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Total Mercury, Methylmercury, and Carbon and Nitrogen Stable Isotope Data for Biota from Selected Streams in Oregon, Wisconsin, and Florida, 2002–04

Data Series 349

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

Cover: Cutthroat trout from Lookout Creek, Oregon (upper left); electroshocking for fish in Lookout Creek, Oregon (upper right); collecting invertebrates in the St. Marys River, Florida (lower right); crayfish from Little Wekiva River, Florida (lower left); grass shrimp from the St. Marys River, Florida (center). (All photographs by the authors.)

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Foreword

The U.S. Geological Survey (USGS) is committed to providing the Nation with credible 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, now 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 condition 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 1991-2001, 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).

Multiple national and regional assessments are ongoing in the second decade (2001—2012) of the NAWQA Program as 42 of the 51 Study Units are reassessed. These assessments extend the findings in the Study Units by determining 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. In addition, national syntheses of information on pesticides, volatile organic compounds (VOCs), nutrients, selected 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
Acting Associate Director for Water

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Conversion Factors

Multiply	By	To obtain
kilometer (km)	0.6214	mile (mi)
square kilometer (km^2)	0.3861	square mile (mi^2)
nanogram (ng)	0.03527×10^{-9}	ounce avoirdupois (lb)
microgram (µg)	0.03527×10^{-6}	gram (g)
kilogram (kg)	2.205	pound, avoirdupois (lb)
millimeter (mm)	0.03937	inch (in.)
pounds per square inch (lb/in^2)	6.895	kilopascal (kPa)

Concentrations of chemical constituents in water are given either in milligrams per liter (mg L^{-1}), micrograms per liter (µg L^{-1}), or nanograms per liter (ng L^{-1}).

Datum

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

Abbreviations and Acronyms

C:N	elemental composition, ratio of carbon to nitrogen
CRM	certified reference material
CVAFS	cold vapor atomic fluorescence spectroscopy
CVAS	cold vapor atomic fluorescence
$\delta^{13}C$	stable isotope ratio of carbon ($^{13}C/^{12}C$) expressed per mil (‰)
$\delta^{15}N$	stable isotope ratio of nitrogen ($^{15}N/^{14}N$) expressed per mil (‰)
DOC	dissolved organic carbon
DTH	depositional-targeted habitat (periphyton or invertebrates)
IAEA	International Atomic Energy Agency
MeHg	methylmercury
NAWQA	National Water-Quality Assessment
NIST	National Institute of Standards and Technology
NWIS	National Water Information System of the U.S. Geological Survey
QA/QC	quality assurance/quality control
‰	per mil (per thousand)
%	percent
RPD	relative percent difference between duplicate concentrations
RSD	percent relative standard deviation of the concentrations
RTH	richest-targeted habitat (periphyton or invertebrates)
SRM	standard reference material
THg	total mercury
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey

Total Mercury, Methylmercury, and Carbon and Nitrogen Stable Isotope Data for Biota from Selected Streams in Oregon, Wisconsin, and Florida, 2002–04

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Abstract

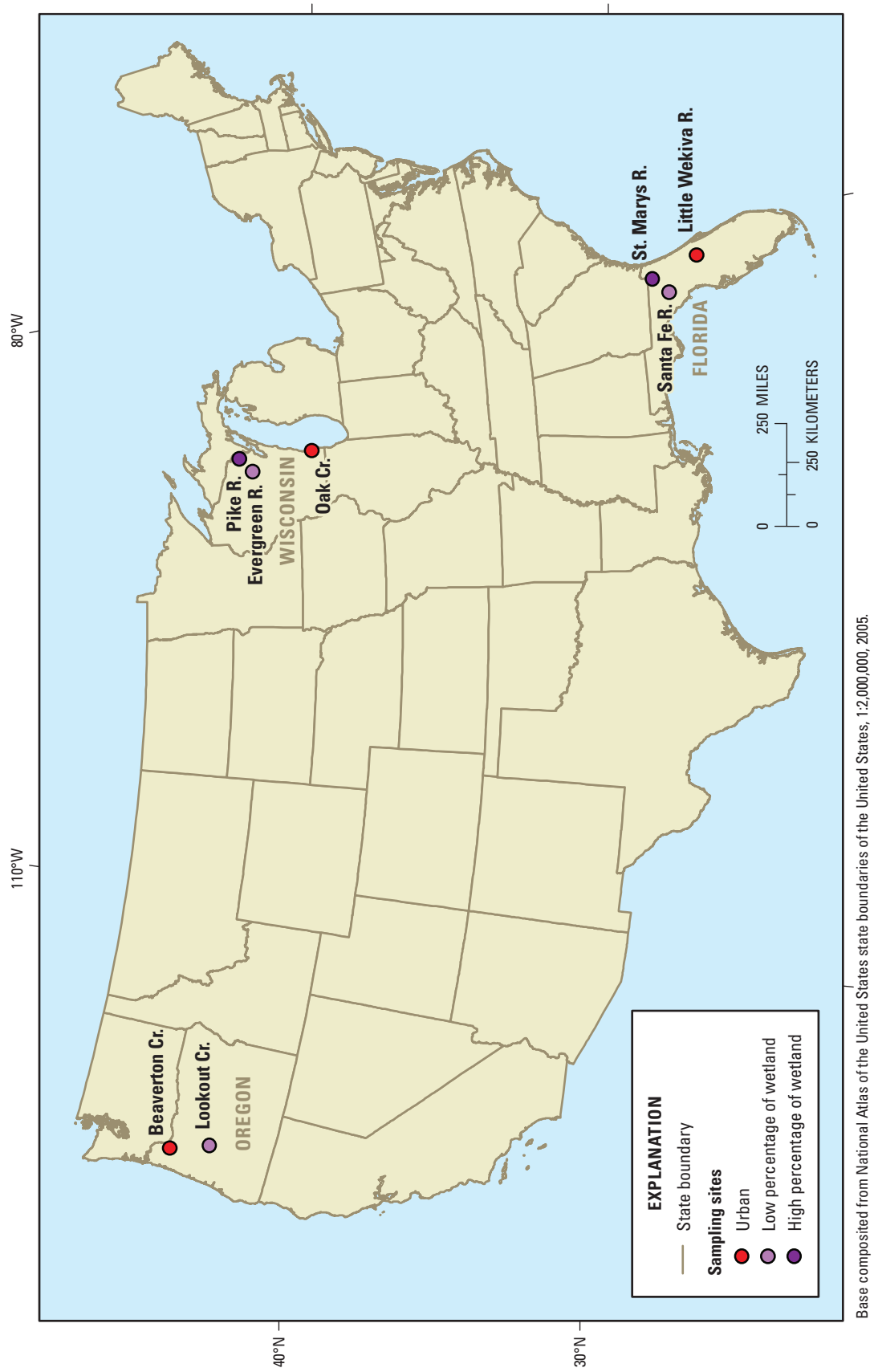
The U.S. Geological Survey National Water-Quality Assessment Program conducted a multidisciplinary study to investigate the bioaccumulation of mercury from 2002 to 2004. Study areas were located in Oregon, Wisconsin, and Florida. Each study area included one urban site, and one or two nonurban sites that had the following attributes: high-percent wetland or low-percent wetland. Periphyton, macroinvertebrates, and forage fish were collected twice per year (during 2003 and 2004) to capture seasonality. Top predators, specifically largemouth bass (*Micropterus salmoides*), brown trout (*Salmo trutta*), and cutthroat trout (*Oncorhynchus clarkii*), were collected once per year (Oregon, Wisconsin, and Florida in 2003; Florida only in 2004). All biota were identified to the lowest possible taxonomic category and were analyzed for mercury and stable carbon and nitrogen isotopes. Periphyton and invertebrates were analyzed for total mercury and methylmercury; fish were analyzed for total mercury only. This report presents (1) methodology and data on mercury, methylmercury, stable isotopes, and (2) other ecologically relevant measurements in biological tissues of periphyton, invertebrates, forage fish, and predator fish.

Introduction

Bioaccumulation of mercury (Hg) in aquatic organisms, particularly fish that are considered recreationally or commercially important, has generated increasing public concern during the last decade. Recent advances have been made in describing some of the factors that influence the transport and the geochemical cycling of Hg in freshwater ecosystems. Even so, specific environmental controls and the relative importance of abiotic and biotic processes responsible for the uptake and biomagnification of Hg by aquatic organisms remain poorly characterized, especially in stream ecosystems. From 2002 to 2004, the U.S. Geological Survey (USGS) National Water-Quality Assessment Program, in collaboration with the Toxics Substances Hydrology Program, conducted studies of stream ecosystems across a wide range of environmental settings to address this information gap. Streambed sediment, surface water, and biota were sampled intensively across eight diverse stream ecosystems in Oregon, Wisconsin, and Florida; this report presents the biological data. The following environmental factors were sampled: streamflow, pH, water temperature, dissolved organic carbon (DOC), sulfide/sulfate, major ions, and nutrients. Companion reports by Marvin-DiPasquale and others (2008) and Brigham and others (2008) present environmental and geochemical data for streambed sediment and surface water, including total Hg and methylmercury (MeHg), methylation/demethylation rates, and characterization of DOC.

Site Description

Sampled streams (tables 1 and 2) were selected to represent a gradient in watershed and stream characteristics, such as land use, Hg loading, hydrogeology, water chemistry, ecological community structure, trophic complexity, and climate (Bell and Lutz, in press; Scudder and others, in press). The streams were located in Oregon, Wisconsin, and Florida (fig. 1). The sampling reach for each stream was selected based on proximity to an operational streamflow gaging station, ease of access, and historical data on ecology and hydrology. The length of each sampling reach was determined by stream size and ranged from about 0.5 to 2 km.



Base compositied from National Atlas of the United States state boundaries of the United States, 1:2,000,000, 2005.
Albers Conical Equal Area Projection, referenced to North American Datum of 1983.

Figure 1. Locations of stream sites sampled for mercury bioaccumulation as part of the USGS National Water-Quality Assessment Program, 2003–04.

Table 1. Stream names, abbreviated names as used in report text and tables, USGS site identifications, and sampling site locations.

[USGS, U.S. Geological Survey; ddd°mm'ss", degrees, minutes, and seconds; NAD 27, North American Datum 1927]

Stream name	Short stream name	USGS station ID	Sampling site latitude (degrees north, NAD 27) (ddd°mm'ss")	Sampling site longitude (degrees north, NAD 27) (ddd°mm'ss")
Lookout Creek near Blue River, Oregon	Lookout Creek, OR	14161500	44°12'35"	122°15'20"
Beaverton Creek, at SW 216th Avenue near Orenco, Oregon	Beaverton Creek, OR	14206435	45°31'15"	122°53'54"
Pike River at Amberg, Wisconsin	Pike River, WI	04066500	45°30'00"	88°00'00"
Evergreen River below Evergreen Falls near Langlade, Wisconsin	Evergreen River, WI	04075365	45°03'57"	88°40'34"
Oak Creek at South Milwaukee, Wisconsin	Oak Creek, WI	04087204	42°55'30"	87°52'12"
St. Marys near MacClenny, Florida	St. Marys River, FL	02231000	30°21'31"	82°04'54"
Santa Fe River at Fort White, Florida	Santa Fe River, FL	02322500	29°50'55"	82°42'55"
Little Wekiva River near Longwood, Florida	Little Wekiva River, FL	02234998	28°42'07"	81°23'32"

Table 2. Summary of site characteristics.

[Modified from Bell and Lutz, in press. km^2, square kilometer; DOC, dissolved organic carbon; mg/L, milligram per liter]

Short stream name	Basin area (km^2)	Dominant land cover	Percent wetland	DOC mean (range) (mg/L)	pH mean (range)	Sulfate mean (range) (mg/L)
Lookout Creek, OR	62.4	forest	0	0.9 (0.5 - 2.9)	7.2 (6.8 - 7.7)	0.2 (0.2 - 0.4)
Beaverton Creek, OR	95.6	urban	0.2	4.3 (3.2 - 7.3)	7.3 (7.0 - 7.5)	7.7 (4.4 - 11.6)
Pike River, WI	660	forest	18	7.6 (2.2 - 18.7)	7.8 (7.2 - 8.5)	7.6 (4.0 - 10.4)
Evergreen River, WI	167	forest	9.3	3.5 (1.7 - 15.6)	7.8 (7.2 - 8.5)	8.4 (5.6 - 10.8)
Oak Creek, WI	64.7	urban	8.1	7.1 (3.5 - 13.1)	7.6 (7.1 - 7.9)	81.0 (21.5 - 135)
St. Mary's River, FL	1,810	forest/forested wetland	27.2	40.5 (8.8 - 61.0)	4.2 (2.9 - 7.5)	2.6 (1.0 - 7.9)
Santa Fe River, FL	2,640	forest	16.8	6.7 (1.9 - 77)	7.3 (5.7 - 7.9)	27.4 (2.4 - 33.6)
Little Wekiva River, FL	115	urban	3	4.3 (1.7 - 15.6)	7.2 (6.9 - 8.7)	17.9 (<0.2 - 23.8)

Data Collection

Trace-metal clean techniques were used for all sample collection and processing (U.S. Environmental Protection Agency, 1996; Cleckner and others, 1999; Olson and DeWild, 1999; Lewis and Brigham, 2004). A list of the taxa collected in all study areas is provided in appendix 1. The list includes available descriptions of associated habitat, life history, and feeding strategies.

Periphyton Collection and Processing

Field protocols used for collecting periphyton (attached algae) for Hg and stable isotope analyses are described in detail in Bell and Scudder (2005). Periphyton samples were collected seasonally at each site: once in spring during high flow, and once in fall during low flow. Two habitat types were targeted at each site: (1) depositional areas (referred to as "depositional-targeted habitat," or DTH) with relatively high organic content, and (2) either cobbles or woody snags, whichever was considered the more productive habitat (referred to as "richest-targeted habitat," or RTH) for periphyton in a specific system. Within each stream, the DTH sample consisted of a composite of algal material from three depositional areas, and the RTH sample consisted of a composite from woody snags or cobbles from five locations in the stream. Samples were elutriated in the field to separate periphyton from sand and debris by shaking and decanting, adding 50 mL reagent water, shaking and decanting twice, and vacuum filtering (<10 lb/in^2) onto quartz fiber filters. The filters were stored in Petri dishes (Teflon® for Hg, polystyrene for other analyses) and frozen until analysis. Subsamples of each periphyton sample were retained and preserved in 5 percent buffered formalin for taxonomic identification.

Invertebrate and Fish Collection and Processing

Aquatic invertebrates, forage fish, and top predator fish were collected and processed as described in detail by Scudder and others (in press). Additional collection methods also are described in Moulton and others (2002). Two species of invertebrates and two species of forage fish were targeted at each site, and each group was collected in spring and fall from a variety of habitat types within each stream. Targeted invertebrates typically were larval stages of insects, although other aquatic invertebrates (such as amphipods, grass shrimp, or snails) and some emerging adult insects also were collected. All invertebrates were identified to the lowest possible taxonomic category in the field, rinsed with site water and deionized water, picked clean of visible debris, and divided into three composites with a targeted sample mass of at least 1 g wet weight per composite. These composites were stored in Teflon® vials on dry ice and maintained frozen until analysis.

Fish specimens were processed individually in the field. Forage fish were identified, weighed, and measured (total length), after which the heads and gut tracts were removed. Each individual carcass (whole fish minus head and gut tract), head, and gut tract was placed in a separate plastic vial or zip-seal plastic bag. These bags were double sealed in another plastic zip-seal bag and frozen until analysis. Each top predator fish was filleted, and its sagittal otoliths (or whole head) and gut tract were removed. Fillets, heads, and gut contents were stored in double zip-seal bags; otoliths were cleaned, dried, and stored in plastic vials. Samples were stored on dry ice for transport, and maintained frozen until analysis.

Analytical Methods

All biological tissue samples were freeze-dried, ground, and analyzed for Hg, stable carbon isotopes ($\delta^{13}C$), and stable nitrogen isotopes ($\delta^{15}N$).

Mercury in Periphyton and Invertebrates

Periphyton and aquatic invertebrates were analyzed for total Hg (THg) and methylmercury (MeHg) by the USGS Wisconsin Mercury Research Laboratory in Middleton,Wisconsin using U.S. Environmental Protection Agency (USEPA) method 1631 for THg and draft method 1630 for MeHg, as adapted for solid material (DeWild and others, 2002; U.S. Environmental Protection Agency, 2002; Bell and Scudder, 2005; DeWild and others, 2004; Olund and others, 2004). Samples were analyzed for THg using a 7:3 nitric and sulfuric acid digestion at 125°C, followed by oxidation with bromine monochloride (BrCl), and cold vapor atomic fluorescence spectroscopy (CVAFS) (Olson and Dewild, 1999). Samples were analyzed for MeHg by distillation, aqueous phase ethylation, and CVAFS. Method bias and precision were assessed using certified reference material (CRM) from the National Research Council (Canada) and sample duplicates (appendixes 6 and 8). For the THg and MeHg analyses, CRM and sample duplicate objectives were set at ±25 percent of theoretical values or reported at a 95-percent confidence interval (appendixes 5, 6, 9, and 10).

Mercury in Fish

Tissue from individual forage fish and top predator fish were freeze-dried and pulverized in preparation for analysis of Hg and stable carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) isotopes. Samples were analyzed for THg by the Texas A&M University Trace Element Research Laboratory (College Station, Texas) by combustion and atomic absorption, using a direct Hg analyzer (Milestone DMA-80) following USEPA Method 7473. Fish were analyzed for THg only, because other studies

have shown that approximately 95 percent of Hg in fish muscle tissue is MeHg (Huckabee and others, 1979; Bloom, 1992). This laboratory meets quality-assurance requirements of the U.S. Fish and Wildlife Service (U.S. Fish and Wildlife Service, 2007). Laboratory quality control included standard reference material (SRM) standards, duplicate samples, and spiked samples (appendixes 5, 7a, 7b, 10, and 12).

Stable Isotopes in Biological Tissues

Subsamples of the dried and ground tissue were sent to the USGS National Research Program Isotopic Tracers Laboratory in Menlo Park, California. All biota were analyzed for $\delta^{13}C$ and $\delta^{15}N$ by using a Carlo Erba 1500® elemental analyzer interfaced with a Micromass Optima® continuous-flow isotope ratio mass spectrometer (Fry and others, 1992; Kendall and others, 2001).

Age Determination of Forage and Top Predator Fish

All fish heads and otoliths were sent to the USGS Cooperative Wildlife Research Unit Laboratory at Clemson University in Clemson, S.C.; otoliths were extracted from the fish heads. Ages were determined using standard age determination techniques as described in Nielsen and Johnson (1983).

Quality Assurance/Quality Control

Analysis of triplicate composite invertebrate samples and individual fish specimens of each species per collection period served as field measures of variability and quality assurance/quality control (QA/QC) for Hg concentrations. In addition, laboratory QA/QC measures for Hg included analysis of CRM and SRM, spikes, and replicates. For CRM/SRM analyses (appendixes 5 and 6), median recoveries for SRMs using invertebrate analysis methods were 106.9 percent for THg (96.86 and 104.3 percent in methods NIST 2976 and IAEA-407, respectively) and 122.0 percent for MeHg (IAEA-407). For invertebrate Hg analyses 29 percent of the THg values fell within the 95 percent confidence interval, and 98 percent were within 25 percent of the SRM target values. All invertebrate MeHg values were higher than target values; 9 percent fell within the 95 percent confidence interval, and 45 percent were within 25 percent of the SRM target values (appendixes 7a and 7b). For fish analyses, the median CRM recovery was 100.0 percent for THg (101.9 and 98.64 percent in methods NRCC DOLT-2 and DORM-2, respectively). About 96 percent of the CRM values of the fish analyses were within 10 percent of the certified concentration (appendix 8).

The median difference (percent relative standard deviation of the concentrations, or RSD) in replicate invertebrate samples was 4.3 percent for THg and 7.5 percent for MeHg (appendixes 9, 11a and 11b). The median difference (relative percent difference between duplicate concentrations, or RPD) for THg in fish was 1.62 percent (appendixes 10 and 12) and was slightly higher for forage fish compared to predator fish. The RPD between duplicate fish samples exceeded 10 percent for three sample sets. For one of these sets (the highest RPD), this exceedence was due to a very low absolute concentration. Spike analyses were done for fish only, and the median spike recovery for THg was 98.6 percent. The median daily detection limit for invertebrates was 0.5 ng g^{-1} dry weight for THg and 0.5 ng g^{-1} dry weight for MeHg. The minimum detection limit for THg in fish was 6 ng g^{-1} dry weight. No samples contained concentrations of Hg less than the detection limit; however, a limited number of invertebrate samples could not be analyzed for THg or MeHg, or both, due to insufficient sample mass.

References

Begon, M., Townsend, C., and Harper, J., 2006, Ecology: From individuals to ecosystems (4th ed.): Oxford, Blackwell Publishing, 738 p.

Bell, A.H., and Scudder, B.C., 2005, Mercury accumulation in periphyton of eight river ecosystems: Journal of the American Water Resources Association, v. 43, no. 4, p. 957-968.

Bell, A.H., and Lutz, M.A., in press, Environmental settings of selected streams sampled during 2002–2006 as part of U.S. Geological Survey studies on bioaccumulation of mercury: U.S. Geological Survey Open-File Report 2007-1277.

Bell, A.H., and Scudder, B.C., 2005, Bioaccumulation of mercury in riverine periphyton: U.S. Geological Survey Open-File Report 2004-1446, 8 p.

Bloom, N.S., 1992, On the chemical form of mercury in edible fish and marine invertebrate tissue: Canadian Journal of Fisheries and Aquatic Sciences, v. 49, p. 1010-1017.

Brigham, M.E., Duris, J.W., Button, D.T., Chasar, L.C., and Wentz, D.A., 2008, Total mercury, methylmercury, and ancillary water-quality and streamflow data for selected streams in Oregon, Wisconsin, and Florida, 2002–06: U.S. Geological Survey Data Series 341, 11 p., accessed September 22, 2008, at http://pubs.er.usgs.gov/usgspubs/ds/ds341

Cleckner, L.B., Gilmore, C.C., Hurley, J.P., and Krabbenhoft, D.P., 1999, Mercury methylation in periphyton of the Florida Everglades: Limnology and Oceanography, v. 44, no. 7, p. 1815-1825.

DeWild, J.F., Olson, M.L., and Olund, S.D., 2002, Determination of methylmercury by aqueous phase ethylation, followed by gas chromatographic separation with cold vapor atomic fluorescence detection: U.S. Geological Survey Open-File Report 01-445, 14 p.

DeWild, J.F., Olund, S.D., Olson, M.L., Tate, M.T., 2004, Methods for the preparation and analysis of solids and suspended solids for methylmercury: U.S. Geological Survey Techniques and Methods 5-A7, 21 p.

Fry, B., Brand, W., Mersch F.J., Tholke, K., and Garrit, R., 1992, Automated analysis systems for coupled $\delta^{13}C$ and $\delta^{15}N$ measurements: Analytical Chemistry, v. 64, no. 3, p. 288-291.

Huckabee, J.W., Elwood, J.W., and Hildebrand, S.G., 1979, Accumulation of mercury in freshwater biota, *in* Nriagu, J.O., ed., Biogeochemistry of mercury in the environment: New York, Elsevier/North-Holland Biomedical Press, p. 277-302.

International Union of Pure and Applied Chemistry, 1993, Glossary for Chemists of Terms used in Toxicology: Pure and Applied Chemistry, v. 65, no. 9, p. 2003-2122.

Kendall, C., and McDonnell, J., 1998, Isotope tracers in catchment hydrology: Elsevier Science, Amsterdam, The Netherlands, 839 p.

Kendall, C., Silvia, S.R., and Kell, V.J., 2001, Carbon and nitrogen isotopic compositions of particulate organic matter in four large river systems across the United States: Hydrological Processes, v. 15, no. 7, p. 1301-1346.

Lajtha, K., and Michener, R., eds., 1994, Stable isotopes in ecology and environmental science: Oxford, Blackwell Scientific Publications, 336 p.

Lewis, M.E., and Brigham, M.E., 2004, Low level mercury: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5, section 5.6.4.B, accessed at http://water.usgs.gov/owq/FieldManual/chapter5/pdf/5.6.4.B_v1.0.pdf

Marvin-DiPasquale, M.C., Lutz, M.A., Krabbenhoft, D.P., Aiken, G.R., Orem, W.H., Hall, B.D., DeWild, J.F., Agee, J.L., and Brigham, M.E., 2008, Total mercury, methylmercury, methylmercury production potential, and ancillary streambed sediment and pore water data for selected streams in Oregon, Wisconsin, and Florida, 2003-04: U.S. Geological Survey Data Series 375, 24 p.

Moulton, S.R., II, Kennen, J.G., Goldstein, R.M., and Hambrook, J.A., 2002, Revised protocols for sampling algal, invertebrate, and fish communities as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 02-150, 72 p.

Nielsen, L.A., and Johnson, D.L., 1983, Age determination, *in* Jearld, A., Jr., ed., Fisheries techniques: Bethesda, Md., The American Fisheries Society, p. 301-324.

Olson, M.L., and DeWild, J.F., 1999, Low-level techniques for the collection and species-specific analysis of low levels of mercury in water, sediment, and biota, *in* Morganwalp, D.W., and Buxton, H.T., eds. 1999, U.S. Geological Survey Toxic Substances Hydrology Program–Proceedings of the technical meeting, Charleston, South Carolina, March 8-12, 1999–Volume 2–Contamination of Hydrologic Systems and Related Ecosystems: U.S. Geological Survey Water-Resources Investigations Report 99-401b, p. 191-199.

Olund, S.D., DeWild, J.F., Olson, M.L., and Tate, M.T., 2004, Methods for the preparation and analysis of solids and suspended solids for total mercury: U.S. Geological Survey Techniques and Methods 5-A8, 23 p.

Scudder, B.C., Chasar, L.C., DeWeese, L.R., Brigham, M.E., Wentz, D.A., and Brumbaugh, W.G., in press, Procedures for collecting and processing aquatic invertebrates and fish for analysis of mercury as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 2008-1208.

Thorp, J.H., and Covich, A.P., eds., 2001, Ecology and classification of North American freshwater invertebrates, San Diego, Academic Publishing, 911 p.

U.S. Environmental Protection Agency, 1996, Method 1669: Sampling ambient water for trace metals at EPA water quality criteria levels: U.S. Environmental Protection Agency, Office of Water, EPA-821-R-96-011, 37 p.

U.S. Environmental Protection Agency, 1998, Method 7473–Mercury in solids and solutions by thermal decomposition, amalgamation, and atomic absorption spectrophotometry: U.S. Environmental Protection Agency, 15 p., accessed January 27, 2008 at http://www.epa.gov/sw-846/pdfs/7473.pdf

U.S. Environmental Protection Agency, 2002, Mercury in water by oxidation, purge and trap, and cold vapor atomic fluorescence, Method 1631, Revision E: U.S. Environmental Protection Agency, Office of Water, EPA-821-R-02-019, 46 p.

U.S. Fish and Wildlife Service, 2007, Analytical Control Facility– Quality control: U.S. Fish and Wildlife Service, Division of Environmental Quality, accessed January 27, 2008, at http://www.fws.gov/chemistry/acf_qaqc.html

Wehr, J.D., and Sheath, R.G., 2003, Freshwater algae of North America: San Diego, Academic Press, 918 p.

Wetzel, R.G., 2001, Limnology, 3d ed., San Diego, Academic Press, 1,006 p.

Appendix Data

These data files are included as part of U.S. Geological Survey (USGS) Data Series 349 and are available for download at http://pubs.usgs.gov/ds/349/. The data were collected during a 2002–04 study of mercury bioaccumulation in eight streams in diverse environmental settings. 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**). Those analytes with no parameter code listed are not entered in NWIS.

Fourteen data tables are included in this data series:

Appendix 1. Taxa and associated ecological information.
List of taxa collected in each of the three study areas (Oregon, Wisconsin, Florida) and associated ecological information. Organisms were identified to the lowest possible taxonomic level in the field.

Appendix 2. Periphyton.
Data for periphyton: biomass expressed as ash-free dry mass (AFDM); Chlorophyll a; total mercury (THg) and methylmercury (MeHg) expressed as dry weight concentrations; stable carbon and nitrogen isotope ratios ($\delta^{13}C$, $\delta^{15}N$); and elemental composition (C:N).

Appendix 3. Invertebrates.
Data for invertebrates: total mercury (THg) and methylmercury (MeHg) expressed as dry weight concentrations; stable carbon and nitrogen isotope ratios ($\delta^{13}C$, $\delta^{15}N$); and elemental composition (C:N).

Appendix 4. Fish.
Data for forage and top predator fish: total mercury (THg) expressed as dry weight concentrations; stable carbon and nitrogen isotope ratios ($\delta^{13}C$, $\delta^{15}N$); and elemental composition (C:N). Moisture content data are based on laboratory wet weights (subsequent to freezing and storage).

Appendix 5. Quality Assurance/Quality Control Standard Reference Material Summary–Wisconsin Mercury Research Laboratory.
Summary of results of Standard Reference Material (SRM) analyses for THg and MeHg in invertebrates.

Appendix 6. Quality Assurance/Quality Control Certified Reference Material Summary–Trace Element Research Laboratory.
Summary of results of Certified Reference Material (CRM) analyses for THg in fish tissue.

Appendix 7a. Quality Assurance/Quality Control Standard Reference Material–Invertebrates.
Raw results of SRM analyses for THg in invertebrates.

Appendix 7b. Quality Assurance/Quality Control Standard Reference Material–Invertebrates.
Raw results of SRM analyses for MeHg in invertebrates.

Appendix 8. Quality Assurance/Quality Control Certified Reference Material–Fish.
Raw results of CRM analyses for THg in fish tissue.

Appendix 9. Quality Assurance/Quality Control Standard Reference Material Summary–Invertebrates.
Summary of results of replicate analyses for THg and MeHg in invertebrate tissue.

Appendix 10. Quality Assurance/Quality Control Certified Reference Material Summary–Fish.
Summary of results of duplicate analyses and spikes for THg in fish tissue.

Appendix 11a. Quality Assurance/Quality Control Standard Reference Material–Invertebrates.
Raw results of replicate analyses for THg in invertebrate tissue.

Appendix 11b. Quality Assurance/Quality Control Standard Reference Material–Invertebrates.
Raw results of replicate analyses for MeHg in invertebrate tissue.

Appendix 12. Quality Assurance/Quality Control Certified Reference Material–Fish.
Raw results of duplicate and spike analyses for THg in fish tissue.

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Glossary

Benthic Associated with (living on or near) the bottom of an aquatic habitat (Thorp and Covich, 2001; Wehr and Sheath, 2003).

Bioaccumulation Gradual increase in the amount of a substance in the tissue(s) of an organism that occurs when the rate of intake (through respiration, ingestion, dermal contact, and other mechanisms) exceeds removal. (International Union of Pure and Applied Chemistry, 1993)

Biomagnification The ecosystem-level process that results in higher concentrations of a substance in organisms at progressively higher trophic levels; that is, the process leading to a higher concentration of a substance in a consumer than in its food (International Union of Pure and Applied Chemistry, 1993).

Community Multispecies assemblages in a given ecosystem; groups of species that interact in a common area through predation, competition, resource sharing, resource partitioning (Begon and others, 2006).

Confidence limits (95%) Upper and lower limits of the 95% confidence interval. This is an estimate of the interval containing the true mean value with 95% degree of certainty. It is defined as

$$\bar{X} \pm t_{(\alpha/2, N-1)} s / \sqrt{N} ,$$

where $\bar{X}$ is the sample mean, t is critical value of the t-distribution, α is the significance level (for 95% CI, $\alpha = 0.05$), N is the sample number, and S is the standard deviation.

Depositional-targeted habitat (DTH) A habitat where fine sediment, such as sand and silt, is deposited (Moulton and others, 2002).

Ecosystem The collective term describing biota (all biological organisms) and their associated abiotic environment.

Elutriate To separate lighter particles (in the context of this report, algal cells) from heavier particles (such as sediment and debris) by progressive washing, settling, and decanting (Moulton and others, 2002; Bell and Scudder, 2007).

Forage fish Primary (herbivores) and secondary consumers (omnivores or carnivores) that are generally smaller fecund species that are forage (prey) for larger predaceous fish.

Macroinvertebrate Invertebrates (organisms without a spinal column) larger than microinvertebrates; that is, organisms retained on a ≥ 2 mm sieve (Thorp and Covich, 2001).

Otoliths Paired calcified structures lying adjacent to the brain in bony fishes. Otoliths grow continuously, and the growth annuli (annual growth rings) are used to estimate growth rate and age. Otoliths are commonly referred to as “ear stones” or “ear bones.”

Periphyton Commonly used to indicate algal cells; however, periphyton collectively refers to fungi, bacteria, algae, and detritus attached to any substrate in an aquatic system (Wehr and Sheath, 2003).

Relative standard deviation The relative standard deviation (RSD) (also referred to as the coefficient of variation) is a measure of precision; expressed as a percentage, it is defined as

$$\%RSD = [(S \times 100) / \bar{X}] ,$$

where S is the sample standard deviation and is the sample mean.

Relative percent difference The relative percent difference (RPD) is measure of precision and is defined by

$$RPD = [X_1 - X_2) / ((X_1 + X_2) / 2] \times 100 ,$$

where X_1, X_2 are duplicate results for the same sample.

Richest-targeted habitat (RTH) Usually riffles or woody snags; this is the habitat type where the taxonomically richest (greatest number of species) algal or invertebrate community is located in a given stream (Moulton and others, 2002).

Stable isotopes Isotopes are atoms of an element with different numbers of neutrons (and, therefore, different masses). Stable isotopes do not decay to other isotopes; variations in the ratios of stable isotopes of a given element are the result of mass-dependent fractionation driven by the physical and chemical properties of the isotopes. Stable isotope compositions of carbon and nitrogen are expressed in del (δ) notation:

$$\delta\ (‰) = [R_x / (R_s - 1)]\ ,$$

where R is the ratio of heavy isotope to light isotope; R_x and R_s are the stable isotope ratios in a sample and standard, respectively. Natural abundance (naturally occurring) isotopic signatures are used to describe physiological processes at an organism level, to trace trophic dynamics at a community level, and to evaluate biogeochemical cycling at the ecosystem level (Lajtha and Michener, 1994; Kendall and McDonnell, 1998).

Top predator fish The predaceous fish species occupying the highest trophic level in a given community or ecosystem; top predator fish may be piscivorous, but are often opportunistic, ingesting a wide range of prey types as prey availability changes (Wetzel, 2001).

Trophic complexity The trophic complexity of a community is described by the number of trophic levels (functionally similar groups of organisms that compete for food resources) in addition to the number of different pathways of energy transfer among trophic levels (Wetzel, 2001).

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