

Environmental Policy & Regulation

Bioavailability of Mercury in Power Plant Wastewater and Ambient River Samples: Evidence that the Regulation of Total Mercury Is Not Appropriate

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ABSTRACT

Mercury (Hg) is a neurotoxin that can cause debilitating effects to human and environmental receptors under high exposure conditions. For industrial and municipal point sources that discharge Hg, wastewater limitations on total Hg (THg) concentrations or loads are typical. While this regulatory practice provides simplicity for regulated industry and water resource agencies (i.e., for analytical detection and reporting purposes), it ignores the important considerations of speciation and bioavailability. In this study, water samples were collected from multiple power plant wastewater, simulated mixing zone, and ambient river locations ($N = 10$ to 20) and were analyzed for bioavailable Hg forms (methylmercury and acid-labile Hg, or BHg), THg, and dissolved Hg. The median concentration of THg in wastewater, mixing zone, and ambient river samples was 7.1, 5.3, and 2.3 ng/L, respectively. The percentages of THg as BHg (median values) were 18.7%, 29.3%, and 8.5% for wastewater, mixing zone, and ambient river samples. The percentages of methylmercury (MeHg) as THg were not statistically different between paired ambient and mixing zone samples ($P > 0.05$); this result indicates that wastewater did not increase the MeHg fraction when mixed with ambient water. Multiple regression analysis indicated that variation in THg for combined wastewater and mixing zone samples could be adequately explained by pooled water quality parameters (total suspended solids, total dissolved solids, sulfate, total organic carbon, pH, specific conductivity; $r^2 = 0.51$; $P < 0.05$); however, no significant regression relationships were apparent for the percentage of BHg. These results, at least for the wastewater samples evaluated, indicate that regulating THg is likely overly conservative, and mechanisms to regulate the bioavailable forms of Hg are needed. If Hg fish tissue monitoring data indicate that concentrations are less than consumption thresholds, metal translator methodologies or bioavailability-based criterion techniques (as currently used for non-Hg trace elements) should be allowed for Hg. *Integr Environ Assess Manag* 2019;15:142–147. © 2018 SETAC

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INTRODUCTION

Anthropogenic releases of mercury (Hg) to the aquatic environment are typically highly regulated because of potential food web bioaccumulation risks. While adverse effects on human health have been documented during accidental high exposure events, several researchers have advocated that potential adverse health effects due to methylmercury (MeHg) exposure should be balanced by health benefits from fish consumption (Myers and Davidson 2007; Rheinberger and Hammitt 2012). In 2001 the US Environmental Protection Agency (USEPA) issued a fish tissue-based water quality criterion for MeHg meant to protect human health from potential adverse effects via fish consumption (0.3 mg/kg wet wt.; USEPA 2001). A relatively small number of states and tribes have adopted this criterion; rather, most states continue to

regulate the discharge of total Hg (THg) with a derived human health-based water quality criterion of 12 ng/L, which itself is based on a laboratory MeHg bioaccumulation study using fathead minnows (Olson et al. 1975). This recommended human health-based criterion is an estimated water threshold that would not exceed the (then) Food and Drug Administration tissue action level of 1.0 mg/kg (USEPA 1985).

Irrespective of the water quality criterion that water resource agencies implement to regulate Hg from industrial and municipal wastewaters, there is little (if any) consideration of what forms of Hg are, or are potentially, bioavailable. In 1993 the USEPA issued a policy memorandum (“Office of Water Policy and Technical Guidance on Interpretation and Implementation of Aquatic Life Metals Criteria”) that specified the regulation of dissolved metals as the preferred approach for preventing adverse effects to aquatic life. A guidance manual to implement this policy was subsequently issued (USEPA 1996a).

More refined speciation techniques have enabled increased reliability of toxicity predictions. Merrington et al. (2016) provided an overview of procedures to regulate the

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bioavailable portion of trace elements. Allen and Hansen (1996) reported the importance of copper speciation to predicting outcomes of toxicity tests. Moreover, some updated and revised water quality criteria are based on empirical bioavailability tools such as the biotic ligand model (e.g., USEPA 2007, 2017). While many states have adopted bioavailability-based metals criteria for the protection of aquatic life (most often through operationally defined dissolved metal ratios or geochemical modeling), bioavailability-based approaches for regulating Hg are lacking.

Because of advances in analytical capabilities, speciation analysis of Hg in aqueous samples is both accurate and precise when quality control criteria are attained (Leermakers et al. 2005; Leopold et al. 2010). The bioavailable fraction of total Hg (BHg) in aqueous samples has typically been described as the sum of acid-labile Hg (Hg_{AL} ; sometimes used synonymously with “inorganic reactive Hg”) and methylated Hg (Gill and Bruland 1990; Morel et al. 1998; WERF 2009). Hg_{AL} is an operationally defined fraction of THg that is readily chemically reduced with $SnCl_2$ at low pH and includes weak organic associations, inorganic complexes, and labile particulate Hg (Mason and Fitzgerald 1990). Marvin-DiPasquale et al. (2008) indicate that these forms are associated with simple anions (e.g., $HgSO_4$, $HgCl_2$) in sediment pore water and/or inorganic Hg (principally Hg^{+2}) that is weakly adsorbed to particle surfaces and are likely available for methylation. Under certain environmental conditions (any combination of low dissolved oxygen, low pH, elevated sulfate), Hg_{AL} can be reduced to MeHg.

Mercury redox chemistry, however, can be affected by many variables, and the assumption that measured “bioavailable” forms and “nonbioavailable” forms are environmentally stable may be erroneous in some settings. For example, Zheng et al. (2012) reported that reduced natural organic matter (NOM) can mediate both reductive and oxidative reactions of Hg, particularly in anoxic environments where microbial reduction of NOM and Hg methylation are both active. Certainly, a portion of THg that is operationally defined as “nonbioavailable” may, in some cases, be subsequently converted to “bioavailable” Hg in situ. Likewise, demethylation processes could promote the opposite.

In the present study, water samples were collected from coal-fired power plant waste streams that were influenced (partially or entirely) by flue gas desulfurization (FGD) wastewater. At the same time, ambient (upstream) Ohio River samples and simulated mixing zone (mix of ambient and waste stream) samples were collected. Four species of Hg were analyzed with the goal of discriminating between bioavailable and nonbioavailable forms. Samples for the analysis of routine water quality variables were also collected and analyzed.

MATERIALS AND METHODS

Study site and field sampling

Water samples were collected at 4 coal-fired power plants in the upper Ohio River, located between river kilometers 122

and 415 (Figure 1). All the power plants burn bituminous Appalachian coal and are equipped with electrostatic precipitators (particulate removal), selective catalytic reduction (NO_x removal) units, and wet FGD (SO_2 removal) units. Table 1 indicates the generating capacity of each facility and the waste stream or streams sampled. The wastewater samples collected at Cardinal Plant, unlike those collected at the other facilities, represented untreated FGD wastewater. Thus, relatively higher concentrations of THg and the various Hg species were expected in these samples (EPRI 2010).

At Gavin, Mitchell, and Cardinal Plants, ambient Ohio River samples were collected just upstream of the wastewater's confluence with the Ohio River. Simulated mixing zone samples were a 1:1 mixture of wastewater and upstream ambient water. Samples were collected between July and December 2016 ($N = 10$ to 20 for each sample type).

Surface grab samples collected for Hg analysis followed “clean sampling” procedures (USEPA 1996b). Samples were also collected for the analysis of routine parameters (pH, specific conductivity, sulfate, sulfide, total organic C, total dissolved solids, total suspended solids).

Laboratory methods

All samples used for the determination of THg, MeHg, and Hg_{AL} were collected in prepreserved sampling containers. Samples for the analysis of THg followed agency protocol (USEPA 2002). The samples were preserved with bromine monochloride and then analyzed with stannous chloride ($SnCl_2$) reduction, dual gold amalgamation, and cold vapor atomic fluorescence spectroscopy. Samples for dissolved Hg (DHg) were filtered upon receipt at the laboratory (<48 hours after collection) and analyzed for soluble Hg. Thermo Scientific Nalgene filters were used (0.45 μm , with a polyethersulfone membrane). Samples for MeHg analysis (unfiltered) were preserved with 0.4% HCl (v/v) and distilled via USEPA Method 1630 (USEPA 1998). Samples for the analysis of Hg_{AL} (also unfiltered) were preserved with HCl. Mercuric ions were reduced to Hg^0 with $SnCl_2$ and then purged onto gold-sand traps. Hg^0 was thermally desorbed and fluorescence was measured, which was proportional to the total Hg collected. The analysis of routine parameters followed Standard Methods or USEPA protocols.

For each sample event and location, the proportion of bioavailable Hg (BHg) was calculated as

$$BHg = [MeHg + Hg_{AL}/THg] \times 100.$$

Because DHg is distributed among several chemical forms (elemental, oxidized inorganic, organic; Gill and Bruland 1990; Morel et al. 1998), this species (while accounted for in a THg analysis) was considered neither bioavailable nor nonbioavailable for the purpose of this study.

The analytical accuracy and precision of THg, MeHg, and Hg_{AL} were evaluated with laboratory fortified blanks, method blanks, duplicates, matrix spikes, and matrix spike duplicates. A certified reference material (SRM NIST 1641d) was analyzed for THg.

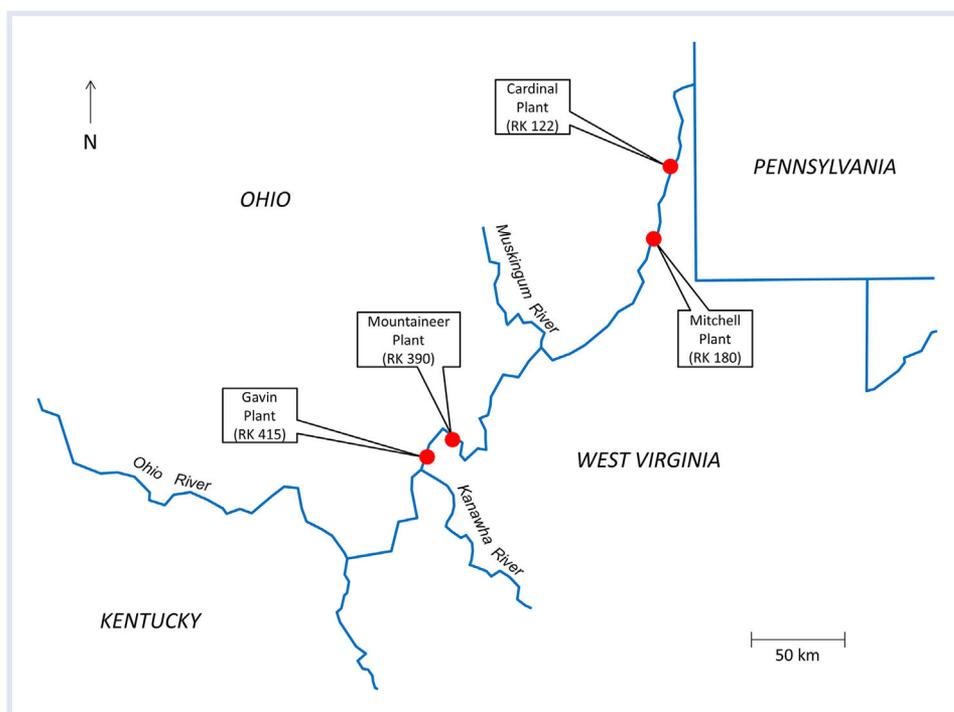


Figure 1. Location of coal-fired power plants where wastewater, mixing zone, and ambient Ohio River water samples were collected during 2016. River kilometer distances from Pittsburgh, Pennsylvania indicated in parentheses.

Data analysis

Summary statistics are provided for all parameters. To evaluate potential differences in Hg species and some routine water quality parameters among the 3 categories of sampling locations, the Kruskal–Wallis test was used. When test results were significant ($P < 0.05$), differences among sampling locations were evaluated with the Mann–Whitney U test. To assess the influence of the various wastewaters on Hg bioavailability in the Ohio River, paired ambient and simulated mixing zone samples were compared with the Wilcoxon signed-rank test. Comparisons were made with percent THg as BHg and percent THg as MeHg. Similar comparisons were conducted with results for undiluted wastewater and simulated mixing zone samples. Though some BHg and MeHg fractions were identified as statistical

outliers, these values were not excluded in resulting box plots. Stepwise multiple regression was used to evaluate the influence of routine water quality variables on measured levels of THg and BHg.

RESULTS AND DISCUSSION

Results of laboratory quality assurance and quality control analyses indicated that 102 of 104 Hg measurements (including THg, MeHg, and Hg_{AL}) attained the acceptable analytical criteria, indicating favorable precision and accuracy. Two fortified blank analyses exceeded the acceptable recovery limit.

Summary statistics for routine water quality parameters and Hg speciation results are provided in Tables 2 and 3, respectively. Concentrations of total dissolved solids, sulfate,

Table 1. Power plant locations, generating capacity, and wastewaters evaluated

Facility	Generating capacity (MW)	Wastewaters evaluated
Cardinal (OH)	1800	Untreated FGD wastewater
Mitchell (WV)	1600	Treated FGD wastewater
		FGD solid waste leachate
		Bottom ash transport water
Mountaineer (WV)	1300	Treated FGD wastewater
		FGD and fly ash solid waste leachate
James M Gavin (OH)	2600	FGD solid waste leachate
		Bottom ash transport water

Table 2. Median concentrations (\pm SD) of routine water quality variables for samples collected at wastewater, mixing zone, and ambient Ohio River locations near 4 Ohio River power plant sites, 2016^a

Sampling location	N	TSS ^b (mg/L)	TDS ^c (mg/L)	SO ₄ (mg/L)	SC (μ hmos/cm 25° C)	Sulfide (mg/L)	TOC (mg/L)	pH (s.u.)
Wastewater	18	6.4 ^b (\pm 17.4)	1840 ^c (\pm 3182)	883 ^c (\pm 1124)	3830 ^c (\pm 4511)	<0.4	3.70 ^b (\pm 3.1)	7.79 ^b
Mixing zone	16	10.4 ^b (\pm 23.3)	1,004 ^c (\pm 1854)	448 ^c (\pm 589)	1530 ^d (\pm 2688)	<0.4	3.27 ^b (\pm 2.98)	7.89 ^b
Ambient	10	11.1 ^b (\pm 40.6)	215 ^b (\pm 27)	74.8 ^b (\pm 24.6)	387 ^b (\pm 36)	<0.4	2.69 ^b (\pm 0.83)	7.89 ^b

^aFor each column, values having the same superscript letter are not significantly different ($P > 0.05$).

^bTotal suspended solids.

^cTotal dissolved solids.

SC = specific conductivity; s.u. = standard unit; TOC = total organic C.

and specific conductivity were significantly higher in both wastewater and mixing zone samples relative to ambient samples ($P < 0.0001$). This result is not surprising because treated FGD wastewater and FGD solid waste leachate are typically enriched with chloride, potassium, and sulfate. Concentrations of total suspended solids, total organic carbon, and pH levels were not significantly different among the 3 sample types ($P > 0.05$). For most samples at all locations, the concentration of sulfide was less than the detection limit (<0.4 mg/L).

Median concentrations of THg, DHg, and MeHg were highest in wastewater samples (Table 3), although the median concentration of THg in these samples (7.1 ng/L) was less than the most stringent water quality criterion (the human health–based criterion) for the Ohio River (12 ng/L). Concentrations of THg in both wastewater and mixing zone samples were significantly higher than those in ambient samples ($P < 0.05$), but the distribution of concentrations between wastewater and mixing zone samples was not significantly different. The median value of Hg_{AL} was highest in mixing zone samples (1.57 ng/L). The percentage of THg as DHg (median values) ranged between 13.6% (mixing zone samples) and 20.0% (ambient samples).

The median percent of BHg as THg was highest in simulated mixing zone samples, intermediate in wastewater

samples, and lowest in ambient samples (Figure 2). Median proportions of BHg as total Hg were all less than 30%. A comparison of paired ambient and mixing zone samples indicated significantly higher BHg fractions in mixing zone samples ($P < 0.05$). No differences in the BHg fractions were found between paired ambient samples and wastewater samples and between wastewater samples and mixing zone samples ($P > 0.05$).

The median percent of THg as MeHg was highest in ambient samples, intermediate in mixing zone samples, and lowest in wastewater samples. These percentages were all less than 2%. There were no significant differences in the percentage of THg as MeHg for all sample type comparisons ($P > 0.05$).

Using data for pooled wastewater and simulated mixing zone samples, multiple regression analysis was performed for THg concentrations. The best fit regression model included most routine water quality variables combined (total suspended solids, total dissolved solids, sulfate, total organic carbon, pH, and specific conductivity; $r = 0.51$; $P < 0.05$). No significant regression models were found when using either Hg_{AL} or MeHg concentrations as dependent variables.

While simulated mixing zone samples had the highest median percentage of BHg (29.3%), these results are somewhat misleading. Mixing zone samples were comprised

Table 3. Median concentrations (\pm SD) of Hg species in wastewater, mixing zone, and Ohio River ambient samples at 4 Ohio River power plant locations, 2016^a

Sampling location	N	THg (ng/L)	DHg (ng/L)	MeHg (ng/L)	Hg _{AL} (ng/L)
Wastewater	16–20	7.1 (\pm 412)	1.09 (\pm 2.11)	0.088 (\pm 0.323)	0.84 (\pm 6.02)
Mixing zone	15	5.25 (\pm 73.9)	0.72 (\pm 0.66)	0.078 (\pm 0.201)	1.57 (\pm 3.46)
Ambient	10	2.3 (\pm 7.3)	0.45 (\pm 0.17)	0.034 (\pm 0.102)	0.08 (\pm 0.69)

^aFor total Hg (THg) measurements, values with the same letter are not significantly different ($P > 0.05$).

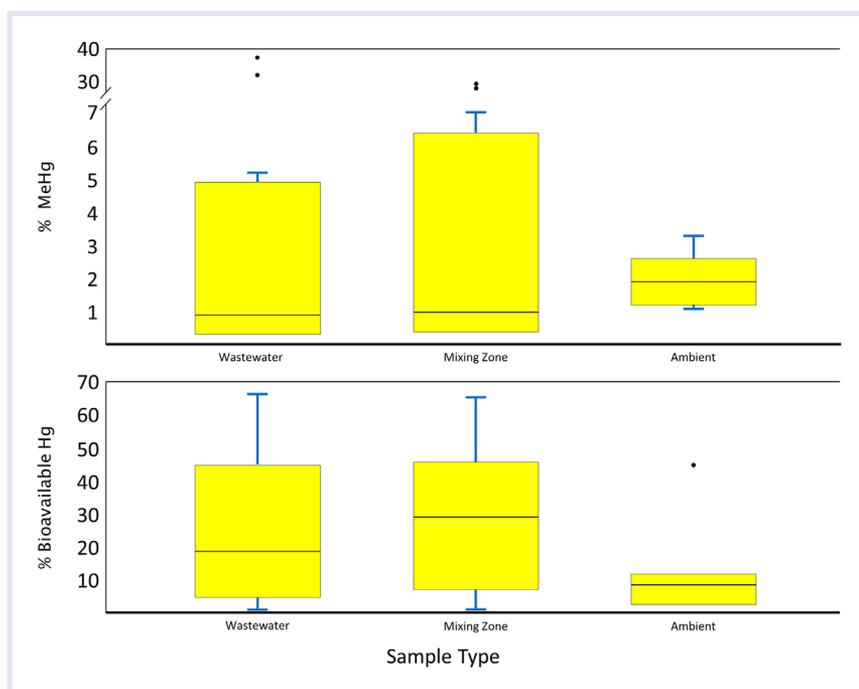


Figure 2. Box plots of % methylmercury (MeHg) and % bioavailable Hg (relative to total Hg) for power plant wastewater, mixing zone, and ambient Ohio River samples, 2016. Median, interquartile ranges, whiskers, and statistical outliers (solid circles) are provided.

of a 1:1 mixture of ambient and wastewater samples, an in situ ratio that would not be allowed for point-source permitting purposes (at average Ohio River flow rates, the proportion of wastewater flows would be no greater than 10%). Thus, while the mixture ratio was regarded as overly conservative and perhaps “worst case,” it was useful for illustrative purposes.

Previous reports of the percentage of BHg relative to THg are few. Gill and Bruland (1990) reported Hg speciation results for lakes (and rivers) in California and Lake Erie. For samples collected from the Sacramento River and the Columbia River, the percentage BHg (sum of reactive inorganic Hg and organo-Hg) as THg ranged between 18% and 20%. In the Lake Erie samples, the fraction of BHg was 50%.

In this study the median concentration of MeHg in ambient samples (0.034 ng/L) was consistent with previous measurements in the Ohio River as reported by ORSANCO (2013); in that study the median concentration of MeHg at 4 Ohio River power plant intake locations was 0.041 ng/L. The low median percentage of THg as MeHg in samples from the current study (<2%) is similar to findings reported for samples collected at the same 4 Ohio River power plant intake locations and at various ambient locations (ORSANCO 2013, 2017). The findings from this study support previous inferences that the methylation potential of the Ohio River is low (EPRI 2013; Reash et al. 2015).

The median concentration of total organic carbon (TOC) in ambient site samples in this study (2.69 mg/L) is similar to historical measurements of TOC at ambient Ohio River locations. ORSANCO (2016) reported that median concentrations of TOC at upper Ohio River sampling locations between 2010 and 2014 varied between 2 and 3 mg/L.

This study had limitations that deserve mention. The overall sample size of Hg speciation analyses was relatively low. Thus, these limited sample sizes did not capture long-term temporal and/or spatial trends of Hg in wastewater and ambient locations. Other types of wastewater (e.g., petrochemical, mining, municipal) were not evaluated. Finally, samples were only collected in the upper one-third of the Ohio River. Nonetheless, this study provides empirical evidence that only a portion of THg in the Ohio River, and in power plant wastewaters, is of concern for ecological and human health risk considerations. Subsequent investigations encompassing larger sample sizes and expanded longitudinal coverage would be useful in evaluating the relevance of results for this study.

Previous researchers have argued that the regulation of THg in freshwater systems may not result in desired reductions in Hg methylation and bioaccumulation and in potential sediment toxicity (Mason and Lawrence 1999; Bigham et al. 2017). Kelly et al. (1995) observed that, in some Ontario lakes, THg levels in water were poor predictors of MeHg levels in water. Policy priorities and remediation costs, thus, should focus on those ecosystems where the methylation potential of Hg is elevated or where measured levels of Hg in biological receptors exceed risk thresholds. The regulation of THg alone may not result in the desired environmental benefit of reduced (and safe) fish tissue Hg levels.

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Data Accessibility—The data used for this study are available as a supplemental spreadsheet.

SUPPLEMENTAL DATA

Table S1 Mercury bioavailability study.

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