

Evaluation of Water Quality Translators for Mercury

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ABSTRACT

This report discusses the technical issues and constraints associated with translation of a mercury fish tissue concentration into a water quality criterion, in the use and implementation of the Environmental Protection Agency's fish-tissue-based criterion for methylmercury (0.3 mg methylmercury/Kg wet weight fish tissue). The report focuses on available analytical methods for evaluating mercury in fish and water; proposed methods for translating a fish tissue concentration for mercury into a concentration in water; and implementation of the mercury criterion in the development of Total Maximum Daily Loads (TMDLs) and water quality-based effluent limits (WQBELs). The approaches to criteria translation are, in order of preference: (1) derive site-specific bioaccumulation factors (BAFs), (2) use a bioaccumulation model, or (3) use EPA's national default translators. The collection of site-specific data allows for the most accurate assessment of bioaccumulation; however, validation of methylmercury analytical techniques is necessary to increase the certainty of results. Models have the potential to account for environmental factors contributing to data variability, but at present the available models are limited to reservoirs and lakes in a few geographic regions. Improvements in national default translators do not decrease the importance of site-specific translators. National default values are likely to be inaccurate on a site-specific basis, given the very high degree of variability observed in mercury bioaccumulation rates. Research is needed to improve the national default translators currently proposed by EPA, and additional data would increase the effectiveness of the translator calculation methods by reducing variability and minimizing the uncertainty of the resulting default values. Given the many uncertainties associated with mercury translators, their use should be limited to cases where site-specific fish tissue data reveal the tissue-based water quality criterion has been exceeded, and point sources make up a significant contribution of the total mercury loading to the water body.

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LEGEND

AVS	acid volatile sulfide
BAF	bioaccumulation factor
CMC	Criteria Maximum Concentration
CWA	Clean Water Act
DOC	dissolved organic carbon
EPA	U.S. Environmental Protection Agency
ELG	effluent limitation guideline
FAV	Final Acute Value
FCV	Final Chronic Value
f_d	dissolved methylmercury as fraction of total mercury in water column
K_D	partition coefficient, dissolved methylmercury to particulate mercury
MS/MSD	matrix spikes and matrix spike duplicates
NAWQA	national ambient water quality assessment
NPDES	national pollutant discharge elimination system
NIST	National Institute of Standards and Technology
OPR	ongoing precision and recovery
PAH	polycyclic aromatic hydrocarbon
PBT	persistent bioaccumulative toxic
POTW	publicly owned treatment works
QA	quality assurance
QC	quality control
RPA	reasonable potential analysis
SOP	standard operating procedure
TMDL	total maximum daily load
TSS	total suspended solids
WLA	wasteload allocation
WQBEL	water quality based effluent limit
WQS	water quality standard

Evaluation of Water Quality Translators for Mercury

Executive Summary

This report was prepared for the purpose of presenting an overview and discussion of the use of the ambient water quality criterion for methylmercury developed by the U.S. Environmental Protection Agency (EPA) in 2001 (66 FR 1344). Unlike all other previous water quality criteria for the protection of human health, the criterion for methylmercury was issued as a fish tissue concentration (i.e., 0.3 mg methylmercury/Kg wet weight fish tissue), because the fish consumption pathway is the main route of exposure to mercury in the environment. As a result, enforcement of water quality standards based upon this criterion requires either fish tissue sampling or the conversion of the criterion to a concentration in water, especially for the development of National Pollutant Discharge Elimination System (NPDES) permits.

Because many permitted dischargers will be faced with meeting the methylmercury criterion, this report focuses on (1) available analytical methods for evaluating mercury in fish and water; (2) proposed methods for translating a fish tissue concentration for mercury into a concentration in water; and (3) implementation of the mercury criterion in the development of Total Maximum Daily Loads (TMDLs) and water quality-based effluent limits (WQBELs). EPA (2002a, 2004a) draft *Guidance for Implementing the January 2001 Methylmercury Water Quality Criterion* addresses these issues and is discussed where applicable.

Analytical Methods

Analytical methods to quantify mercury in environmental samples continue to be refined. EPA Method 1631 is currently used to measure total mercury in water samples, while EPA Method 747B/SW-846 can be used to analyze total mercury in solid or semi-solid samples. The basic method for quantifying methylmercury is EPA Method 1630. Modifications to this method are described in the UW-Madison Standard Operating Procedure (SOP) and the United States Geological Service (USGS) Method. The selection of a method to test for either total mercury or methylmercury should be based on several factors, including method detection limit, validity of the method, and quality control procedures. All of these factors are important because they determine the quality and usability of the generated data.

Mercury Translation

The translation from methylmercury in fish to total mercury in water can be viewed as a single step (i.e., the direct method) or a multi-step process. In the direct method, total mercury in fish is directly translated to total mercury in water. In contrast, in the multi-step approach methylmercury in fish is converted to dissolved methylmercury in water and then to total mercury in water. The major challenge in developing mercury translators is the enormous variability in the site-specific potential for mercury methylation and bioaccumulation. Among the important factors that contribute to the variability in mercury bioavailability and bioaccumulation are pH, dissolved organic carbon (DOC), salinity, water flow (e.g., stream flow, lake flushing rate, etc.), temperature, reduction-oxidation potential, sulfide and sulfate, suspended solids, nutrient loading, fish age and size, prevalence of wetlands and forested land cover in the watershed, and concentration-dependent demethylation (Brumbaugh et al. 2001; Qian et al. 2001; Kamman et al. 2004; Marvin-DiPasquale et al. 2000; Sonesten 2003a; Rose et al. 1999; Ullrich et al. 2001; Watras et al. 1998).

EPA (2002a, 2004a) draft guidance recommends the following approaches to criteria translation, in order of preference: (1) derive site-specific bioaccumulation factors (BAFs), (2) use a bioaccumulation model, or (3) use EPA's national default translators (EPA 2004a). EPA (2004a) does not recommend any specific bioaccumulation models for mercury, although a modeling approach is preferred over the use of default translators. Despite extensive study, there are no accurate, nationally applicable models for predicting mercury bioaccumulation. As an alternative to process-based or mechanistic mathematical models, the draft EPA guidance suggests that regression models incorporating such variables as pH, DOC, and fish age may be acceptable for criteria translation purposes.

In addition, the draft EPA guidance includes empirically-derived default values to simplify the translation from the fish tissue criterion to a water quality criterion for total mercury. Two sets of values are provided: (1) BAFs describing the relationship between dissolved methylmercury in water and methylmercury in fish, and (2) fraction dissolved (f_d) values describing the relationship between total mercury and dissolved methylmercury in water.

Moreover, the guidance describes partition coefficients (K_D values) that account for the role of total suspended solids (TSS) in determining dissolved mercury concentrations, although default values are not identified.

Despite the identification of default translators, the preferred approach to criteria translation, as recommended by EPA, is the use of site-specific translators. Provided that the study design accommodates an expectation of high variability, it should be possible to effectively develop site-specific translators for mercury.

Mercury Translations in TMDL/Permits

For many facilities, the major issue in developing mercury translators is incorporating the fish tissue mercury criterion into a discharge permit limit. Permits may be reopened and reassessed as part of a TMDL implementation. At least 45 states have fish consumption advisories due to mercury and over 1,000 water bodies are listed as being impaired due to mercury, thus triggering TMDLs. Several methods are generally used in developing mercury TMDLs: (1) the concentration in fish tissue; (2) the concentration in the water column; or (3) the concentration in sediment. Of the three methods, using a fish tissue concentration as the TMDL target is the most direct measure of the desired endpoint, protection of human health.

Determining how to allocate loadings among point and nonpoint sources is the next major part of a TMDL. EPA (2002a, 2004a) offers three approaches to allocation of loadings, depending upon the relative contributions of point source and nonpoint source loadings. First, where point source loadings dominate, the TMDL should specify reductions in these loadings, alone or together with nonpoint source loadings, to attain water quality standards (WQS). Second, where point source loadings are small, reductions in nonpoint sources are expected to achieve the TMDL. The third scenario also involves relatively small contributions from point sources, but reductions in nonpoint sources are not expected to be sufficient to attain WQS.

The approaches used to implement a mercury TMDL will vary depending upon the allocations, as described above. Where there are waste load allocations (WLAs) to point sources, the traditional method to implement a TMDL is likely to be used (i.e., through the NPDES permit). The implementation of effluent limitations for mercury and other pollutant parameters can be approached through several mechanisms including reasonable potential and development of WQBELs. Where a permittee anticipates mercury limits being incorporated into their permit, they should be prepared to proactively take steps to understand the concentrations of mercury that are discharged from their facility (and the nature of these sources), the receiving stream characteristics (including sediment and aquatic species), and the policies of permitting and regulating mercury by the permitting authority. Despite the cost, collection of site-specific data affords dischargers opportunities to develop more realistic bioaccumulation scenarios, potentially allowing the calculation of a more appropriate water quality criterion.

Summary and Recommendations

This report summarizes the issues associated with translation of a mercury fish tissue concentration into a water quality criterion. Section 2 presents an overview of the analytical methods available for analyzing total mercury and methylmercury in fish tissue, water, and sediment. Section 3 provides an analysis of the methods and models available to translate the fish tissue concentration into a water quality criterion. Section 4 describes the impacts of the mercury water quality criteria on TMDL development and NPDES permits. Finally, Section 5 discusses the applicability of these issues for other metals. Although the report addresses many topics, it also identified several areas where additional research or study is necessary to fully understand mercury translation in the environment. The following bullets summarize the key findings and recommendations for additional study:

- Validation of methylmercury analytical techniques is necessary to increase the certainty of results.
- Research is needed to improve the national default translators currently proposed by EPA. EPA's data set of BAF, f_b , and K_D values should be updated with more recent studies. Additional data would increase the effectiveness of the translator calculation methods by reducing variability and minimizing the uncertainty of the resulting default values.
- National default values are likely to be inaccurate on a site-specific basis, given the very high degree of variability observed in mercury bioaccumulation rates. Models have the potential to account for environmental factors contributing to this variability, but at present the available models are limited to reservoirs and lakes in a few geographic regions.
- Improvements in national default translators do not decrease the importance of site-specific translators. It is feasible to develop site-specific translators, although the level of sophistication will be driven by costs

associated with data collection and testing. As the complexity of the site increases, the perceived need to investigate factors that may contribute to spatial or temporal variation in mercury bioaccumulation will also increase.

- Given the many uncertainties associated with mercury translators, their use should be limited to cases where fish tissue data show exceedance of the tissue-based water quality criterion, and point sources make up a significant contribution to mercury loading in the system. This recommendation is in contrast to the draft EPA guidance, which would allow states to adopt water-based rather than tissue-based mercury criteria for the protection of human health.
- Underlying the use of any type of mercury translator is the premise that mercury levels in fish will respond in a linear manner to reductions in mercury loading.
- It is reasonable to assume that the states, given their lack of resources, will follow the simplest approach, whether or not it is the most scientifically valid. Thus, whole waterbodies or even entire states are likely to be designated impaired and subject to TMDL analyses due to levels of methylmercury in fish, regardless of whether or not site-specific data exist.
- Changes to permit limits are likely to be derived in the absence of a RPA and the accurate consideration of non-point source contributions. To allow permit revisions, many states may default to a state-wide BAFs to allow translation of the fish tissue criterion into a water column value, which can then be implemented in NPDES permits in the traditional manner.
- As the use of EPA Method 1631 becomes more widespread, many dischargers are expected to face extremely low permit limits. At a minimum this will likely entail monitoring for mercury and developing a plan to minimize mercury in discharge waters. In some cases, permittees may be required to monitor ambient waters and local fish tissue.
- The collection of site-specific data affords dischargers opportunities to develop more realistic bioaccumulation scenarios. The cost of this could be shared between groups of dischargers within a waterbody.

1 Introduction

This report presents an overview and discussion of the use of the ambient water quality criterion for methylmercury developed by the U.S. Environmental Protection Agency (EPA) in 2001 (66 FR 1344). Unlike all other previous water quality criteria for the protection of human health, the criterion for methylmercury was issued as a fish tissue concentration (i.e., 0.3 mg methylmercury/Kg wet weight fish tissue), as the fish consumption pathway is the main route of exposure to mercury in the environment. As a result, enforcement of water quality standards based upon this criterion requires either fish tissue sampling or the conversion of the criterion to a concentration in water, especially for the development of National Pollutant Discharge Elimination System (NPDES) permits.

Because many permitted dischargers will be faced with meeting the methylmercury criterion, this report focuses on (1) available analytical methods for evaluating mercury in fish and water;; (2) the proposed methods for translating a fish tissue concentration for mercury into a concentration in water; and (3) implementation of the mercury criterion in the development of Total Maximum Daily Loads (TMDLs) and water quality-based effluent limits (WQBELs). EPA (2002a, 2004a) draft *Guidance for Implementing the January 2001 Methylmercury Water Quality Criterion* addresses these issues and is discussed below in each relevant section.

Issues regarding mercury are significant because of mercury's complex cycling in the environment, high bioaccumulative potential, and high toxicity. Generally, inorganic mercury is deposited to water bodies through atmospheric deposition (the major pathway), runoff, stormwater discharge, or other industrial or municipal discharges. Once in the water body, mercury deposited in sediments or dissolved in water may be methylated by bacteria to form methylmercury. Methylmercury is highly bioaccumulative and is the form of mercury that is accumulated up the food web. EPA's criterion for methylmercury assumes, therefore, that all mercury in fish is methylated. While this may be true, as discussed in Section 3.1, use of appropriate analytical methods for both total mercury and methylmercury are critical to fully understand site-specific mercury cycling. In addition, as discussed in EPA (2002a) guidance, several methods are available to translate the fish tissue criterion to a water concentration. Although site-specific data may provide the most reliable results, other default options are available, if needed. Finally, EPA (2002a; 2004a) attempts to provide recommendations for using the mercury criterion in the development of TMDLs and WQBELs, and the implementation of these in the NPDES permitting program. Several

different approaches have been used for mercury TMDLs, as no single approach is universally applicable given the site-specific factors that influence mercury deposition, transformation, and accumulation and the nature of the contributing sources to a water body. The approaches that have been taken and their acceptability to EPA, or lack of acceptability, are illustrated with examples.

To address these issues, Section 2 provides a discussion of the analytical methods for evaluating total mercury and methylmercury in fish tissue and water. Section 3 discusses the various methods for conducting a translation of the methylmercury criterion. Section 4 addresses the use of the criterion in developing TMDLs and WQBELs. Finally, Sections 5 and 6 present a discussion of the applicability of the issues to other metals and conclusions/recommendations, respectively. Appendix A provides a bibliography of recent studies that can be used to update default mercury translators. Appendix B describes several mercury TMDLs that are used to illustrate the different approaches.

2 Analytical Methods

Analytical methods to quantify different species of mercury in environmental samples continue to be refined. Section 2 discusses the various methods available for evaluating the forms of mercury in fish tissue and water and reviews factors to consider in selecting an appropriate analytical method.

2.1 METHODS FOR EVALUATING TOTAL MERCURY IN FISH TISSUE AND WATER

For measuring total mercury, EPA Method 1631 can be used on water samples, while EPA Method 747B/SW-846 can be used for solid or semi-solid samples. A brief description of each method, its detection limits, precision, accuracy, and costs is provided below.

2.1.1 Method 1631

EPA Method 1631 can be used for the determination of total mercury in filtered and unfiltered water (EPA 2002b). Method 1631 is carried out in several steps. First, the various mercury species are oxidized to Hg(II) with bromine monochloride (BrCl). Second, the mercury is reduced to elemental mercury (Hg(0)) with hydroxylamine hydrochloride (NH₂OH·HCl) and stannous chloride (SnCl₂). Third, mercury is purged from the sample onto gold traps for pre-concentration. The gold traps are heated to release the elemental mercury and the detection is performed with cold-vapor atomic fluorescence spectroscopy (CVAFS). Method 1631 has a method detection limit of 0.2 ng/L. This is a performance-based method and actual criteria data are determined before and during analysis. Precision is based on replicate analysis of actual samples and has a limit of 21% RSD (relative standard deviation). Accuracy is determined using National Institute of Standards and Technology (NIST) standards for ongoing precision and recovery (OPR) and matrix spikes/matrix spike duplicates (MS/MSD). OPR samples must fall within 77 – 123% and MS/MSD samples between 71 – 125%. Many laboratories perform this analysis; costs range from \$55 – 200 per sample with a four week reporting time.

2.1.2 Method 7471B/SW-846

EPA Method 7471B/SW-846 is designed for solid or semi-solid samples, but may also be used for water or aqueous samples, as the analysis portion of this method is covered by EPA Method 245.5 (EPA 1994a). Using this method, solid or liquid samples are digested using a potassium permanganate and acid digestion. The acid digestion ensures that all forms of mercury are oxidized to a water soluble Hg(II) form. The detection is performed using cold-vapor atomic adsorption spectroscopy (CVAAS). In CVAAS the sample is reacted with stannous chloride and the mercury is converted to elemental mercury. The elemental mercury is purged from the sample with nitrogen to an atomic adsorption detector. Typical method detection limits for mercury using this method are 0.2 µg/L. This is a performance based method and actual criteria data are determined before and during analysis. Precision is based on replicate analysis of actual samples although no limits were set in the method. Accuracy is determined using NIST standards for ongoing precision and recovery (OPR) and matrix spikes and matrix spike duplicates (MS/MSD). OPR samples must fall within 90 – 110% and MS/MSD samples between 85 – 115%. Many laboratories perform this analysis and the costs range from \$51 – 200 per sample with a four week reporting time.

2.2 METHODS FOR EVALUATING METHYLMERCURY IN FISH TISSUE AND WATER

The basic method for quantifying methylmercury is EPA Method 1630. Modifications to this method are described in the UW-Madison Standard Operating Procedure (SOP) and the United State Geological Service (USGS) Method.

2.2.1 Method 1630

EPA Method 1630 is the main method for quantifying methylmercury in fish tissue and aqueous samples (EPA 2001a), although it has not yet been promulgated. The specific steps in Method 1630 include: 1) distillation to remove the methylmercury from the sample; 2) ethylation to a methylethylmercury form and collection on a graphitic carbon adsorbent trap; and 3) thermal desorption through a gas chromatography column to separate the mercury species before reduction to elemental mercury in a decomposition furnace. The elemental mercury is detected using CVAFS. Method 1630 has a method detection limit (MDL) of 0.02 ng/L (or ng/g) but has the ability to detect as low as 0.009 ng/L.

Also included in this Method is Appendix A for the determination of dimethylmercury. The method for dimethylmercury varies from the original Method 1630 by eliminating the distillation step and purging the entire sample onto the graphitic carbon adsorbent trap. The elemental mercury is separated from the dimethylmercury by using gas chromatography. Two mercury peaks, representing the elemental and dimethylmercury species, are separated in time and are detected by CVAFS. Appendix A for the determination of dimethylmercury has a MDL of 2 pg/L. This is a performance based method and actual criteria data are determined before and during analysis. Precision is based on replicate analysis of actual samples and has a limit of 31% RSD. Accuracy is determined using NIST standards for OPR and MS/MSD. OPR samples must fall within 67 – 133% and MS/MSD samples between 65 – 135%. Several studies have been performed citing the Appendix A method that may be eventually used for validation. In the interim, Appendix A is still considered draft. Unlike EPA Method 1630, the method for dimethylmercury does not specifically discuss how to analyze solids such as fish tissue.

2.2.2 UW-Madison SOP

The UW-Madison SOP was developed at the University of Wisconsin-Madison (Hurley et al. 1988). It is virtually identical to Method 1630 with the major exception being the distillation of a larger sample volume, resulting in a lower method detection limit. Copper sulfate is added to the distillation to bind sulfates. This method has a method detection limit of 30 pg/L for methylmercury. The same laboratories that perform Method 1630 could perform this analysis at a cost of approximately \$200 per sample with a four week reporting time.

2.2.3 USGS Method

Similar to the UW-Madison SOP, the USGS method is virtually identical to Method 1630 with a slightly larger sample volume used in the distillation (DeWild et al. 2001). Copper sulfate is also added to the distillation to bind sulfides. The USGS method has a MDL of 40 pg/L for methylmercury. The same laboratories that perform Method 1630 could perform this analysis and the cost is approximately \$200 per sample with a four week reporting time.

2.3 CLEAN HANDS SAMPLING

EPA Method 1669 covers the sampling and preservation of samples using clean hands techniques (EPA 1996a). Specifically, the method details the procedures for collecting water samples using a two person team, wherein one person becomes the “clean hands” sampler, while the second person, the “dirty hands” sampler, is available to operate equipment and perform tasks where contamination may be present. Procedures such as preparation of blanks, duplicates, spikes, sample filtration, and preservation are described in this method.

2.4 CONSIDERATIONS IN THE SELECTION OF AN ANALYTICAL METHOD

The selection of a method to test for either total mercury or methylmercury should be based on several factors, including method detection limit, validity of the method, and quality control procedures. These factors determine the quality and usability of the generated data. Without specific quality assurance (QA) criteria, comparison between samples and matrix interference may not be possible. Similarly, if QA criteria are too wide, the results may be highly variable between samples. Tighter QA criteria allow the generation of higher quality data.

The MDL is determined by the actual method used to analyze mercury. For example, methods using atomic fluorescence have a lower MDL than atomic adsorption for the determination of total mercury. In particular, atomic fluorescence has a broader linear dynamic range and greater sensitivity than atomic adsorption.

In comparison to total mercury methods, methods for methylmercury are still experimental and have not been fully validated by EPA. Indeed, many more studies have been performed on development and validation of total mercury methods compared to methylmercury, due to greater interest by EPA and industry. Methylmercury analysis is typically performed on fish tissue to better understand bioaccumulation of mercury and determine the partitioning between total mercury and methylmercury.

Similarly, many laboratories are capable of running both EPA Methods 1631 and 7471B for total mercury and have developed competitive services to support this need, including well developed quality assurance/quality control (QA/QC) procedures. In contrast, there are only a few laboratories capable of performing methylmercury analysis. As a result, most analytical laboratories have not developed methods for methylmercury analysis as a routine service. Prior to analyzing samples for methylmercury, it is critical to ensure that the laboratory has the capability to generate results that meet QA/QC standards.

3 Translators Issues

This section discusses EPA's draft guidance and evaluates the available options for establishing site-specific translators. Translation of EPA fish tissue criterion into a water column concentration will typically be necessary when developing water quality- based effluent limits. However, in some cases, it may not be necessary to translate EPA's fish tissue methylmercury criterion to a water quality criterion (EPA 2004a). For example, if elevated mercury concentrations in fish are due primarily to atmospheric or other non-point inputs, point source dischargers may simply be required to maintain mercury levels in effluent at or below current levels (see Section 4).

3.1 OVERVIEW OF TRANSLATIONS WITHIN AND BETWEEN MEDIA

Conceptually, the translation from methylmercury in fish to total mercury in water can be viewed as a single step (i.e., direct method) or a multi-step process, as shown in Figure 1. In the direct method, total mercury in fish is directly translated to total mercury in water. In contrast, the multi-step approach (methylmercury in fish to dissolved methylmercury in water to total mercury in water) reflects the fact that in water, methylmercury is much more bioaccumulative, and dissolved methylmercury is much more bioavailable than particulate mercury. In addition, translation from methylmercury in fish to total mercury in fish may also be implemented, since total mercury is easier and less expensive to measure. However, even the inclusion of various intermediate steps in the translation process is actually a gross oversimplification of the very complex processes of mercury bioaccumulation. From an implementation perspective, it often may be more practical to collapse the translation into a simple ratio between readily measurable endpoints, namely total mercury in fish and water (see Section 3.3).