., ed., variously dated
Analytes										
Mercury										
Unfiltered total mercury	UTHg	yes	no	no	yes	yes	yes	no	USGS WMRL	U.S. Environmental Protection Agency, 2002 Olson, M.L., Cleckner, L.B., Hurley, J.P., Krabbenhoft, D.P., and Heelan, T.W., 1997 Olson, M.L., and DeWild, J.F., 1999
Filtered total mercury	FTHg	yes (CHEY only)	yes	yes	yes	no	no	yes	USGS WMRL	U.S. Environmental Protection Agency, 2002 Olson, M.L., Cleckner, L.B., Hurley, J.P., Krabbenhoft, D.P., and Heelan, T.W., 1997 Olson, M.L., and DeWild, J.F., 1999

Table 2. Summary of samples collected and methods used for the determination of field properties and streamflow, and laboratory analysis of mercury, organic carbon, major ions, and suspended sediment in stream water, 1998–2005.—Continued

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; --, not applicable; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; 1998, National Mercury Pilot Study conducted in 1998; 2004–05, national study of mercury conducted in 2004–05; NWQL, National Water Quality Laboratory; 2002, national study of mercury conducted in 2002. See text for methods used in sample collection and processing]

Constituent	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Laboratory	Reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004		
Mercury										
Unfiltered methylmercury	UMeHg	yes	no	no	yes	yes	yes	no	USGS WMRL	Bloom, N., 1989 Horvat, Milena, Liang, Lian, and Bloom, N.S., 1993 Olson, M.L., and DeWild, J.F., 1999 DeWild, J.F., Olson, M.L., and Olund, S.D., 2002
Filtered methylmercury	FMeHg	yes (CHEY only)	yes	yes	yes	no	no	yes	USGS WMRL	Bloom, N., 1989 Horvat, Milena, Liang, Lian, and Bloom, N.S., 1993 Olson, M.L., and DeWild, J.F., 1999 DeWild, J.F., Olson, M.L., and Olund, S.D., 2002
Particulate total mercury	PTHg	no	yes	yes	no	no	no	yes	USGS WMRL	U.S. Environmental Protection Agency, 2002 Olund, S.D., DeWild, J.F., Olson, M.L., and Tate, M.T., 2004
Particulate methylmercury	PMeHg	no	yes	yes	no	no	no	yes	USGS WMRL	DeWild, J.F., Olson, M.L., and Olund, S.D., 2002 DeWild, J.F., Olund, S.D., Olson, M.L., and Tate, M.T., 2004

Table 2. Summary of samples collected and methods used for the determination of field properties and streamflow, and laboratory analysis of mercury, organic carbon, major ions, and suspended sediment in stream water, 1998–2005.—Continued

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; --, not applicable; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; 1998, National Mercury Pilot Study conducted in 1998; 2004–05, national study of mercury conducted in 2004–05; NWQL, National Water Quality Laboratory; 2002, national study of mercury conducted in 2002. See text for methods used in sample collection and processing]

Constituent	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Laboratory	Reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004		
Organic carbon										
Dissolved organic carbon	DOC	yes	yes	yes	yes	yes	yes	yes	USGS NRP OCTL (1998; 2004–05, NECB-reg, UMIS-reg); USGS WMRL (CHEY-reg)	Aiken, G.R., 1992
									USGS NWQL (2002, DELR-reg)	Brenton, R.W. and Arnett, T.L., 1993
Hydrophobic acid fraction	HPOA_FRAC_PCT	yes	no	yes	no	no	no	yes	USGS NRP OCTL	Aiken, G.R., McKnight, D.M., Thorn, K.A., and Thurman, E.M., 1992
Hydrophilic acid fraction, low molecular weight	HPI_FRAC	no	no	yes	no	no	no	yes	USGS NRP OCTL	Aiken, G.R., McKnight, D.M., Thorn, K.A., and Thurman, E.M., 1992
Transphilic organic acid fraction	TPIA_FRAC	no	no	yes	no	no	no	yes	USGS NRP OCTL	Aiken, G.R., McKnight, D.M., Thorn, K.A., and Thurman, E.M., 1992
Hydrophilic acid fraction	HPIA_FRAC	yes	no	no	no	no	no	no	USGS NRP OCTL	Aiken, G.R., McKnight, D.M., Thorn, K.A., and Thurman, E.M., 1992
Specific ultraviolet absorbance	SUVA	yes	no	yes	no	no	no	yes	USGS NRP OCTL	Weishaar, J.L., Aiken, G.R., Bergamaschi, B.A., Fram, M.S., Fujii, Roger, and Mopper, Kenneth, 2003

Table 2. Summary of samples collected and methods used for the determination of field properties and streamflow, and laboratory analysis of mercury, organic carbon, major ions, and suspended sediment in stream water, 1998–2005.—Continued

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; --, not applicable; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; 1998, National Mercury Pilot Study conducted in 1998; 2004–05, national study of mercury conducted in 2004–05; NWQL, National Water Quality Laboratory; 2002, national study of mercury conducted in 2002. See text for methods used in sample collection and processing]

Constituent	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Laboratory	Reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004		
Major ions										
Sulfate	Sulfate	yes	yes	yes	yes	yes	yes	yes	USGS WMRL; USGS NWQL; USGS Atlanta, Ga. (NECB-reg, 1999 samples)	Fishman, M.J., and Friedman, L.C., 1989
Suspended sediment										
Concentration	SS	yes	yes	yes	yes	yes	no	no	USGS Sediment Laboratory (Vancouver, Wash.; Iowa City, Iowa)	Guy, H.P., 1969
Grain size (percent fines)	SS_Pct_063	yes	yes	yes	yes	no	no	no	USGS Sediment Laboratory (Vancouver, Wash.; Iowa City, Iowa)	Guy, H.P., 1969

Table 3. Summary of samples collected and methods used for sample collection and processing and laboratory analysis of mercury, organic matter, sulfur, and size characterization in bed sediment, 1998–2005.

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; 1998, National Mercury Pilot Study conducted in 1998; SGL, Sulfur Geochemistry Laboratory; 2002, national study of mercury conducted in 2002; 2004–05, national study of mercury conducted in 2004–05]

Analyte	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Sample collection and processing reference	Laboratory	Laboratory analysis reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004			
Mercury											
Total mercury	STHg	yes	yes	yes	yes	yes	yes	yes	Krabbenhoft, D.P., Wiener, J.G., Brumbaugh, W.G., Olson, M.L., DeWild, J.F., and Sabin, T.J., 1999 Lutz, M.A., Brigham, M.E., and Marvin-DiPasquale, Mark, 2008	USGS WMRL	Olund, S.D., DeWild, J.F., Olson, M.L., and Tate, M.T., 2004
Methylmercury	SMeHg	yes	yes	yes	yes	yes	yes	yes	Krabbenhoft, D.P., Wiener, J.G., Brumbaugh, W.G., Olson, M.L., DeWild, J.F., and Sabin, T.J., 1999 Lutz, M.A., Brigham, M.E., and Marvin-DiPasquale, Mark, 2008	USGS WMRL	DeWild, J.F., Olund, S.D., Olson, M.L., and Tate, M.T., 2004

Table 3. Summary of samples collected and methods used for sample collection and processing and laboratory analysis of mercury, organic matter, sulfur, and size characterization in bed sediment, 1998–2005.—Continued

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; 1998, National Mercury Pilot Study conducted in 1998; SGL, Sulfur Geochemistry Laboratory, 2002; national study of mercury conducted in 2002; 2004–05, national study of mercury conducted in 2004–05]

Analyte	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Sample collection and processing reference	Laboratory	Laboratory analysis reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004			
Organic matter											
Loss on ignition	LOI	yes	yes	yes	no	yes	yes	yes	Krabbenhoft, D.P., Wiener, J.G., Brumbaugh, W.G., Olson, M.L., DeWild, J.F., and Sabin, T.J., 1999 Lutz, M.A., Brigham, M.E., and Marvin-DiPasquale, Mark, 2008	USGS WMRL	Heiri, Oliver, Lotter, A.F., and Lemcke, Gerry, 2001
Sulfur											
Acid-volatile sulfide	AVS_DryWt, Pct_AVS_WetWt	yes	yes	yes	no	no	yes	yes	Krabbenhoft, D.P., Wiener, J.G., Brumbaugh, W.G., Olson, M.L., DeWild, J.F., and Sabin, T.J., 1999 Lutz, M.A., Brigham, M.E., and Marvin-DiPasquale, Mark, 2008	USGS WRML (1998, NECB-reg)	Allen, H.E., Fu, G., Boothman, W., DiToro, D.M., and Mahony, J.D., 1991

Table 3. Summary of samples collected and methods used for sample collection and processing and laboratory analysis of mercury, organic matter, sulfur, and size characterization in bed sediment, 1998–2005.—Continued

[See Appendix 1 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; 1998, National Mercury Pilot Study conducted in 1998; SGL, Sulfur Geochemistry Laboratory, 2002; national study of mercury conducted in 2002; 2004–05, national study of mercury conducted in 2004–05]

Analyte	Column name from Appendix 1. Water and sediment sample data	National mercury studies			CHEY-reg	DELR-reg	NECB-reg	UMIS-reg	Sample collection and processing reference	Laboratory	Laboratory analysis reference
		1998	2002	2004–05	1998–99	1999–2000	1999–2000	2004			
Sulfur											
									Krabbenhoft, D.P., Wiener, J.G., Brumbaugh, W.G., Olson, M.L., DeWild, J.F., and Sabin, T.J., 1999	USGS SGL (2002 and 2004–05, UMIS-reg)	Allen, H.E., Fu, G., Boothman, W., DiToro, D.M., and Mahony, J.D., 1991
									Lutz, M.A., Brigham, M.E., and Marvin-DiPasquale, Mark, 2008		Bates, A.L., Spiker, E.C., Orem, W.H., Burnett, W.C., 1993
											Tuttle, M.L., Goldhaber, M.B., and Williamson, D.L., 1986
Size characterization											
Grain size (percent fines)	Bed_Pct_063	yes	yes	yes	no	yes	no	no	Krabbenhoft, D.P., Wiener, J.G., Brumbaugh, W.G., Olson, M.L., DeWild, J.F., and Sabin, T.J., 1999	USGS Sediment Laboratory (Vancouver, Wash.; Iowa City, Iowa)	Guy, H.P., 1969
									Lutz, M.A., Brigham, M.E., and Marvin-DiPasquale, Mark, 2008		

Table 4. Summary of samples collected and methods used for sample collection and processing and laboratory analysis of mercury in fish and fish age determination, 1998–2005.

[See Appendix 4 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; --, not applicable; USGS, U.S. Geological Survey; CERC, Columbia Environmental Research Center; NWQL, National Water Quality Laboratory; TERL, Trace Element Research Laboratory; WMRL, Wisconsin Mercury Research Laboratory]

Analyte	Column name from Appendix 4. Fish data	National mercury studies			CHEY-reg	DELR-reg		NECB-reg	UMIS-reg
		1998	2002	2004–05	1998–99	1999	2001	1999–2000	2004
Mercury in fish									
Total mercury in fish tissue	THg_Dry, THG_Wet	yes	yes	yes	No samples collected	yes	yes	yes	yes
Laboratory	--	USGS CERC	USGS NWQL	Texas A&M University TERL	--	USGS WMRL	USGS NWQL	USGS WMRL	River Studies Center, University of Wisconsin, La Crosse, Wis.
Reference	--	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	--	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008
		Brumbaugh, W.G., Krabbenhoft, D.P., Helsel, D.R., Wiener, J.G., and Echols, K.R., 2001	U.S. Environmental Protection Agency, 1996b	U.S. Environmental Protection Agency, 1998		Brumbaugh, W.G., Krabbenhoft, D.P., Helsel, D.R., Wiener, J.G., and Echols, K.R., 2001	U.S. Environmental Protection Agency, 1996b	Brumbaugh, W.G., Krabbenhoft, D.P., Helsel, D.R., Wiener, J.G., and Echols, K.R., 2001	Christensen, V.G., Wente, S.P., Sandheinrich, M.B., and Brigham, M.E., 2006
		Olson, M.L., and DeWild, J.F., 1999	U.S. Environmental Protection Agency, 1996c			Olson, M.L., and DeWild, J.F., 1999	U.S. Environmental Protection Agency, 1996c	Olson, M.L., and DeWild, J.F., 1999	U.S. Environmental Protection Agency, 2002

Table 4. Summary of samples collected and methods used for sample collection and processing and laboratory analysis of mercury in fish and fish age determination, 1998–2005.

[See Appendix 4 for definition of column names and units of measurement. The 1998 National Mercury Pilot Study was conducted with the U.S. Geological Survey Toxic Substances Hydrology Program and National Water-Quality Assessment Program. CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; DELR-reg, regional study of mercury in the Delaware River Basin; NECB-reg, regional study of mercury in the New England Coastal Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; --, not applicable; USGS, U.S. Geological Survey; CERC, Columbia Environmental Research Center; NWQL, National Water Quality Laboratory; TERL, Trace Element Research Laboratory; WMRL, Wisconsin Mercury Research Laboratory]

Analyte	Column name from Appendix 4. Fish data	National mercury studies			CHEY-reg	DELR-reg		NECB-reg	UMIS-reg
		1998	2002	2004–05	1998–99	1999	2001	1999–2000	2004
Mercury in fish									
Otoliths, scales, spines	Age, Ave_Age	yes	yes	yes	No samples collected	No samples collected	No samples collected	No samples collected	No samples collected
Laboratory	--	USGS CERC	USGS South Carolina Cooperative Wildlife Research Unit Laboratory	USGS South Carolina Cooperative Wildlife Research Unit Laboratory	--	--	--	--	--
Reference	--	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	Scudder, B.C., Chasar, L.S., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., 2008	--	--	--	--	--
		Brumbaugh, W.G., Krabbenhoft, D.P., Helsel, D.R., Wiener, J.G., and Echols, K.R., 2001	Brumbaugh, W.G., Krabbenhoft, D.P., Helsel, D.R., Wiener, J.G., and Echols, K.R., 2001	Brumbaugh, W.G., Krabbenhoft, D.P., Helsel, D.R., Wiener, J.G., and Echols, K.R., 2001					
		Jearld, A., Jr., 1983	Jearld, A., Jr., 1983	Jearld, A., Jr., 1983					
		Porak, W., Coleman, W.S., and Crawford, S., 1988.	Porak, W., Coleman, W.S., and Crawford, S., 1988.	Porak, W., Coleman, W.S., and Crawford, S., 1988.					

containers were positioned in front of the boat and extended upstream into the current. Samples from nonwadeable sites were also collected from bridges using the reed-and-suspension method and dip or isokinetic, depth-integrated methods on the upstream side of the bridge (Wilde and others, 1999).

Dip samples were collected for THg and MeHg in fluorocarbon polymer (Teflon®) bottles supplied by the USGS Wisconsin Mercury Research Laboratory (WMRL) in Middleton, Wis., or in new polyethylene terephthalate copolyester, glycol-modified (PETG) containers; the latter were typically used in 2002 and beyond. All fluorocarbon polymer sample bottles were rigorously cleaned by the WMRL (Olson and DeWild, 1999), partially filled with 1 percent hydrochloric acid (HCl, OmniTrace™) and shipped in double zip-seal plastic bags. All PETG bottles (Nalgene®) were new (cap seals intact) and were stored in double zip-seal plastic bags until use. Dip samples for DOC and sulfate analysis were collected using a Teflon® bottle or from the PETG bottles that were used for the Hg sample. Samples for suspended-sediment concentration and grain-size analysis were collected in widemouth 1-liter plastic bottles or other appropriate suspended-sediment sampling bottle, using methods comparable to those used for the other water-quality samples (as noted above, either dip in approximate centroid of flow, or with an isokinetic sampler).

Stream-Water Sample Processing

Stream-water samples were processed for the analysis of unfiltered THg (UTHg) and unfiltered MeHg (UMeHg) or for FTHg, FMeHg, PTHg, and PMeHg. The type of analysis for each study is shown in table 2. In this report, “filtered” refers to constituents that pass through a filter, and includes dissolved and filter-passing colloidally bound constituents. “Particulate” refers to constituents retained on the filter and includes sediment and organic detritus.

Stream-water samples collected for the analysis of UTHg were preserved in the field by addition of 6 *N* HCl to a final concentration of approximately 1 percent by volume. Samples collected for UMeHg analysis were frozen immediately on dry ice until analysis. Samples collected for analysis of FTHg and FMeHg were vacuum filtered through combusted, 47 millimeter (mm), 0.7-micrometer (µm) quartz-fiber filters in the field in a sample processing chamber (Lane and others, 2003); preserved with 6 *N* HCl to a concentration of approximately 1 percent by volume; double bagged in zip-seal bags; and stored in a closed dark cooler until analysis (Olson and DeWild, 1999; Lewis and Brigham, 2004). The volume of water filtered through a filter was recorded for subsequent conversion of particulate amount to mass per volume. The quartz-fiber filters were transferred to Teflon® Petri dishes, double-bagged in new plastic zip-seal bags, and immediately placed on dry ice; filters were maintained frozen until analysis of PTHg and PMeHg.

Samples collected for the analysis of DOC and sulfate were processed in the field as directed by the USGS NFM (Lane and others, 2003; Wilde and others, 2004). DOC samples were filtered through combusted, 0.7-µm glass fiber filters, chilled, and stored at 4° Celsius (C). Sulfate samples were filtered through 0.45-µm membrane or polysulfone capsule filters, and stored until analysis. Unfiltered and unpreserved suspended-sediment samples were stored in original collection bottles until analysis.

Bed-Sediment Sample Collection

Detailed descriptions of bed-sediment sampling methods for all USGS NAWQA Hg sampling efforts and regional Hg studies are provided in Krabbenhoft and others (1999) and Lutz and others (2008). Clean sampling methods were adapted from Shelton and Capel (1994). An acid-cleaned Teflon® or plastic scoop was used to collect the top (approximately) 2 centimeter (cm) of bed sediment from 5 to 10 depositional areas; these samples were composited in acid-cleaned Teflon®, plastic, or glass containers into one sample for each stream site.

Bed-Sediment Sample Processing

Bed-sediment composites were immediately homogenized by stirring with a Teflon spatula and subsampled in the field. Bed-sediment subsamples for analysis of total Hg (STHg) and MeHg (SMeHg) were placed in acid-cleaned polypropylene or Teflon® vials and stored frozen until analysis. AVS subsamples were placed in clean polypropylene jars, and samples for the 1998 and 2002 national studies and NECB regional study were preserved with 0.3 milliliter of zinc acetate. All AVS samples were stored frozen until analysis. Subsamples for the analysis of bed-sediment grain size were placed in polypropylene jars and stored until analysis. Detailed descriptions of the steps for processing bed-sediment samples are available in Krabbenhoft and others (1999) and Lutz and others (2008).

Fish Sampling and Processing

Fish sampling and sample processing methods are detailed in Scudder and others (2008). Field personnel collected up to eight largemouth bass or other black bass (*Micropterus* spp.), approximately 3 to 4 years old, whenever possible, at each sampling site. If bass were difficult to collect in the target age range or were not present in a study area, alternate predator fish (piscivorous or insectivorous) species were chosen from a priority list provided in Scudder and others (2008). Multiple sampling methods were used, including electrofishing, seining, and hook and line.

Fish samples collected during the 1998 National Mercury Pilot Study were rinsed in stream water, measured as individual samples for weight and length, placed whole in double zip-seal bags, and frozen until they were analyzed (Brumbaugh and others, 2001). For the 2002 and 2004–05 national studies and the NECB regional study, fish were weighed, measured and processed in the field to obtain axial muscle (skinless fillet) tissue, as detailed by Scudder and others (2008). Fillets were rinsed with deionized water, placed in double zip-seal bags, and frozen until analysis. For the DELR regional study, fish were weighed and measured in the field and processed either in the laboratory or field to obtain fillet samples. Fish collected during the UMIS regional study were stored on wet ice in plastic bags with sample water from the collection site until transfer to refrigerators. After length and weight measurements in a laboratory setting, fish were frozen until analysis (Christensen and others, 2006). Fish samples were not collected during the CHEY regional study.

Otoliths, scales, or spines were obtained from each fish collected during the national studies for age determination. Similar materials were not obtained from fish collected during the DELR, NECB, and UMIS regional studies.

Analytical Methods

Multiple analytical laboratories were used for the national and regional Hg studies; a summary of methods and laboratories is provided in tables 2–4.

Stream Water

All samples for unfiltered, filtered, and particulate THg and MeHg were analyzed by the WMRL. The analytical procedures for determining THg in unfiltered and filtered water are detailed in U.S. Environmental Protection Agency (USEPA) Method 1631 (U.S. Environmental Protection Agency, 2002), as adapted by the WMRL (Olson and others, 1997; Olson and DeWild, 1999). Briefly, THg samples were oxidized with bromine monochloride (BrCl) under acidic conditions, treated with stannous chloride ($SnCl_2$) to reduce ionic Hg to elemental Hg, and quantified with cold vapor atomic fluorescence spectroscopy (CVAFS). Samples were analyzed for MeHg by distillation, aqueous phase ethylation, purge and trap of volatile ethylmercury derivatives, gas-chromatographic separation, pyrolization, and CVAFS determination (Bloom, 1989, as modified by Horvat and others, 1993; Olson and DeWild, 1999; DeWild and others, 2002). Concentrations of PTHg and PMeHg were determined by analyzing the 0.7-μm quartz-fiber filters obtained from the stream-water filtration for FTHg and FMeHg. Filters for PTHg analysis were thawed and placed in Teflon bottles, and BrCl was added. Samples were oxidized, treated with $SnCl_2$, and analyzed according to USEPA Method 1631 using CVAFS (U.S. Environmental Protection Agency, 2002; Olund and others, 2004). Filters for PMeHg analysis were placed in Teflon bottles for distillation and reagents were added. Samples were distilled, and the distillates were analyzed following the procedures described for aqueous MeHg (DeWild and others, 2002; DeWild and others, 2004).

Concentrations of DOC for the 1998 and 2004–05 national studies and the NECB and UMIS regional studies were determined by the USGS National Research Program Organic Carbon Transformations Laboratory (NRP OCTL) in Boulder, Colo., using a persulfate wet oxidation method as described in Aiken (1992). DOC samples for the CHEY regional study were analyzed at the WMRL using the same wet oxidation method. Water samples for the 2002 national study and the DELR regional study were analyzed for DOC concentration at the USGS National Water Quality Laboratory (NWQL) in Denver, Colo., following a UV-promoted persulfate oxidation and infrared spectroscopy (Brenton and Arnett, 1993). For samples analyzed by the NRP OCTL, carbon fractions of DOC (hydrophobic organic acids, hydrophilic acids, and transphilic acids) were measured by a modified version of the XAD-8 and XAD-4 methods (Aiken and others, 1992). SUVA was calculated by measuring the UV absorbance at 254 nanometers and dividing by the DOC concentration (Weishaar and others, 2003).

Water samples were analyzed for sulfate by the WMRL (1998 National Mercury Pilot Study), the NWQL (2002 and 2004–05 NAWQA national studies; CHEY, DELR, NECB, and UMIS regional studies), and the USGS laboratory in Atlanta, Ga. (1999 NECB regional study). All laboratories used ion chromatography for analysis (Fishman and Friedman, 1989).

Suspended-sediment samples were analyzed by the USGS sediment laboratories in Vancouver, Wash., and Iowa City, Iowa, for concentration and grain size (percent fines) (Guy, 1969).

Bed Sediment

Bed-sediment samples were analyzed by the WMRL for STHg and SMeHg concentrations using similar analytical procedures as those described above for aqueous and particulate samples, with some modification (DeWild and others, 2004; Olund and others, 2004). The WMRL also determined LOI, a measure of organic carbon content of sediment, as described by Heiri and others (2001), and sediment dry weight (percent solids) by drying wet sediment at 105°C.

AVS was analyzed by the WMRL (1998 National Mercury Pilot Study, NECB regional study) and by the USGS Sulfur Geochemistry Laboratory (SGL) (2002 and 2004–05 national studies, UMIS regional study) in Reston, Va. The WMRL procedure required acidification of each sample with 6 *N* HCl, trapping released sulfide in an antioxidant buffer, and determination of sulfide by ion-specific electrode (Allen and others, 1991). At the SGL, AVS was separated using an HCl extraction and reprecipitation as silver sulfide. Percent by weight of AVS in the sediment sample was subsequently determined gravimetrically (Canfield and others, 1986; Tuttle and others, 1986; Allen and others, 1991; Bates and others, 1993).

Bed sediment was analyzed for grain size (percent fines) by the USGS sediment laboratories in Vancouver, Wash., and Iowa City, Iowa (Guy, 1969).

Fish

Fish samples collected during the 1998 National Mercury Pilot Study were processed and analyzed at the USGS Columbia Environmental Research Center (CERC) in Columbia, Mo. Fish less than approximately 50 grams (g) in weight were analyzed whole or whole minus head; fish greater than 50 g were filleted to obtain a sample of skin-off axial muscle (Brumbaugh and others, 2001). All whole fish and fish muscle tissue were freeze-dried to a constant weight, finely ground, and stored in a dessicator until analysis. For each site, samples from the same species and of similar size were composited and analyzed as one sample. Fish samples were analyzed for THg using an acid/microwave digestion, BrCl oxidation, and CVAFS (Olson and DeWild, 1999; Brumbaugh and others, 2001). Only THg was analyzed because approximately 95 percent of Hg in fish is MeHg (Huckabee and others, 1979; Bloom, 1992; Wiener and Spry, 1996).

Fillet samples for the 2002 national study were analyzed at the NWQL. Each sample was freeze-dried to constant weight and finely ground, and fish of a single species from each site were analyzed as one composite sample. THg was determined using acid/microwave digestion, BrCl oxidation, and CVAFS in accordance with USEPA method 3052 for digestion (U.S. Environmental Protection Agency, 1996b) and USEPA method 7474 (U.S. Environmental Protection Agency, 1996c) for Hg analysis.

Fillet samples collected during the 2004–05 national study were analyzed for THg at the Texas A&M University Trace Element Research Laboratory (TERL) in College Station, Tex. Each sample was freeze-dried to constant weight, finely ground, and analyzed individually (1–13 fish per site) using USEPA method 7473 (combustion-trapping-atomic absorption; U.S. Environmental Protection Agency, 1998).

Skin-off fillet samples collected during the DELR regional study in 1999 and during the NECB regional study were analyzed at the WRML using a process similar to that used by CERC to analyze the 1998 National Mercury Pilot Study fish samples. The skin-off fillets collected during the 2001 DELR regional study were analyzed by the NWQL according to methods described for the 2002 national fish samples.

Fish collected for the UMIS regional study were analyzed as composites samples of whole fish, skin-on fillets, or skin-off fillets by the River Studies Center, University of Wisconsin, La Crosse, Wis. (Christensen and others, 2006). Subsamples from composite samples were digested using a modification of USEPA Method 1631 (U.S. Environmental Protection Agency, 2002; Christensen and others, 2006). Digested subsamples were analyzed by flow-injection CVAFS.

For the national studies and the UMIS regional study, moisture content (percent water) was determined by weight loss upon freeze-drying and is reported as the weight percentage of the original wet sample. Moisture content was not reported for the DELR and NECB regional studies. Fish ages were estimated from sagittal otoliths, scales, or spines by the CERC (1998 National Mercury Pilot Study) or the USGS South Carolina Cooperative Fish and Wildlife Research Unit (2002 and 2004–05 national studies) (Jearld, 1983; Porak and others, 1988; Brumbaugh and others, 2001; Scudder and others, 2008).

The Integrated Taxonomic Identification System (ITIS) codes (*http://www.itis.gov/*) for all fish species sampled are included in Appendix 4.

Quality Assurance

The quality of stream-water, bed-sediment, and fish data was assured through a variety of quality-control (QC) samples collected in the field and laboratory. These included blank and replicate samples, spike recoveries, and certified and standard reference materials. Blank and replicate field QC samples were used to assess the bias and variability that may be introduced by sample collection, processing, and analysis. Laboratory QC samples were routinely analyzed to calibrate instruments, identify procedural problems, validate data, and provide defensible analytical data. Field QC samples will be discussed for water and bed sediment only; information on laboratory QC samples for water and bed sediment is available in references listed in tables 2–4. Field QC samples were not collected for fish because the nature of this medium does not allow collection of true field blanks or replicates. Laboratory QC samples to be discussed for fish include blanks, replicate and spiked samples, and reference materials. Summary and analysis of QC data are grouped by analyte and laboratory.

Quality of Water and Bed-Sediment Data

Field-blank samples for water were collected using inorganic- and organic-free water that contained no detectable concentrations of the analytes of interest. The blank water was processed in the field and analyzed identically to environmental samples. Field-blank samples were used to determine if contamination of samples occurred during sampling, processing, and analysis.

Most field-replicate samples were two or more samples collected in sequence, processed, and analyzed using identical methods. Two separate field samples were split into subsamples to make two replicate sets. For filtered samples, the filter was changed before the processing of each sample. Replicate sample data (environmental sample and additional QC samples) yield information on overall precision of the field and laboratory methods.

Blanks

Data for field-blank samples are summarized in table 5. UMeHg, FMeHg, PMeHg, and sulfate were not detected in any blank samples. DOC concentrations in blank samples from the 2002 national study were not detected above the method detection limit (MDL); six samples had concentrations that were detected at or below the MDL.

Concentrations of UTHg, FTHg, PTHg, and DOC (2004–05 national study) were greater than the respective MDLs in multiple blank samples. All six blank samples for UTHg had concentrations greater than the MDL of 0.04 nanogram per liter (ng/L). Concentrations of UTHg in all environmental samples for the 1998 National Mercury Pilot Study and the CHEY, DELR, and NECB regional studies were greater than the maximum blank concentration of 0.22 ng/L. For these studies, UTHg concentrations were measured values for unfiltered samples. Concentrations of UTHg for the 2002 and 2004–05 national studies and UMIS regional study were calculated values (sum of FTHg and PTHg); blank values are not reported for calculated UTHg.

Most (25 of 27) blank samples for FTHg had concentrations detected above the MDL of 0.04 ng/L, with concentrations ranging from 0.02 to 0.4 ng/L. There was, however, little overlap between concentrations of FTHg in blank samples and most environmental samples (fig. 2*A*). Concentrations of PTHg were detected in less than 50 percent (8 of 18) of the blank samples. Overlap of some of the higher PTHg concentrations in blanks with concentrations in environmental samples may indicate positive bias of PTHg for some environmental data values (fig. 2*B*).

For the 2004–05 national study, only two blank samples were available for DOC and SUVA analysis. To increase sample size for analysis, data for these two samples were combined with blank sample DOC data collected between 2003 and 2006 from site-specific Hg studies conducted by the NAWQA Program (Brigham and others, 2008). The site-specific studies used essentially the same collection, processing, and analytical methods as the 2002 and 2004–05 national studies. Twelve of 15 blank samples for DOC from the combined studies were detected at concentrations greater than the MDL; however, only one environmental sample for the 2004–05 national study had a DOC concentration less than the maximum blank concentration. Concentrations of DOC in all remaining environmental samples for the 2004–05 national study were at least three times greater than the maximum blank concentration. Blank-sample data for SUVA are included in this report even though SUVA is a calculated value (UV absorbance at 254 nanometers divided by DOC concentration). Only two environmental samples from the 2004–05 national study had SUVA concentrations that were less than or equal to the maximum blank concentration of 1.7 liters per milligram carbon per meter. SUVA data can be useful in interpretation and analysis of Hg and DOC data.

Replicates

Replicate data for water and bed-sediment samples are summarized in tables 6–7. Variability in sample data can be estimated by analyzing the distribution of replicate data using the standard deviation (SD) or the relative standard deviation (RSD, standard deviation divided by the mean concentration). For many water-quality analytes, the SD of replicate sets generally is uniform at low concentrations, but SD increases with increasing concentration. At higher concentrations, the RSD generally is uniform (Mueller and Titus, 2005). Variability of an analyte with a wide range of concentrations can be estimated by dividing the data into low- and high-concentration ranges based on the distribution of the SD or the RSD (Anderson, 1987). For low-concentration ranges, the mean SD of replicates is used to estimate variability; for high-concentration ranges, the mean RSD is used (Mueller and Titus, 2005).

For all replicate data, only replicate pairs with detected concentrations for each sample were used to estimate variability. As the first step in assessing variability of data collected during the national and regional studies, the SD and the RSD for each analyte were plotted (ordinate) against the mean replicate concentration (abscissa). The values at which concentrations were divided into low- and high-concentration ranges were estimated by fitting a loess [local regression nonparametric smoothing technique (S-Plus 7.0 for Windows, Insightful Corporation, Seattle, Wash.)] curve through the data and visually examining the SD or RSD plots for gaps in mean concentration and a change in the slope of the loess curve. For some analytes, there was no obvious difference in SD or RSD throughout the range of mean replicate concentration. Variability for these analytes was estimated by the RSD for the entire range of concentrations, which may overestimate the variability for a low range of concentration data.

In general, SDs for FTHg and FMeHg in the lower concentration ranges were similar to, though numerically slightly less than, the respective values for PTHg and PMeHg (table 6). In the higher concentration ranges, the RSD for FTHg was about one-third that for PTHg, whereas the RSD for FMeHg was about twice that for PMeHg (table 6). Moreover, RSD values for UTHg and UMeHg were as much as 4–5 times greater than the respective values of filtered or particulate values (tables 6, 7). These data suggest that the presence of particulate matter can have important effects on analyte variability.

SDs for DOC and sulfate in stream water in the low range of concentrations were 0.08 and 0.09 mg/L, respectively. RSD values for DOC, sulfate, and SUVA in high-concentration ranges also were low, less than or equal to 2.5 percent. RSD values for bed-sediment samples of LOI for the entire concentration range and AVS for the high-concentration range were 8.4 and 18.3 percent, respectively.

Table 5. Summary of data for stream-water field-blank samples, 1998–2005.

[See Appendix 2 for definition of column names. MDL, method detection limit; >, greater than; ng/L, nanogram per liter; CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; < less than; --, not applicable; 2002 and 2004–05, national studies of mercury conducted in 2002 and 2004–05; UMIS-reg, regional study of mercury in the Upper MIssissippi River Basin; mg/L, milligram per liter; NWQL, National Water Quality Laboratory; E, estimated; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; $L\ mgC^{-1}\ m^{-1}$, liter per milligram carbon per meter]

Analyte	Column name from Appendix 2. Field-blank sample data	Unit	Study	Laboratory	MDL	Number of blanks	Number of blanks > MDL	Concentration in blank		
								Minimum	90th percentile	Maximum
Mercury										
Unfiltered total mercury	UTHg	ng/L	CHEY-reg	USGS WMRL	0.04	6	6	0.05	0.19	0.22
Unfiltered methylmercury	UMeHg	ng/L	CHEY-reg	USGS WMRL	0.04	6	0	<0.001	--	<0.03
Filtered total mercury	FTHg	ng/L	2002 and 2004–05; CHEY-reg; UMIS-reg	USGS WMRL	0.04	27	25	0.02	0.22	0.4
Filtered methylmercury	FMeHg	ng/L	2002 and 2004–05; CHEY-reg; UMIS-reg	USGS WMRL	0.04	29	0	0.002	--	<0.04
Particulate total mercury	PTHg	ng/L	2002 and 2004–05; UMIS-reg	USGS WMRL	variable[1]	18	8	<0.062	0.495	0.644
Particulate methylmercury	PMeHg	ng/L	2002 and 2004–05; UMIS-reg	USGS WMRL	variable[1]	21	0	<0.008	--	<0.038
Organic carbon										
Dissolved organic carbon	DOC	mg/L	2002	USGS NWQL	0.3	13	0	E 0.2	--	E 0.3
Dissolved organic carbon	DOC	mg/L	2004-05[2]	USGS NRP OCTL	0.2	15	12	0.1	0.4	0.5
Specific ultraviolet absorbance	SUVA	$L \times mgC^{-1}\ m^{-1}$	2004-05[2]	USGS NRP OCTL	none[3]	11	--	0.1	0.9	1.7

Table 5. Summary of data for stream-water field-blank samples, 1998–2005.—Continued

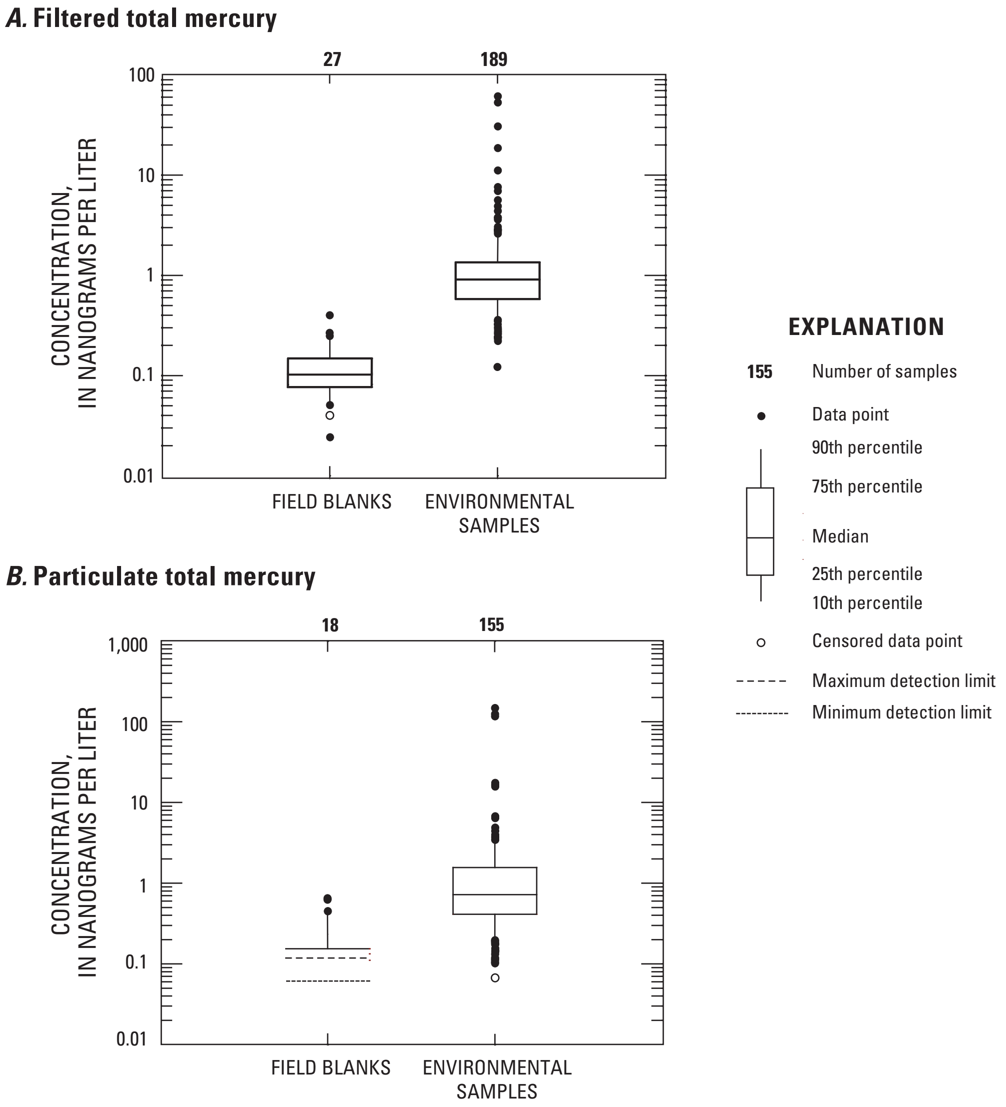
[See Appendix 2 for definition of column names. MDL, method detection limit; >, greater than; ng/L, nanogram per liter; CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; < less than; --, not applicable; 2002 and 2004–05, national studies of mercury conducted in 2002 and 2004–05; UMIS-reg, regional study of mercury in the Upper MIssissippi River Basin; mg/L, milligram per liter; NWQL, National Water Quality Laboratory; E, estimated; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; L mgC^{-1} m^{-1}, liter per milligram carbon per meter]

Analyte	Column name from Appendix 2. Field-blank sample data	Unit	Study	Laboratory	MDL	Number of blanks	Number of blanks > MDL	Concentration in blank Minimum	Concentration in blank 90th percentile	Concentration in blank Maximum
Major ions										
Sulfate	Sulfate	mg/L	2002	USGS NWQL	0.1	12	0	<0.1	--	<0.1
Sulfate	Sulfate	mg/L	2004–05; UMIS-reg	USGS NWQL	0.18	4	0	<0.18	--	<0.18

[1] Analytical results for PTHg and PMeHg are reported on a ng/L basis by dividing the mass of PTHg or PMeHg on a filter by the volume of stream water filtered. The MDL varies depending on the volume of water filtered (DeWild and others, 2004; Olund and others, 2004). See Appendix 2 for detection limits.

[2] Only two blank samples for DOC and SUVA were available for the 2004–05 national study. These data were combined with DOC and SUVA blank data from detailed USGS mercury studies conducted from 2003 through 2006 to increase sample size for analysis.

"[3] No MDL; SUVA is a calculated value: UV absorbance at 254 nanometers divided by DOC concentration.



A. Filtered total mercury

B. Particulate total mercury

Figure 2. Statistical distribution of field blanks and stream-water environmental samples of (*A*) filtered total mercury (FTHg) concentrations and (*B*) particulate total mercury (PTHg) concentrations, 1998–2005.

Table 6. Estimates of variability for lower and higher concentration ranges of stream water and bed sediment replicate samples, 1998–2005.

[See Appendix 3 for definition of column names. SD, standard deviation; RSD, relative standard deviation; ng/L, nanogram per liter; 2002, national study of mercury conducted in 2002; CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; mg/L, milligram per liter; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; NWQL, National Water Quality Laboratory; 2004–05, national study of mercury conducted in 2004–05; SGL, Sulfur Geochemistry Laboratory]

Analyte	Column name from Appendix 3. Field-replicate sample data	Unit	Study	Laboratory	Low-concentration range			High-concentration range		
					Number of replicate sets	Range	Variability (mean SD)	Number of replicate sets	Range	Variability (mean RSD, in percent)
					Stream water					
Mercury										
Filtered total mercury	FTHg	ng/L	2002; CHEY-reg; UMIS-reg	USGS WMRL	18	0.22–1.37	0.09	5	1.72–10.9	1.3
Filtered methyl-mercury	FMeHg	ng/L	2002; CHEY-reg, UMIS-reg	USGS WMRL	10	0.05–0.45	0.01	1	3.48–4.06	10.9
Particulate total mercury	PTHg	ng/L	2002; UMIS-reg	USGS WMRL	17	0.123–2.34	0.111	6	15.3–162	4.1
Particulate methylmercury	PMeHg	ng/L	2002; UMIS-reg	USGS WMRL	17	0.023–0.287	0.014	1	1.83–1.97	5.2
Organic carbon										
Dissolved organic carbon	DOC	mg/L	UMIS-reg[1]	USGS NRP OCTL	16	0.9–11.1	0.08	2	40.0–42.0	1.3
Major ions										
Sulfate	Sulfate	mg/L	2002; CHEY-reg; UMIS-reg	USGS WMRL, USGS NWQL	14	1.8–69.6	0.09	1	807–811	0.3
					Bed sediment					
Sulfur										
Acid-volatile sulfide	Pct_AVS_ WetWt	percent	2002 and 2004–05; UMIS-reg	USGS SGL	9	6.46^{-17}–4.16^{-4}	6.51^{-5}	7	2.78^{-4}–1.14^{-3}	18.3

[1] Only one replicate set for DOC was available for the UMIS regional study. These data were combined with DOC replicate data from detailed USGS mercury studies conducted from 2003 through 2006 to increase sample size for replicate analysis.

Table 7. Estimates of variability for stream water and bed sediment replicate samples, 1998–2005.

[See Appendix 3 for definition of column names. RSD, relative standard deviation; ng/L, nanogram per liter; CHEY-reg, regional study of mercury in the Cheyenne–Belle Fourche River Basins; USGS, U.S. Geological Survey; WMRL, Wisconsin Mercury Research Laboratory; mg/L, milligram per liter; 2002, national study of mercury conducted in 2002; NWQL, National Water Quality Laboratory; $L\times mgC^{-1}\times m^{-1}$, liter per milligram carbon per meter; UMIS-reg, regional study of mercury in the Upper Mississippi River Basin; NRP OCTL, National Research Program Organic Carbon Transformations Laboratory; ng/g, nanogram per gram; 2004–05, national study of mercury conducted in 2004–05; DELR-reg, regional study of mercury in the Delaware River Basin]

Analyte	Column name from Appendix 3. Field-replicate sample data	Units	Study	Laboratory	Number of replicate sets	Range	Variability (mean RSD, in percent)
Stream Water							
Mercury							
Unfiltered total mercury	UTHg	ng/L	CHEY-reg	USGS WMRL	5	2.36–63.5	7.2
Unfiltered methylmercury	UMeHg	ng/L	CHEY-reg	USGS WMRL	3	0.06–0.20	19.8
Organic carbon							
Dissolved organic carbon	DOC	mg/L	2002	USGS NWQL	13	1.2–7.7	2.5
Specific ultraviolet absorbance	SUVA	$L\times mgC^{-1}\times m^{-1}$	UMIS-reg[1]	USGS NRP OCTL	10	2.5–4.8	2.1
Streambed sediment							
Mercury							
Total mercury	STHg	ng/g	2002 and 2004–05; CHEY-reg; DELR-reg[2]; UMIS-reg	USGS WMRL	20	2.34–678	11.9
Methylmercury	SMeHg	ng/g	2002 and 2004–05; CHEY-reg; UMIS-reg	USGS WMRL	12	0.05–5.17	14.5
Organic matter							
Loss on ignition	LOI	percent	2002 and 2004–05; UMIS-reg	USGS WMRL	17	0.005–0.16	8.4

[1] Only one replicate set for SUVA was available for the UMIS regional study. These data were combined with SUVA replicate data from detailed USGS mercury studies conducted from 2003 through 2006 to increase sample size for replicate analysis.

[2] The two replicate samples for the DELR regional study were split samples.

Quality of Fish Data

Laboratory QC samples for fish included procedural blanks, replicate samples, spiked samples, and certified and standard reference materials. Procedural blanks measure the amount of Hg that may be introduced in a sample during sample processing in the laboratory. Replicate samples, created by taking subsamples from the original homogenized fish sample, were used to measure the variability in the analytical procedures for fish analysis. Laboratory spiked samples were used to measure the bias and precision of analytical methods by determining the amount of spike recovery in a sample. Low recovery of the spiked analyte can indicate Hg degradation, analytical interference from the sample matrix, and (or) poor analytical recovery. Reference materials from the USGS CERC, the National Institute of Standards and Technology (NIST), and the National Research Council Canada (NRCC) were used to assess the accuracy of analytical methods. Reference materials used were CERC whole striped bass (material I.D. STB), NIST albacore tuna fillet (SRM 50) and mussel tissue (SRM 2976), NRCC dogfish (*Squalus acanthias*) muscle (DORM-1 and DORM-2), NRCC dogfish liver (DOLT-2), and NRCC lobster hepatopancreas (TORT-2) (table 8). Laboratory QC samples were available for the national studies and the UMIS regional study.

QC results for fish sampled during the 1998 National Mercury Pilot Study and analyzed at the CERC are discussed in Brumbaugh and others (2001) and are summarized here. Concentrations in eight of nine method blank samples were near or less than the instrument detection level. The one elevated blank sample resulted in an increased MDL for samples analyzed in the digestion block, but virtually all sample concentrations were considerably greater than the blank concentration. As a result, there was no indication of systematic contamination of samples due to sample collection, processing, and analysis. The RSDs for the triplicate samples ranged from 0.6 to 7.5 percent (n=8) and averaged 2.6 percent. Percent recoveries for the predigestion spikes of MeHg averaged 102.3 percent (SD 4.2, n=16). Percent recoveries for the postdigestion spikes of divalent Hg (Hg^{2+}) averaged 98.5 percent (SD 5.1, n=25). Measured concentrations of the reference tissue samples for the CERC whole striped bass, NIST albacore tuna fillet, and NRCC DORM-1 materials were in good agreement with the certified ranges (table 8). Overall, QC results for the 1998 National Mercury Pilot Study fish samples analyzed at the CERC indicated low bias and good reproducibility.

QC data for the 2002 fish samples analyzed for Hg at the NQWL included concentrations measured in blank samples and recoveries of reference tissue samples (J.R. Garbarino, USGS, written commun., 2007). Concentrations of Hg in 11 blank samples were less than or equal to the MDL. For reference samples analyzed from February 2002 through February 2003, measured concentrations of the reference tissue samples for NRCC DOLT-2 and NRCC DORM-2 were within one SD of certified ranges (table 8). For the same time period, recoveries averaged 115.5 percent (SD 34.9 percent, n=10) for DOLT-2 and 88.5 percent (SD 17.4 percent, n=9) for DORM-2. Overall, the quality-control results associated with the 2002 fish samples analyzed at the NWQL indicated low bias and moderate variability.

For the 2004–05 fish samples analyzed at the TERL, 8 of 19 procedural blanks had Hg concentrations that were slightly greater than the MDL. These detections of Hg in blank samples with concentrations equal to or slightly greater than the MDLs do not affect the interpretation of Hg in the 243 fish samples analyzed at the TERL because the minimum sample concentration (0.056 microgram per gram ([µg/g]), dry weight) was at least 6 times greater than the highest blank equivalent concentration (0.009 µg/g, dry weight). The RSDs for the replicate samples were between 0 and 8 percent (n=23), with an average of 2 percent. Percent recoveries for the spike samples averaged 99.7 percent (SD 3.3, n=23). Measured concentrations of the reference tissue samples for the NRCC DOLT-2 and DORM-2 materials were within certified ranges (table 8). Average percent recoveries for the reference materials were 104.2 percent (SD=6.6 percent, n=19) for DOLT-2 and 99.7 percent (SD 2.5, n=19) for DORM-2. QC results for the 2004–05 national study fish samples analyzed at the TERL indicated low bias and good reproducibility.

QC results for fish sampled during the UMIS regional study and analyzed at River Studies Center, University of Wisconsin, La Crosse, Wis., are discussed in Christensen and others (2006) and are summarized here. The average RSD for 60 triplicate subsamples of homogenized fish was 4.5 percent, and values ranged from 0.4 to 8.3 percent. Percent recoveries of predigestion subsamples of homogenized fish averaged 96.5 percent (n=60) and ranged from 84.1 to 112.1 percent. Measured concentrations of reference tissue samples of NIST mussel tissue, NRCC lobster hepatopancreas, and NRCC dogfish muscle were within the certified ranges (table 8). The QC results for the UMIS regional study fish samples analyzed at the University of Wisconsin, La Crosse, River Studies Center indicated low bias and good reproducibility.

Ancillary Data

Selected ancillary data for geographic information, basin hydrologic estimates, and Hg source estimates are available for the basins upstream from most Hg study sites. Ancillary data are not included for 18 sites because specific land-cover data were not available or basin boundaries were not determined. The 18 sites are listed in Appendix 5.

Geographic Information

Boundaries representing the basin upstream from study sites were delineated by USGS personnel from a variety of sources. Most basin boundaries were previously compiled for the USGS NAWQA Program from 1:24,000-scale to

Table 8. Measured total mercury concentrations for certified and standard reference materials from the Columbia Environmental Research Center, National Institute of Standards and Technology, and National Research Council Canada used for laboratory quality assurance of fish samples, 1998–2005.

[µg/g, micrograms per gram; wt, weight; CERC, Columbia Environmental Research Center; USGS, U.S. Geological Survey; NIST, National Institute of Standards and Technology; NRCC, National Research Council Canada; DORM, dogfish (*Squalus acanthias*) muscle; DOLT, dogfish liver; NWQL, National Water Quality Laboratory; TERL, Trace Element Research Laboratory, Texas A&M University; UMIS, Upper Mississippi River Basin; RSC, River Studies Center, University of Wisconsin, La Crosse, Wis. Data for 1998 from Brumbaugh and others (2001); data for UMIS regional study from Christensen and others (2006)]

Reference material	Laboratory	Number of samples	Measured concentration, µg/g, dry wt		Certified concentration range, µg/g, dry wt
			Mean	Standard deviation	
1998 National Mercury Pilot Study					
CERC whole striped bass	USGS CERC	3	2.21	0.01	2.26 ± 0.51
NIST albacore tuna fillet		3	0.99	0.04	0.95 ± 0.10
NRCC DORM-1		3	0.9	0.06	0.80 ± 0.07
2002 national study					
NRCC DOLT-2	USGS NWQL	10	2.47	0.75	2.14 ± 0.28
NRCC DORM-2		9	4.11	0.81	4.64 ± 0.26
2004–05 national study					
NRCC DOLT-2	TERL	19	2.23	0.14	2.14 ± 0.28
NRCC DORM-2		19	4.63	0.12	4.64 ± 0.26

UMIS regional study

Reference material	Laboratory	Number of samples	Mean, µg/g, dry wt	95-percent confidence interval, µg/g, dry wt	Certified concentration range, µg/g, dry wt
NIST mussel tissue	RSC	12	0.0605	0.0575–0.0641	0.061 ± 0.0036
NRCC lobster hepatopancreas		12	0.258	0.242–0.274	0.27 ± 0.06
NRCC dogfish muscle		11	0.841	0.795–0.887	0.798 ± 0.074

1:250,000-scale digital topographic and hydrologic maps (Nakagaki and Wolock, 2005). Other basin boundaries were generated using 30-meter (m) resolution Elevation Derivatives for National Applications reach catchments (U.S. Geological Survey, 2002). All digital basin boundaries were managed both as vector data sets (hereinafter referred to as “coverages”) and as 30-m resolution raster data sets (hereinafter referred to as “grids”). Drainage areas for the basins were calculated from the coverages.

Several ancillary data sets were processed in a Geographic Information System (GIS) to characterize natural features and human influences within the Hg study basins. The basin boundaries gridded at 30-m resolution for most variables were intersected with each national ancillary data layer to calculate a basin average for the ancillary data feature. For land cover, percentages of land-cover classes were calculated. The basin boundary coverages were used to calculate total stream length and number of historical and active Hg and gold mining sites in the basin.

Land-cover information was obtained from 30-m National Land Cover Data (NLCD) (U.S. Geological Survey, 1999), which were based on satellite imagery from the early to mid-1990s (Vogelmann and others, 2001), and USGS Land Use and Land Cover (LULC) data (Price and others, 2007), which were derived from aerial photography from the 1970s to mid-1980s. The NLCD was enhanced with selected land-use categories of LULC data, as described in Nakagaki and Wolock (2005), because the LULC data were a better source for some land categories that are difficult to distinguish using only satellite imagery. This “enhanced” NLCD includes 21 land-cover classifications from the original NLCD plus an additional four categories from the LULC (LULC tundra, NLCD/LULC forested residential, LULC residential, and LULC orchards/vineyards/other) (Nakagaki and Wolock, 2005).

The enhanced NLCD was used to derive percentages of land-cover classes in each basin. The enhanced NLCD also was classified into broader categories representing urban, agriculture, forest, wetland, undeveloped, and other land cover. To address the possibility that conditions observed at a sampling site were influenced more by land cover closer to the site than by land cover farther from the site, land-cover percentages weighted by the inverse distance from the site also were calculated and are included in the ancillary database.

Key soil characteristics, including percent organic matter content by weight, soil erodibility factor, percent land surface slope, depth to seasonally high water table, percent hydric soils, soil porosity, and soil drainage class, were compiled from State Soil Geographic (STATSGO) data (U.S. Department of Agriculture, 1994). Percent organic matter, soil erodibility factor, and land surface slope were obtained from the tabular data files presented in Wolock (1997) and were linked by mapping unit identification code to a 100-m resolution national grid of STATSGO geographic mapping units. Hydric soils and soil porosity were calculated for each mapping unit using methods from Wolock (1997). Soil drainage class was compiled from Schwarz and Alexander (1995), who converted the categorical classes into numeric values. Hydric soils, soil porosity, and soil drainage class also were linked to the 100-m soils mapping unit grid. For each basin, a weighted average was calculated for each soil characteristic based on the areas of the soils mapping units within the basin.

Elevation data at 30-m resolution were extracted from USGS National Elevation Data (U.S. Geological Survey, 2001). The 30-m data were resampled to 100-m resolution, and the 100-m data were used to calculate the elevation of each sampling site and the mean and maximum elevation of each basin. Stream segments from the 1:100,000-scale medium-resolution National Hydrography Dataset (U.S. Geological Survey and U.S. Environmental Protection Agency, 2003) were clipped to the study basin boundaries to calculate total stream length within each basin.

The 2000 Census block group boundaries (U.S. Census Bureau, 2001) gridded at 30-m resolution were used with 2000 block group population density calculated from the Census Summary File 1 published by GeoLytics (2001) to derive a weighted average 2000 population density by block group for each basin. The source of impervious surface area was a 1-kilometer (km) resolution grid prepared by Elvidge and others (2004).

Basin Hydrologic Estimates

Mean annual precipitation at 1-km resolution was derived from the Parameter-elevation Regressions on Independent Slopes Model (PRISM). PRISM uses point data, a digital elevation model, and other spatial data to generate estimates of precipitation and other climatic parameters (Daly and others, 1994; Daly and others, 1997). PRISM mean annual precipitation (1961–1990) at 2-km resolution was obtained from the Spatial Climate Analysis Service at Oregon State University. Mean annual precipitation was estimated for each basin by using the PRISM data.

National 1-km resolution grids (D.M. Wolock, USGS, written commun., 2007) were used to calculate a mean for each basin for base-flow index, potential and actual evapotranspiration, and topographic wetness index. Base-flow index is the ratio of base flow (component of streamflow that can be attributed to ground-water discharge into streams) to total flow, expressed as a percentage. The base-flow index grid was created by interpolating base-flow index point values estimated for USGS streamflow gages (Wolock, 2003a, 2003b). PRISM temperature data (Daly, 2006) and the Hamon equation (Hamon, 1961) were used by Wolock to generate a 1-km grid of mean annual potential evapotranspiration (D.M. Wolock, USGS, written commun., 2007). PRISM climate data also were used in a water-balance model to estimate actual evapotranspiration (Wolock and McCabe, 1999). Average topographic wetness index was developed from 1-km resolution digital elevation model data (Wolock and McCabe, 2000). Mean annual runoff (1951–1980) characteristics were derived from Gebert and others (1987).

Mercury Source Estimates

Data from the National Atmospheric Deposition Program (NADP) provided information about measured Hg and sulfate wet deposition. Annual wet deposition data for 2000–2003 for sites in the Mercury Deposition Network (Roger Claybrooke, Illinois State Water Survey, written commun., 2005) were averaged for 2000–2003. The station averages were converted to a grid surface by using inverse distance weighting interpolation for the eastern part of the United States. Data were available for only seven sites in the western part of the United States, and this was deemed insufficient for interpolation. Instead, the mean deposition of those seven sites was assigned to the western part of the country. Digital isopleths maps of annual sulfate wet deposition data for 2000–2003 were downloaded from the NADP (National Atmospheric Deposition Program, 2006). One surface map representing an average sulfate wet deposition for 2000–2003 was generated using GIS software to compute the mean from the four individual sulfate maps. The average Hg and sulfate wet deposition maps for 2000–2003 were clipped to the basin boundaries to calculate basin mean wet deposition of Hg and sulfate.

Modeled wet and dry Hg deposition rates were estimated using results from the Trace Elements Analysis Model (TEAM) (Seigneur and others, 2004). The resolution of each TEAM grid cell was 100-km 100-km.

Large-scale potential sources of Hg from past or current mining operations were extracted from the Mineral

Availability System/Mineral Industry Location System (MAS/MILS) (V.C. Stephens, USGS, written commun., 2004), which is part of the Mineral Resources Data System (MRDS) (U.S. Geological Survey, 2004). Mining sites in each basin were identified as (1) Hg mining operations, in general, (2) Hg "producers," (3) gold mining operations, in general, and (4) gold "producers." Producers included current or past producer mining operations.

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Appendixes

Appendix Data

These data files are included as part of U.S. Geological Survey (USGS) Data Series 307 and are available for download at *http://pubs.usgs.gov/ds/307/*. See report text for details about the study and for information on sources and compilation of ancillary data. The data tables are available for download in two file formats, Microsoft© Excel (.xls) and comma-separated values (.csv) text. The Excel files are formatted to properly display the data. Users with software that reads Excel files are encouraged to download the Excel versions of the data files. If you cannot read Excel files, .csv files are provided. The first row of each data table contains USGS National Water Information System (NWIS) numeric and alpha parameter codes and parameter descriptions (example, P_63745_Total mercury, biota, tissue, recoverable, dry weight, nanograms per gram). The second row contains abbreviated parameter descriptions (example, THG_TIS_DW). Analytes with no parameter code listed are not entered in NWIS.

Five data tables are included in this data series:

Appendix 1. Water and sediment sample data.

Mercury and methylmercury sample data for water and streambed sediment, including field parameters and additional water-quality analytes, collected during 1998–2005 at stream and river sampling sites. The Excel file can be accessed at *ds307_Appendix01.xls*. The .csv files can be accessed at *ds307_Appendix01.notes.csv* and *ds307_Appendix01.data.csv*.

Appendix 2. Field-blank sample data.

Feld-blank sample data for mercury and methylmercury in water, and additional water-quality analytes. The Excel file can be accessed at *ds307_Appendix02.xls*. The .csv files can be accessed at *ds307_Appendix02.notes.csv* and *ds307_Appendix02.data.csv*.

Appendix 3. Field-replicate sample data.

Field-replicate sample data for mercury and methylmercury in water and streambed sediment, and additional water-quality analytes. The Excel file can be accessed at *ds307_Appendix03.xls*. The .csv files can be accessed at *ds307_Appendix03.notes.csv* and *ds307_Appendix03.data.csv*.

Appendix 4. Fish data.

Mercury sample data, including physical characteristics and additional sample information, for fish collected during 1998–2005 at stream and river sampling sites. The Excel file can be accessed at *ds307_Appendix04.xls*. The .csv files can be accesssed at *ds307_Appendix04.notes.csv* and *ds307_Appendix04.data.csv*.

Appendix 5. Ancillary data.

Selected data for geographic information, basin hydrologic estimates, and mercury source estimates for basins upstream from most study sites. The Excel file can be accessed at *ds307_Appendix05.xls*. The .csv files can be accessed at *ds307_Appendix05.notes.csv* and *ds307_Appendix05.data.csv*.

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