

DRAFT

Methodology for Derivation of Pesticide Sediment Quality Criteria
for the Protection of Aquatic Life

Phase II: Methodology Development and Derivation of
Bifenthrin Interim Bioavailable Sediment Quality Criteria



Prepared for the Central Valley Regional Water Quality Control Board

Tessa L. Fojut, Ph.D.,

Martice Vasquez, Ph.D.,

Kelly Trunnelle, Ph.D.,

and

Ronald S. Tjeerdema, Ph.D.

Department of Environmental Toxicology

University of California, Davis

February 2014

Disclaimer

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Executive summary

The goal of this project is to develop a methodology for derivation of pesticide sediment quality criteria (SQC) for the protection of aquatic life in the Sacramento and San Joaquin River basins of California. The project will be accomplished in three phases. Phase I was an extensive review, comparison and evaluation of existing sediment criteria derivation methodologies used worldwide (Fojut et al. 2011). This is a report of the results of Phase II, which is the development of a new sediment criteria derivation methodology, based on the findings of the Phase I review. The new methodology, termed the University of California Davis Sediment Methodology (UCDSM), incorporates the latest available research on nonpolar organic contaminant bioavailability, aquatic ecotoxicology and environmental risk assessment. As part of Phase II, the UCDSM was used to derive sediment criteria for bifenthrin, which are included in this report as an illustration of the method. Phase III will be to further apply the UCDSM by deriving criteria for additional pesticides of concern in the Sacramento and San Joaquin River basins that are the cause of listings under Section 303(d) of the Clean Water Act (CRWQCB-CVR 2010).

The goal of this methodology is to consider bioavailability in both deriving SQC and determining compliance to the greatest extent possible. It is imperative to incorporate bioavailability into the derivation of SQC to produce criteria that can be used across sediments, which we have termed bioavailable sediment quality criteria (BSQC). Bioavailable concentrations may be measured or estimated in several ways. Acceptable current methods include 1) normalizing the measured sediment concentration to the organic carbon content, and 2) normalizing the measured whole interstitial water concentration to the dissolved organic carbon content, 3) estimating the freely dissolved concentration in interstitial water via solid-phase microextraction or other non-depleting equilibrium-based techniques, and 4) estimating the “bioaccessible” concentration via Tenax[®] extraction or other depletion techniques. BSQC can be expressed as both sediment and aqueous interstitial water concentrations. It is possible to calculate the aqueous interstitial water concentration from the sediment concentration and vice versa using a partitioning coefficient.

The UCDSM is based on the University of California Davis methodology (UCDM) for deriving water quality criteria (TenBrook et al. 2010), in that both an acute and chronic BSQC are derived using either an assessment factor (AF; less than 5 taxa) approach or a species sensitivity distribution (SSD; more than 5 taxa), depending on the number of taxa represented in the acute and chronic SSTT data sets. The pesticide-specific AFs derived in the UCDM are recalculated for the UCDSM to include newly available data, including for pyrethroid insecticides, and to be more relevant to benthic organisms by requiring benthic crustacean data instead of a Daphnid for criteria calculation via AF. The default acute-to-chronic ratio (ACR) derived in the UCDM is also updated with pyrethroid data and recalculated for inclusion in the UCDSM. Guidance on the collection, evaluation, and prioritization of physicochemical and

ecotoxicity data is adapted from the UCDM (TenBrook et al. 2010), as many procedures are applicable to both water column and sediment data. The derived BSQC are compared to exposure effects data for sensitive species, ecosystems, and threatened and endangered species to determine if the derived criteria are protective based on all available data. Both the UCDSM and the UCDM (TenBrook et al. 2010) provide guidance to assess the bioaccumulation of pesticides that may affect terrestrial wildlife or humans. As in the UCDM, the UCDSM also provides guidance to determine if water quality (e.g., pH, temperature) and mixture effects on toxicity can be incorporated into criteria compliance.

For compliance monitoring, bioavailability should also be accounted for by measuring or estimating concentrations using one of the techniques recommended above for use in toxicity tests. Total sediment or total interstitial water concentrations are not preferred for compliance monitoring for nonionic hydrophobic compounds ($\log K_{ow} > 3$) because sorption to total or dissolved organic carbon will likely confound results. At this time, there are very few spiked-sediment toxicity studies, and even fewer that report freely dissolved concentrations in interstitial water or use it to calculate an effect level. Until such data are available, the use of SSDs will be limited for sediment criteria derivation.

As an illustration of the UCDSM, the method is used to derive acute and chronic BSQC for bifenthrin, a pyrethroid insecticide. Acceptable acute toxicity values were available for two of the five taxa requirements, *Hyalella azteca* and *Chironomus dilutus*. The available acute SSTT data were all reported as OC-normalized sediment concentrations, and as such an acute BSQC of 27 ng/g OC was derived using an assessment factor. Because no chronic data were identified, the default ACR was used to derive the chronic BSQC of 5 ng/g OC. None of the available toxicity values were lower than the derived acute and chronic BSQC, but it should be noted that no relevant or reliable chronic SSTT data were identified for bifenthrin. In accordance with the bifenthrin WQC, mixtures with other pyrethroids should be incorporated into criteria compliance by assuming additivity for the BSQC.

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List of acronyms and abbreviations

ACR	Acute-to-chronic ratio
AF	Assessment factor
ANZECC	Australia and New Zealand Environment and Conservation Council
ARMCANZ	Agriculture and Resource Management Council of Australia and New Zealand
ASTM	American Society for Testing and Materials
BAF	Bioaccumulation factor
BCF	Bioconcentration factor
BCF _{fish}	Bioconcentration factor for fish on a wet weight basis (concentration in fish/concentration in water)
BMF	Biomagnification factor
BMF _{fish}	Biomagnification factor for fish (concentration in fish/concentration in food)
BSAF	Biota-sediment accumulation factor
BSQC	Bioavailable sediment quality criteria
CAS	Chemical Abstract Service
CCME	Canadian Council of Ministers of the Environment
C_d	Freely dissolved chemical concentration
CDFW	California Department of Fish and Wildlife
CDPR	California Department of Pesticide Regulation
C_{DOC}	Dissolved organic carbon-complexed chemical concentration
C_{iw}	Chemical concentration in interstitial water
CRWQCB	California Regional Water Quality Control Board, Central Valley Region - CVR
$C_{s,oc}$	Organic carbon normalized sediment concentrations
DDT	Dichlorodiphenyltrichloroethane
DOC	Dissolved organic carbon
DOE	United States Department of Energy
DOM	Dissolved organic matter
EC _x	Concentration that affects x% of exposed organisms
ECB	European Chemicals Bureau
EPA	Environmental Protection Agency
ERL	Environmental risk limit
ERL(sed)	Environmental risk limit for the sediment compartment using EqP theory
EqP	Equilibrium partitioning
ESG	Equilibrium sediment guideline
EU	European Union
FCV	Final chronic value
f_{oc}	Fraction of organic carbon
HPLC	High performance liquid chromatography
HSDB	Hazardous Substance Data Bank
ICE	Interspecies Correlation Estimation
IUPAC	International Union of Pure and Applied Chemistry

<i>K</i>	Interaction coefficient
<i>K_d</i>	Solid-water partition coefficient
<i>K_{DOC}</i>	Dissolved organic carbon-water partition coefficient
<i>K_{OC}</i>	Organic carbon-water partition coefficient (also described as organic carbon-normalized solid-water partition coefficient)
<i>K_{OW}</i>	Octanol-water partition coefficient
<i>K_x</i>	Interaction coefficient for a synergist/antagonist at concentration x
L	Less relevant/reliable
λ	Factor by which a population increases in a given time
LC _x	Concentration lethal to x% of exposed organisms
LL	Less relevant and less reliable
LN	Less relevant and not reliable
LOEC	Lowest observed effect concentration
LR	Less relevant but reliable
MATC	Maximum acceptable toxicant concentration
<i>m_{DOC}</i>	Concentration of dissolved organic carbon in the whole interstitial water
MPC	Maximum permissible concentration
N	Not relevant/reliable
NOAA	National Oceanic and Atmospheric Administration
NOEC	No observed effect concentration
NSTP	National Status and Trends Program
OC	Organic carbon
OECD	Organisation for Economic Co-operation and Development
OM	Organic matter
OPP	Office of Pesticide Programs
OPPTS	Office of Prevention, Pesticides and Toxic Substances
PAH	Polycyclic aromatic hydrocarbons
PBO	Piperonyl butoxide
PCB	Polychlorinated biphenyls
PEC _{oral}	Predicted environmental concentration a predator will receive in prey (food)
PEC _{sed}	Predicted chemical concentration in sediment
PEC _{water}	Predicted chemical concentration in water
<i>pK_a</i>	Acid dissociation constant
QSAR	Quantitative structure activity relationship
R	Relevant/reliable
<i>r</i>	Intrinsic rate of population growth
RIVM	National Institute for Public Health and the Environment, Bilthoven, The Netherlands
RL	Relevant but less reliable
RN	Relevant but not reliable
RR	Relevant and reliable
RWQCB	Regional Water Quality Control Board

SPME	Solid-phase microextraction
SSD	Species sensitivity distribution
SSTT	Spiked-sediment toxicity testing
SQC	Sediment quality criteria
SQG	Sediment quality guideline
SWRCB	State Water Resources Control Board
TES	Threatened and endangered species
TU	Toxic unit
UCDM	University of California Davis water quality criteria derivation methodology
UCDSM	University of California Davis sediment quality criteria derivation methodology
US	United States
USEPA	United States Environmental Protection Agency
USFDA	United States Food and Drug Administration
WQC	Water quality criteria

1 Introduction

The goal of this project is to develop a methodology for derivation of pesticide sediment quality criteria (SQC) for the protection of aquatic life in freshwater ecosystems, particularly for the Sacramento and San Joaquin River basins of California. The surface waters of these basins receive pesticide inputs in runoff and drainage from agriculture, silviculture, and residential and industrial storm water (CRWQCB-CVR 2011). The term pesticide is defined by the California Regional Water Quality Control Board, Central Valley Region (CRWQCB-CVR) as “(1) any substance, or mixture of substances which is intended to be used for defoliating plants, regulating plant growth, or for preventing, destroying, repelling, or mitigating any pest, which may infest or be detrimental to vegetation, man, animals, or households, or be present in any agricultural or nonagricultural environment whatsoever, or (2) any spray adjuvant, or (3) any breakdown products of these materials that threaten beneficial uses” (CRWQCB-CVR 2011).

The project will be accomplished in three phases. Phase I was an extensive review, comparison and evaluation of existing criteria derivation methodologies used worldwide (Fojut et al. 2011, 2013). This document is a report of the results of Phase II, which is the development of a new sediment criteria derivation methodology, based in part on the findings of the Phase I review. The new methodology, termed the University of California Davis Sediment Method (UCDSM), is based on a bioavailability approach that incorporates the latest available research into nonpolar organic contaminant bioavailability, aquatic ecotoxicology, and environmental risk assessment. As part of Phase II, the UCDSM is used to derive bioavailable sediment quality criteria (BSQC) for bifenthrin and the derivation is included in this report as an illustration of the method. Phase III is to apply the new methodology and derive criteria for additional pesticides of concern in the Sacramento and San Joaquin River basins that are listed under Section 303(d) of the Clean Water Act (CRWQCB-CVR 2010).

The mission of California’s nine Regional Water Quality Control Boards (RWQCBs) is “to develop and enforce water quality objectives and implementation plans which will best protect the beneficial uses of the State's waters, recognizing local differences in climate, topography, geology and hydrology” (California SWRCB 2011). Toward that mission, each RWQCB is responsible for development of a “basin plan” for its hydrologic area. The “Water Quality Control Plan (Basin Plan) for the Sacramento River and San Joaquin River Basins” contains the following language regarding toxic substances in general, and pesticides in particular (CRWQCB-CVR 2011):

“...waters shall be maintained free of toxic substances in concentrations that produce

detrimental physiological responses in human, plant, animal, or aquatic life."

"No individual pesticide or combinations of pesticides shall be present in concentrations that adversely affect beneficial uses."

"Discharges shall not result in pesticide concentrations in bottom sediments or aquatic life that adversely affect beneficial uses."

"Pesticide concentrations shall not exceed the lowest levels technically and economically achievable."

1.1 Summary of Phase I

In Phase I, Fojut et al. (2011, 2013) identified three main approaches for development of sediment quality guidelines (SQGs): empirical, mechanistic and spiked-sediment toxicity testing (SSTT). The term sediment quality guideline is used in most other jurisdictions, rather than sediment quality criteria, because these types of values are typically only used as triggers in risk assessment, rather than for regulatory compliance. In general, the empirical approaches generate concentration ranges that are very likely, likely, or not likely to cause adverse effects, but they do not establish a cause-effect relationship for a single chemical. The mechanistic approach uses the equilibrium partitioning model to generate single concentrations not to be exceeded that are based on the existence of a water quality criterion for the compound of interest. The third approach uses SSTT data to derive criteria with statistical distributions or by applying an assessment factor (AF; sometimes called safety factors). Several of the methodologies incorporate multiple approaches and recommend deriving criteria from SSTT data if they are available, or comparing the derived criteria to these data if they are limited. In the conclusions of the Phase I report the viability of each approach was assessed. An empirical approach could not provide an acceptable level of certainty in SQC because the majority of data used in these approaches do not demonstrate cause-effect relationships between chemical concentrations and adverse effects. A mechanistic approach could be used for pesticides with existing water quality criteria (WQC), but some of the underlying assumptions of this approach have not been completely validated, leading to higher uncertainty in the values. Finally, the SSTT approach was recommended because it has a strong technical foundation as the data clearly link cause and effect, however, it was noted that there are very few SSTT data available for pesticides and experimental uncertainties may hinder the use of what little data there are.

The most significant conclusion of Phase I was that bioavailability of contaminants must be incorporated to generate reliable SQC. It is an important factor to consider when establishing any type of numeric value and is particularly relevant for

hydrophobic pesticides, which are the most likely to be sediment contaminants. Sediments are very heterogeneous and when they are contaminated this leads to varied uptake and toxicity of those contaminants to aquatic organisms. Within sediments, chemicals can either be bound to the particles or freely dissolved in the interstitial water and they can change from one phase to the other depending on conditions. Interstitial water, sometimes also referred to as porewater, is the water that saturates sediments and is found in the interstices between individual sediment particles. The bioavailable fraction of a contaminant refers to the fraction of a chemical that is available for uptake by organisms via all exposure routes, which for sediment contaminants includes uptake of freely dissolved chemicals from interstitial water (either internally or externally through a dermal membrane) and ingestion of and direct contact with bound contaminants. Sediments are so varied that the concentration that causes acute lethality for a given species in one sediment sample may be completely nontoxic in a different sediment sample. This is why accounting for bioavailability in SQC was identified as the most significant factor in the Phase I review.

1.2 Use of the UCDSM

The UCDSM is considered a companion method to the University of California Davis methodology (UCDM) for deriving water quality criteria (TenBrook et al. 2010), and as such, many procedures used to generate sediment criteria are similar to those used to generate water quality criteria. Risk assessment procedures, such as the evaluation of ecotoxicity data, data prioritization, and criteria calculation techniques (species sensitivity distributions, assessment factors, and acute-to-chronic ratios) are applicable to both water and sediment data. The essential approaches in the UCDSM are the same as those used in the UCDM, with appropriate adjustments made to focus on organisms with sediment-dwelling life stages.

To use the UCDSM, training or expertise in toxicity testing, ecotoxicology, environmental toxicology, environmental chemistry or other related disciplines is required. The goal is to provide a systematic process for evaluating data and calculating criteria, but knowledge of these disciplines and the ability to exercise best professional judgment based on experience are required to arrive at appropriately protective criteria.

It is recommended that the resulting criteria are used for risk assessment purposes or that they are compared to environmental monitoring data as a line of evidence for determining pesticides that may cause or contribute to toxicity. The BSQC generated using the UCDSM have a high degree of uncertainty and thus may not be appropriate to use as strict regulatory values. The main reason for the high degree of uncertainty is that there are very few SSTT data available to use in the method. Because there are few toxicity values or alternative criteria that can be compared to the resulting criteria, the BSQC cannot be well-calibrated or “ground-truthed” to ensure that the criteria are

reasonable and protective. This is one of the main differences between the water quality criteria UCDM and the sediment quality criteria UCDSM – there were many available aqueous toxicity data and water quality criteria to compare to during the development of the UCDM. Due to the high degree of uncertainty and inability to test the method with large data sets, the criteria generated with the UCDSM are referred to as interim BSQC, with the idea that as more data are generated, the method can be fully tested and updated to produce unqualified BSQC.

1.2.1 Goal of UCDSM

Like the UCDM, the goal of the UCDSM is to extrapolate from available pesticide toxicity data for a limited number of species to a concentration (criteria) that should not produce detrimental physiological effects in aquatic ecosystems. These criteria aim to protect all species in an ecosystem, but particularly focus on benthic species or those species with part of their lifecycle in the benthic zone. The UCDSM was designed for the Sacramento and San Joaquin River watersheds, but is generally applicable to freshwater ecosystems in North America. Simple modifications could be made to adapt this method for estuarine or saltwater criteria or other geographic areas.

1.2.2 Overview of the UCDSM approach

This section provides a high-level overview of the UCDSM approach to orient the reader to the document. This section is a narrative of the sequence of procedures to derive criteria, and the process is also illustrated in two flow charts: Figure 1 illustrates data collection, organization, and prioritization, and Figure 2 illustrates the criteria calculation process.

The methodology begins with collection of both physicochemical and spiked-sediment toxicity test (SSTT) ecotoxicity data (section 2.1). Acceptable ecotoxicity data must incorporate an estimate of bioavailability, which is further described in section 1.2.3. Once the data is collected, it is evaluated to ensure that only high quality data are used directly in criteria calculations (section 2.3). Ecotoxicity data are evaluated based on numeric scoring of the study design and documentation and acceptability of study parameters (section 2.3.2). After ecotoxicity data are scored, they are separated into two categories: 1) the acceptable data set that rates as relevant and reliable and can be used in criteria calculation and 2) the supplemental data set that rates as less relevant and/or less reliable. The acceptable data set is separated into acute and chronic data (section 2.4), and they are prioritized so that there is a single toxicity value for each species (section 2.5). These data sets can then be used to calculate criteria.

A statistical approach, utilizing species sensitivity distributions (SSDs) is recommended in the UCDSM (section 3.4), but there are minimum data requirements that

must be met to use a SSD (section 3.4.1.1). The minimum five taxa requirements have not been met for any pesticides thus far because there are only two taxa for which standard test methods are available. The SSD procedure is described in this report (section 3.4.2), but it is unlikely that it will be used until more SSTT data are available for more diverse taxa. Because SSDs are not practical at this time, and may never be unless standard methods are developed for additional taxa, an assessment factor (AF) procedure is used for acute criteria calculation. The goal of the AF procedure is to approximate the 5th percentile of the species sensitivity distribution, without actually fitting a statistical distribution to the data set. To calculate criteria with an assessment factor, the lowest toxicity value in the data set is divided by an AF, and the result is an estimate of the 5th percentile of the distribution. The rationale for assessment factors is given in section 3.5.1 and the straightforward procedure is in section 3.5.2. To calculate chronic criteria for data sets that do not meet the minimum five taxa requirements, an acute-to-chronic ratio (ACR) is applied to the 5th percentile of the distribution estimated using the AF procedure (section 3.6). If paired acute and chronic data are available, the ACR can be calculated from experimental data (section 3.6.1 and 3.6.2), or if paired data is not available, a default ACR is used (section 3.6.3). Once criteria are calculated based on single species SSTT data, the final criteria statements include an averaging period and an allowable exceedance frequency (section 3.7).

Before finalizing the criteria, any data indicating mixture toxicity effects or water quality effects on toxicity (e.g., temperature-dependence) are reviewed. If there are sufficient data, these effects are quantified for mixtures (section 4.2) or water quality effects (section 4.3) and incorporated into the final criteria statement. The criteria are also compared to any available data for sensitive species (section 5.1), threatened or endangered species (section 5.2), or from multispecies ecosystem studies to ensure they are protective of these populations. The criteria may be adjusted if these studies indicate that the criteria may not be protective of these taxa (section 5.5). Finally, the criteria are examined to check that if they are met, they will not cause adverse effects from transfer to other environmental compartments (section 6.1) or due to bioaccumulation up the food chain (section 6.2).

The final criteria are referred to as interim bioavailable sediment quality criteria (interim BSQC) and are summarized in a final criteria statement (section 7.1) including a list of the assumptions and limitations of the calculation (section 7.2).

1.2.3 Accounting for Bioavailability

The essential premise of the UCDSM is to account for bioavailability in both deriving criteria and determining compliance. In the UCDSM, the definition of bioavailability follows that from the National Research Council (NRC 2003), which is given as “the individual physical, chemical, and biological interactions that modify the

amount of chemical actually absorbed or adsorbed (bioavailability) and available to cause a biological response in plants and animals exposed to chemicals associated with soils and sediments.” Stated another way, bioavailability is how much of the total chemical in the environment actually interacts with organisms. Once a chemical is taken up by an organism, either internally or on the surface, it may accumulate, be transformed, be excreted or cause adverse effects (toxicity) in the organism. The bioavailable fraction of a contaminant refers to the fraction of the total chemical pool that is available for uptake by organisms via all exposure routes. Bioavailability and subsequent toxicity is dependent on many factors, including sediment characteristics (e.g., particle size, source or type of organic matter), organism characteristics (e.g., behavior, feeding), chemical properties, contact time, environmental conditions (e.g., temperature, pH), biological activity in the ecosystem (e.g., biotic transformation, cycling, and burial), and potentially others (Davies et al. 1999, Diaz and Rosenberg 1996; You et al. 2011).

Directly measuring bioavailability would involve measuring chemical residues in organisms or interactions of chemicals with organism membranes; any measurement that does not involve an organism is an estimate of bioavailability (McLaughlin and Lanno 2014). Many studies have suggested that bioavailability of contaminants in sediments is accurately predicted by the concentration that is freely dissolved in interstitial water, which has been estimated with passive sampling devices (Harwood et al. 2012; Hunter et al. 2008; You et al. 2011). Interstitial water is described as the water in the interstices between individual sediment particles; this is also referred to as pore water in some of the literature. The estimated freely dissolved interstitial water concentration may not be equal to the true bioavailable fraction because it may overlook the ingestion and direct contact exposure routes, and any measurement that does not include measuring the chemical in an organism will always be an estimate of what organisms may experience. However, recent studies have demonstrated that the correlations between the freely dissolved concentration and uptake or toxicity have much less variation compared to correlation with whole sediment concentrations.

Bioavailability of sediment contaminants is closely related to the binding or uptake of contaminants to sediments, which is also referred to as sorption. It has been demonstrated that nonionic organic compounds, such as many pesticides, primarily sorb to organic matter (OM) contained in sediments or to dissolved organic matter (DOM) in interstitial water (Schwarzenbach et al. 2003). The abundance of OM is usually expressed as the organic carbon (OC) content or the dissolved organic carbon (DOC) content of a sorbent because organic carbon is what is typically measured as a surrogate for organic matter. The amount of a chemical that will sorb to solids is described as a ratio called a solid-water partition coefficient (K_d , also called a distribution coefficient). The solid-water partition coefficient is the ratio of the mass of the chemical sorbed to solids (C_s) to the mass dissolved in water (C_w). Because OM primarily controls uptake (sorption) and

release (desorption) of these compounds to or from sediments, solid-water partition coefficients are often normalized to the OC content to reduce variability of the partition coefficients across different sediments. Partition coefficients are OC-normalized by dividing them by the OC content (fraction of sediment that is OC by mass, f_{OC}). However, even when contaminant concentrations in sediments are OC-normalized, biological uptake and toxicity are not accurately predicted in many cases. This is because naturally-occurring organic matter found in sediments and soils comes from varied sources and has gone through varied processes, making it an extremely heterogeneous matrix (Schwarzenbach et al. 2003).

Some researchers have tried to better characterize organic matter and determine the different components that may exhibit stronger or weaker sorption of HOCs, but this has proven to be very complex (summarized in Schwarzenbach et al. 2003). However, one part of organic matter that has been well-characterized is black carbon, also called soot or charcoal, which is a product of incomplete combustion. It has been observed that sorption of most HOCs to black carbon (BC) is stronger than sorption to natural (non-combusted) organic carbon. Most studies on sorption to black carbon have measured polychlorinated biphenyls (PCBs) and polyaromatic hydrocarbons (PAHs) and less is known about sorption of pesticides to black carbon. One study measured sorption of pyrethroids to black carbon and reported only modest increased sorption to black carbon compared to natural OC (Yang et al. 2009). Black carbon may be more or less important depending on the types of sites that are monitored (some sites are highly enriched with BC) and on the physicochemical properties of the pesticides of interest (e.g., pyrethroids have a high molecular weight that may hinder sorption to BC; Yang et al. 2009). The importance of accounting for BC as well as OC has been recognized by the US EPA and they have incorporated BC into their guidance on equilibrium partitioning sediment benchmarks for the protection of benthic organisms (USEPA 2012). In typical SSTTs and environmental monitoring, the black carbon content of sediment is not measured, thus, it is unlikely that this parameter will be available to be incorporated in the UCDSM. In addition, a black carbon-water partition coefficient would be needed for the pesticide of interest, and these values are not widely available.

Because the unique characteristics of organic matter affect how contaminants bind to it, attempts at predicting sorption or bioavailability across varied sediments can give mixed results. OC-normalized sediment concentrations and DOC-normalized interstitial water concentrations reduce some of the variation across sediments, but sometimes have poor correlations with biological uptake and toxicity. The freely dissolved concentration (C_d) is not equivalent to the bioavailable concentration, but it is directly related to a contaminant's chemical activity. Chemical activity can be related to potential for biological uptake and to cause toxicity because chemical activity “quantifies the potential for spontaneous physicochemical processes, such as diffusion, sorption, and

partitioning,” including diffusive uptake into benthic organisms (Reichenberg and Mayer 2006). Chemical activity is a somewhat abstract concept, but it can be described as the relative energetic level of a chemical at equilibrium. The chemical activity of a particular contaminant will determine its concentration in an organism at equilibrium. When a contaminant is adsorbed to sediment particles, the chemical activity of the contaminant decreases with increasing sorption, and subsequently both the concentration in biota and the freely dissolved concentration would also decrease with increasing sorption (Reichenberg and Mayer 2006). The concept of chemical activity is important in the scientific rationale underpinning the relationship between chemical uptake by a passive sampling device to the freely dissolved concentration, discussed more below in section 1.2.3.3.

Other researchers have worked on analytical methods that estimate the “bioaccessible” concentration, rather than the bioavailable concentration of sediment contaminants. Bioaccessibility is also called extractability or simply accessibility, and can be defined as the amount of chemical that has the potential to become available by desorbing from sediment or dissolved organic matter (Semple et al. 2004, You et al. 2011). Unlike the freely dissolved concentration, the bioaccessible concentration is not directly related to chemical activity, but extractions that characterize or estimate the fraction that rapidly desorbs from sediment have been shown to have good correlations with biota uptake or toxicity (Harwood et al. 2013a, Lydy et al. 2007, You et al. 2006). These types of methods are included in the UCDSM, with the caveat that further method development and standardization is needed before they should be used in criteria derivation.

A study that clearly illustrates how bioavailability and sorption are related was performed by Xu et al. (2007). The researchers measured pesticide concentrations lethal to 50% of exposed organisms (LC_{50}) in three different sediments using *Chironomus dilutus* (formerly *C. tentans*). They measured concentrations in whole sediment and interstitial water and estimated the freely dissolved concentrations in interstitial water and expressed the LC_{50} s in five different ways: total sediment concentrations based on dry weight, OC-normalized sediment concentrations, total interstitial water concentrations, DOC-normalized interstitial water concentrations, and freely dissolved interstitial water concentrations. They demonstrated that the LC_{50} s for the three different sediments were highly variable when they were expressed as whole sediment or whole interstitial water concentrations (% coefficient of variation [%CV]: 33.2-73.9%) and they were less variable when expressed as OC-normalized sediment or DOC-normalized interstitial water concentrations (%CV: 3.7-41.0%). Variation was greatly reduced when LC_{50} s were expressed as freely dissolved interstitial water concentrations (%CV: 0.9-13.0%). This study and others have demonstrated that the freely dissolved interstitial water concentration is a better predictor of bioavailability compared to other available measures

(Hunter et al. 2008, Muir et al. 1985, You et al. 2009). However, standard methods for directly measuring or estimating freely dissolved interstitial water concentrations are not widely available or practiced in commercial laboratories. Normalizing sediment concentrations to OC content or interstitial water concentrations to DOC content have also been demonstrated to be good predictors of bioavailability (Amweg et al. 2005, 2006; Trimble et al. 2008; Weston et al. 2004, 2005, 2008; Xu et al. 2007). Techniques that estimate the “bioaccessible” fraction, or the fraction of a bound contaminant that can potentially be released from sediments, also correlate bioavailability and may be useful in deriving BSQC (You et al. 2009, 2011).

In the UCDSM, the most robust and certain criteria will be generated from SSTT data, with measured effects concentrations that incorporate bioavailability. For these criteria to be protective of aquatic ecosystems, compliance monitoring must also incorporate bioavailability, so that criteria and monitoring results are comparable. In the UCDSM, bioavailable concentrations may be measured or estimated in several acceptable ways: 1) the whole sediment concentration can be measured and normalized to the organic carbon content (and black carbon content if information is available), 2) the whole interstitial water concentration can be measured and normalized to the dissolved organic carbon content, or 3) the freely dissolved concentration in interstitial water can be estimated via passive sampling techniques. The interim BSQC can be expressed as sediment or aqueous interstitial water concentrations, depending on the types of data available. In general, it is not recommended that toxicity values reported in one phase (e.g., OC-normalized sediment concentrations) are converted to other phases (e.g., freely dissolved interstitial water concentrations) because partition coefficients are highly variable and add uncertainty. If site-specific partition coefficients are available, conversion between phases can be appropriate and is described in sections 1.2.3.1 and 1.2.3.2. Including multiple methods for incorporating bioavailability allows for flexibility and using the maximum amount of acceptable toxicity data.

1.2.3.1 Bulk sediment: Organic carbon normalization

The most common way to account for bioavailability is OC-normalization of sediment concentrations. To calculate the OC-normalized sediment concentration ($C_{s,OC}$; typically ng/g OC) from the dry weight (DW) whole sediment concentration (C_s ; typically ng/g DW or $\mu\text{g}/\text{kg}$ DW), the OC content of the sediment must be known. If the OC content is reported as the percentage of OC in sediment (%OC), it can be divided by 100% to convert it to the fraction of OC in sediment (f_{OC} ; unitless). Then the OC-normalized concentration can be calculated as follows:

Eq 1:

$$C_{s,OC} = \frac{C_s}{f_{OC}}$$

The advantage of using OC-normalized sediment concentrations is that bulk sediments and their organic carbon content are routinely measured in both SSTTs and environmental monitoring. This approach is limited by the high variability of bioavailability across sediments due to the unique characteristics of organic matter, meaning that the OC-normalized BSQC may not be a good predictor of ambient toxicity.

OC-normalized sediment concentrations can be converted to freely dissolved interstitial water concentrations using a partition coefficient, but the partition coefficient likely introduces a high degree of uncertainty unless site-specific partition coefficients are available. Partition coefficients have high variability across sorbents and can be difficult to measure accurately for highly hydrophobic compounds, such as pyrethroid pesticides (Mayer et al. 2014, Schwarzenbach et al. 2003). However, there is utility in this conversion for combining toxicity values reported in different phases. The equation to calculate the freely dissolved concentration from the OC-normalized sediment concentration using the OC-water partition coefficient (K_{OC}) is given here:

Eq 2:

$$C_d = \frac{C_{s,OC}}{K_{OC}}$$

If information on sorption to black carbon is also available, this can be incorporated into the calculation of the freely dissolved concentration. The USEPA (2012) gives the following equation:

Eq 3:

$$C_d = \frac{C_s}{f_{OC}K_{OC} + f_{BC}K_{BC}C_d^{n-1}}$$

where:

f_{BC} is the fraction of black carbon by weight in sediment (g BC/g dry weight)

K_{BC} is the black carbon-water partition coefficient (L/kg BC)

n is the Freundlich exponent (dimensionless)

Freundlich exponents and K_{BC} s are chemical-specific and are available for some pyrethroid pesticides (Yang et al. 2009). To solve for C_d , an iterative approach must be used because it appears on both sides of the equation; statistical functions in spreadsheet or statistical programs can be used for this purpose (USEPA 2012).

1.2.3.2 Interstitial water: Dissolved organic carbon normalization

In this approach, interstitial water is isolated from bulk sediment by centrifuging to separate the solids from the water. Then, interstitial water can be extracted and analyzed following typical aqueous analytical methods. The advantage of this approach is that the analysis is available from commercial laboratories and follows existing standardized methods for sample handling and analysis (USEPA 2001). One limitation of this approach is that centrifuging the sediment may disturb equilibrium between the solids, dissolved organic matter, and the aqueous phases (USEPA 2001). Thus, the measured concentration may not be an accurate representation of the freely dissolved concentration.

Binding of contaminants to dissolved organic carbon can be significant, so for the interstitial water concentration to be used in the UCDSM, it must be normalized to the DOC content of the interstitial water. This is not a common practice because the DOC concentration is not measured in most monitoring data, but it is a standard measurement that many commercial laboratories can perform if requested. If this data is available, the whole interstitial water concentration (C_{iw} ; typically $\mu\text{g/L}$) can be normalized to DOC by dividing by the concentration of DOC ($[DOC]$; typically kg/L) as follows:

Eq 4:

$$C_{iw,DOC} = \frac{C_{iw}}{[DOC]}$$

where:

$C_{iw,DOC}$ is the DOC-normalized interstitial water concentration (e.g., $\mu\text{g/kg}$, mg/kg).

In order to convert from either the whole interstitial water or DOC-normalized interstitial water concentration to a freely dissolved concentration, a DOC-water partition coefficient (K_{DOC} ; typically L/kg) is needed. Site-specific partition coefficients are preferred because DOC binding affinities vary widely. If site-specific values are unavailable, as is likely to be the case, the geometric mean of acceptable partition coefficients can be used. The first expression below defines K_{DOC} :

Eq 5:

$$K_{DOC} = \frac{C_{iw,DOC}}{C_d} = \frac{(C_{iw} - C_d)/[DOC]}{C_d}$$

To calculate the freely dissolved concentration if $C_{iw,DOC}$ is not known or to calculate it from C_{iw} , a substitution for $C_{iw,DOC}$ is made in the latter expression of Eq 5 based on C_{iw}

being the sum of the freely dissolved chemical and chemical bound to DOC whole interstitial water, as follows:

Eq 6:
$$C_{iw} = C_d + C_{iw,DOC}[DOC]$$

To solve for C_d , the latter expression of Eq 5 is rearranged, which results in the following expression:

Eq 7:
$$C_d = \frac{C_{iw}}{K_{DOC}[DOC] + 1}$$

1.2.3.3 Interstitial water: Freely dissolved concentration via passive sampling

Passive samplers provide a way to measure the freely dissolved concentration in interstitial water. As discussed above (section 1.2.3), the freely dissolved concentration is directly related to chemical activity, and thus offers a more direct assessment of potential toxicity to benthic organisms (Mayer et al. 2014). This section is intended to serve as a brief overview of the current state of science and available techniques that are based on the concept of equilibrium partitioning. This section is not intended as technical guidance on how to choose the appropriate passive sampling method or operate passive sampling devices. Readers are directed to the literature for the most updated methods and practices. This review relies heavily on a recent series of articles that resulted from a technical workshop entitled “Guidance on Passive Sampling Methods to Improve Management of Contaminated Sediments” held by the Society of Environmental Toxicology and Chemistry in 2012 (Ghosh et al. 2014, Greenberg et al. 2014, Lydy et al. 2014, Mayer et al. 2014, Parkerton & Maruya 2014). This series of articles provides an excellent foundation to understand these methods and how they may be applied in the future.

Passive sampling devices consist of a polymer that acts as a sorbent for hydrophobic organic chemicals. Passive sampling methods based on equilibrium partitioning are sometime called non-depletion techniques because in order to maintain equilibrium, the passive sampler cannot significantly deplete the contaminants from the sediment or interstitial water matrices (Mayer et al. 2003). A basic description of non-depletion passive sampling devices is that the polymer device is placed in or on sediment and HOCs sorb to it and eventually reach equilibrium between the sediment, interstitial water, and the polymer (Mayer et al. 2014). The goal is to choose a polymer configuration that sorbs only a small percentage of the HOCs so that equilibrium is not

disturbed, otherwise C_d measured by the passive sampler may not be equal to C_d in the interstitial water before equilibrium was disturbed, leading to an inaccurate measurement of C_d (Lydy et al. 2014). Once equilibrium is attained, the device is removed, extracted, and the extract concentration is measured with typical analytical methods. The extract concentration can then be related back to the freely dissolved concentration in interstitial water based on a known polymer-water partition coefficient. The most common of these techniques for passive sampling is matrix-solid-phase microextraction (matrix-SPME), which is configured as a thin fiber coated with a polymer. Matrix-SPME has been used to assess uptake and toxicity of sediment-bound pesticides to aquatic organisms in several studies (Ding et al. 2012a, Ding et al. 2012b, Ding et al. 2013, Harwood et al. 2013a, Xu et al. 2007).

To determine which type of non-depletive technique to use, one must choose the polymer type and device configuration and decide whether it will be applied in the laboratory (*ex situ*) or in the field (*in situ*). The choice of polymer and configuration will depend on the physicochemical properties of the target analytes, the time to equilibrium, and the analytical detection limits of the analytes. In the *ex situ* approach, field sediments are taken to the laboratory or sediments are spiked in the laboratory (e.g., in a SSTT) and then the passive sampler is placed in the sample. This approach allows the conditions of the passive sampler to be controlled and recorded and may be more acceptable for use in SSTTs or compliance monitoring because the methods can be standardized and controlled. In the *in situ* approach, the passive sampler is placed in the field for a period long enough to reach equilibrium or to estimate C_d because it is difficult to ensure that equilibrium has been attained in the field. The *in situ* approach may be preferred for characterizing field conditions that are difficult to recreate in the lab, such as variations in interstitial water concentrations with depth (Ghosh et al. 2014). Based on the current state of science, *ex situ* (laboratory) measurements are recommended for use in SSTTs and compliance monitoring of C_d in sediment samples for the UCDSM.

Various polymers have been employed in different devices, such as polydimethylsiloxane, polyethylene, and polyoxymethylene. These polymers can be configured in various ways in the passive sampling device, such as coated fibers, sheets, or vials containing thin films (Lydy et al. 2014). There are two main types of passive sampler techniques, those that operate at equilibrium conditions, and those that incorporate uptake kinetics, meaning a chemical concentration is measured at a specific time, which is then corrected to the equilibrium concentration based on knowledge of the uptake kinetics (Lydy et al. 2014). To determine which type of passive sampling technique to employ, the main considerations are available exposure time compared to time to equilibrium and the analytical detection limits of the target compounds (Lydy et al. 2014). The available exposure time and time to equilibrium will determine whether an equilibrium or kinetic technique should be used. The detection limits of target

compounds will determine the volume of the sampler – a larger volume is able to sorb more mass, which will increase the ability to detect it. However, the larger the sampler volume, the longer it takes to attain equilibrium. To derive C_d from the concentration in the passive sampling device (C_p) at equilibrium, the equilibrium partition coefficient between the polymer and the analyte (K_{pw}) must be experimentally derived. It is difficult to define the uptake kinetics into passive samplers because there may be many processes involved the mass transfer kinetics of a contaminant, and these processes are difficult to isolate and quantify (Mayer et al. 2014). Accurately determining K_{pw} can be challenging, so published partition coefficients for commonly used polymers and analytes can be used when available. However, it should be noted that variation between suppliers and batches of materials may affect partitioning, and thus estimates of C_d . For this reason, Ghosh et al. (2014) recommend purchasing large batches of polymers so K_{pw} does not vary, and when a new batch is purchased K_{pw} should be re-confirmed, although the differences are expected to be small. When C_p and K_{pw} are known, then C_d can be calculated as C_p/K_{pw} .

The advantage of equilibrium techniques is that equilibrium partitioning to passive samplers is well understood and defined, which allows for accurate and precise measurements, and results that can be replicated (Mayer et al. 2014). In addition, these techniques have great flexibility because the polymer and configuration can be optimized for the analytes, and they can be used either in the field or the laboratory, and can be exposed simultaneously with organisms (You et al. 2011). Reported freely dissolved interstitial water concentrations in toxicity tests are easily incorporated into the UCDSM because there is no need to normalize the concentrations to other parameters. One major limitation of passive sampling methods is that widely-adopted standardized methods are not yet available. However, this field of research is developing rapidly, and standard methods may be available in the near future. Until standard guidance on passive sampling methods is available, the use of these techniques for regulatory monitoring or performing toxicity tests may not proliferate. Another disadvantage of equilibrium techniques is that they can be time-intensive and labor-intensive because it can take weeks to months for the passive sampler to reach equilibrium with sediments (You et al. 2011). In addition, because these passive sampling methods have a low capacity for sorption in order to avoid depleting the contaminant and disturbing equilibrium, they are not always able to sorb a large enough amount of contaminant to be detected by analytical methods, which can be a problem for compounds that are toxic at very low trace levels (e.g., pyrethroids).

1.2.3.4 Bioaccessibility: Rapidly desorbing fraction

The bioaccessible fraction is the fraction of contaminant that rapidly desorbs from sediment, which is an estimate of contaminants that will potentially be available to organisms (You et al. 2011). Unlike the equilibrium-based passive sampling methods, techniques to measure bioaccessibility rely on disturbing the equilibrium so that the

sediment sample is mildly extracted. These are sometimes termed depletion techniques because they deplete a fraction of the contaminant from the sediment matrix, in contrast to the non-depleting techniques. The only depletion technique that is commonly used is Tenax® extraction. Tenax is a polymer configured as a powder or beads that is placed in a water-sediment sample and it acts as an “infinite sink,” meaning HOCs continuously desorb from sediment and sorb to Tenax (Pignatello 1990). The assumption for a Tenax extraction is that equilibrium is not attained during the extraction, and instead desorption kinetics are characterized. The extraction time and conditions (e.g., temperature, mixing) affect the extent of contaminant sorption to the Tenax; for precise results, the extraction conditions and duration must be well controlled. After the Tenax sorbs the contaminant(s) for the specified duration, the Tenax is removed from the sample jar and solvent extracted, and then standard analytical methods can be used to quantify the contaminant concentration or mass (typically reported as µg/g sediment, or µg/g OC).

Sequential extractions can be performed by removing the Tenax at certain time intervals and replacing it with fresh Tenax to characterize the desorption kinetics of the contaminant. The fraction that rapidly desorbs has been correlated to contaminant accumulation in biota (Mehler et al. 2011, You et al. 2007). A simplified single time point extraction (e.g., 6 hours or 24 hours) has also been used as an estimate of the rapidly desorbing fraction and correlations with accumulation and toxicity of pesticides in biota have been observed using this technique (Harwood et al. 2012, 2013a, 2013b).

Like equilibrium-based passive sampling methods, the main disadvantage of Tenax extraction is that widely available standard methods are not available, and uses for regulatory monitoring or toxicity testing will likely not become more prevalent until such methods are available. However, a single time point Tenax extraction could provide a quick, inexpensive estimate of the bioaccessible concentration for environmental monitoring or SSTTs. Tenax has the advantage of extracting a larger fraction of the contaminant compared to equilibrium-based techniques, which increases detection of trace level contaminants (You et al. 2011). Compounds that rapidly degrade may also be more easily detected using Tenax than equilibrium-based techniques because a single time point Tenax extraction can be as short as 6-24 hours, compared to the weeks-months it may take to reach equilibrium (You et al. 2011).

1.2.4 Relevant compounds

The UCDSM is intended for deriving sediment quality criteria for pesticides. In this method, pesticides are defined as (1) any substance or mixture of substances that is intended to be used for defoliating plants, regulating plants growth, or for preventing, destroying, repelling, or mitigating any pest, which may infest or be detrimental to vegetation, man, animals, or households, or be present in any agricultural or non-

agricultural environment whatsoever, or (2) any spray adjuvant, or (3) any breakdown products of these materials that threaten beneficial uses. While this method is appropriate for both legacy and current-use pesticides, we focus the examples on current-use pesticides throughout the method.

The recommended techniques to account for bioavailability, described above, were made based on a large review of the literature that focuses on nonionic hydrophobic organic compounds. Thus, the UCDSM is only intended to be used for organic pesticides that are both nonionic and hydrophobic. The pesticides must be organic because metals or other inorganic compounds have significantly different physicochemical properties and are not likely to behave according to the assumptions inherent in the techniques for accounting for bioavailability. In addition, certain procedures were derived using only data on organic pesticides and have not been validated for metals or other inorganic compounds (noted in AF section 3.5 and the default ACR section 3.6.3). Relevant pesticides must be nonionic, that is, they do not form ions in waters with pH ranges typically found in the environment (pH 4-10); nonionic compounds are neutral, that is, they do not form charged ions (cations and anions). Relevant pesticides should also have a $\log K_{ow} > 3$, which is a common way to define hydrophobic, although exceptions could be made if the compound has been demonstrated to be a sediment contaminant of concern.

2 Data

In order to derive scientifically defensible BSQC, high quality ecotoxicity data must be collected and evaluated for relevance and reliability. The UCDSM provides guidance on where to locate ecotoxicity and physical chemical property data as well as how to determine the quality of data collected. This guidance is based on that provided by TenBrook et al. (2010) for the derivation of WQC and some of that information has been repeated here for completeness. A flow chart to guide users through the process of data collection, compilation, and organization has been included (**Figure 1**).

2.1 Data collection

Data are the basis for any approach to deriving criteria. This section thoroughly describes what types of data to collect, where to search for data, and how to evaluate the data to ensure that only reliable data are used to calculate criteria. A detailed protocol for the collection and evaluation of both physicochemical and ecotoxicity data ensures that criteria will be derived from a thorough, complete and high quality data set.

2.1.1 Data sources and literature searches

Locations and sources of quality ecotoxicity and physicochemical data have been detailed previously (TenBrook et al. 2010; Fojut et al. 2013). These sources have been reviewed for their applicability to sediment toxicity data and the relevant sources have been identified (Table 1). Original data sources should always be evaluated if data are reported in compilations, handbooks or review articles, etc. In terms of a literature search, all available literature should be evaluated for a chemical of concern, tracing back to the initial synthesis or identification of the chemical. TenBrook et al. (2010) compiled a list of electronic resources including web site addresses. This list has been reviewed and updated for the purposes of deriving BSQC (Table 2).

2.1.2 Physicochemical data

A list of the physicochemical properties to be collected is provided in Table 3, which is identical to that of the UCDM (TenBrook et al. 2010). Since many jurisdictions worldwide incorporate sediment quality guidelines into an aquatic assessment framework, similar documents were evaluated to collect and gather quality data for use in the development of sediment and water quality criteria (CCME 1995; RIVM 2001). The efforts by TenBrook et al. (2009, 2010) represent a comprehensive and robust protocol based on international guidance for collecting and evaluating physicochemical data that is applicable to both aquatic and sediment criteria derivation. As a result, the UCDSM is based on the same data collection and evaluation procedures as the UCDM. The collected physicochemical data should be placed in a table and presented as part of the criteria report. If multiple similar and acceptable values for a parameter are available, the geometric mean of these values should be reported and used in any subsequent calculations involving the specific parameter (i.e., K_{OC} , K_{DOC}).

2.1.3 Ecotoxicity data

A list of the types of ecotoxicity studies to be collected for BSQC derivation is provided in Table 4 (based on TenBrook et al. 2010). As the UCDSM aims to be applicable to sediment ecosystems of the United States, the method focuses on collection of freshwater ecotoxicity data representing species found in North America. Sections 2.1.3.1 through 2.1.3.4 discuss the types of ecotoxicity data of interest, which are also listed in Table 4.

2.1.3.1 Single-species SSTTs

Single-species SSTTs establish a direct relationship between test chemical concentrations and the observed effects. These types of data could be used to directly

calculate the BSQC if ample data are available. Tests that fit in this category expose a single species in a laboratory system containing spiked sediment and overlying water. Tests that spike the overlying water instead of the sediment should be collected as supplemental data, but are not used to derive BSQC. Multispecies SSTTs are considered separately in Section 2.1.3.2. More detailed descriptions of the durations, endpoints and resulting toxicity values associated with SSTTs are given in the subsequent sections.

2.1.3.1.1 Definitions of acute and chronic exposure tests

Both acute and chronic data should be collected. In general, chronic tests expose organisms in early life stages for partial or full life cycles, while acute tests expose organisms for short periods not constituting a substantial portion of the life cycle. Definitions of acute and chronic toxicity data for sediment exposures are provided below.

Acute:

1. Invertebrate tests with exposures lasting 10-14 days including survival and growth endpoints (ASTM 2013; MacDonald and Ingersoll 2002),
2. Amphibian tests with exposures lasting 10 days (ASTM 2007).

Chronic:

Partial or full lifecycles

1. Invertebrate tests with exposures lasting 20-60 days, preferably early lifestage, including survival, growth, and possibly reproduction and emergence (ASTM 2013; MacDonald and Ingersoll 2002; RIVM 2001),
2. Any test with algae, protozoa, or plants (RIVM 2001).

Standard methods are not currently available for all taxa, so for species not included in these lists, the reader should check for new methods and if not available, use best professional judgment to determine which category a particular test fits into. Standard SSTT methods for algae and macrophytes are not currently available, although tests are being developed by researchers that may be standardized in the future (Zhang et al. 2012). Although standard method guidance is not available, the limited algal and plant SSTT data available for freshwater species may provide valuable information for assessment of herbicides or other pesticides for which algae and plants are the most sensitive species.

2.1.3.1.2 Toxicity values (regression analysis vs. hypothesis tests)

Toxicity values from regression analysis (lethal concentration and effects concentration of $x\%$ of exposed organisms; LC_x/EC_x) are recommended in the UCDSM. However, because hypothesis tests (no observed and lowest observed effect

concentration; NOEC/LOEC) have historically been used, particularly for longer-term chronic tests, these data may also be used. Point estimates that result from regression analysis should always be interpolated values, meaning the effect concentration is within the range of tested concentrations. Extrapolated point estimates that are outside of the range of tested concentrations have the potential to be model dependent, rather than dependent on observed effects (Moore and Caux 1997).

Acute toxicity values from short-term tests are typically reported as point estimates (i.e., LC/EC₅₀), which are derived from a regression equation that relates observed effects to a particular concentration. Point estimates at a 50% effect concentration from acute tests should be used to calculate species mean acute toxicity values.

Chronic toxicity values from longer tests may be reported as point estimates, typically ranging from EC₅-EC₂₀. For the UCDSM, acceptable chronic point estimates (EC_x) must be for % effects levels of $10 \leq x \leq 20$, i.e., EC₁₀ – EC₂₀ values. The 10% effect level was chosen based on an analysis of 48 pesticide toxicity data sets by Moore and Caux (1997), who found that point estimates may become model dependent at less than 10% effect and confidence intervals become excessively large at 5% effects and below. However, if low effects levels are based on interpolation rather than extrapolation, the model-dependence and large confidence intervals are less important (Stephan and Rogers 1985). The upper range of 20% effect level is based on guidance from Warne and van Dam (2008) on what is considered the range of low percent effect point estimates (i.e., 5-20%). Only low percent effect levels are included for chronic effects because an (almost) no effect level is intended (van der Hoeven et al. 1997). EC₁₀ values are the most commonly reported sublethal point estimates, but effect levels between 10-20% are acceptable for the UCDSM to include as much useful data as possible.

Chronic toxicity values are more often reported as a NOEC and LOEC from a hypothesis test. Occasionally chronic toxicity values are reported as the maximum acceptable toxicant concentration (MATC), which is calculated as the geometric mean of the LOEC and NOEC and is presumed to approximate the true no-effect concentration. MATCs are acceptable for calculating species mean chronic toxicity values when point estimates are not available. In the analysis of pesticide data sets by Moore and Caux (1997), they demonstrated that NOECs represented a reduction in control response between 10 and 30%, and most LOECs represented reductions greater than 30%. This study and others have indicated that hypothesis test results likely underestimate sublethal toxicity and are highly variable and dependent on experimental design, which is why they are not the preferred chronic toxicity values in the UCDSM.

2.1.3.1.3 Endpoints

Appropriate endpoints for BSQC derivation include those that measure survival, growth, or reproductive effects. Standard endpoints are preferred over non-standard endpoints in criteria derivation because SSTTs are still in a developmental state, even for species with a longer history of testing (i.e., midges and amphipods). Standard endpoints measured in acute tests for midge (Chironomidae) and amphipod (*Hyalella azteca*) include survival and growth measured as a change in biomass. Growth measured as a change in biomass is measured by pooling individuals in a replicate and this is the preferred method because it provides more robust statistical analysis. Biomass should be measured on a dry weight basis for or ash-free dry mass basis, depending on the organism (ASTM 2013). Growth measured as a change in weight is measured in individuals and is not preferred because the method has more variability. Depending on the organism, other measures of growth are also considered standard, such as head capsule width, length (ASTM 2013). Standard reproduction endpoints may include number of young per female, number of egg cases oviposited, number of eggs produced, number of hatched eggs, and others depending on organism and standard guidance (ASTM 2013). Other acceptable endpoints recommended in standard guidance may include emergence of adults, molting frequency, behavior, and others depending on the standard guidance for the test organism (ASTM 2013). Non-standard endpoints that would be acceptable if data for standard endpoints are not available include measures of immobility, instantaneous growth rate, as well as population level endpoints, such as r (intrinsic rate of population growth) and λ (factor by which a population increases in a given time). Other endpoints may be used in criteria derivation if those endpoints have been linked to effects on survival, growth, or reproduction. Reproductive effects can include histopathological effects on reproductive organs, spermatogenesis, fertility, pregnancy rate, number of eggs produced, egg fertility, and hatchability (RIVM 2001). Emergence, sediment avoidance, and burrowing activity are also considered relevant non-standard endpoints (ECB 2003).

2.1.3.1.4 Non-standard tests (data on bioavailability, mixtures, etc.)

While SSTTs at standard conditions are used directly in criteria calculation, SSTTs conducted using non-standard conditions should also be collected to assess the possibility of test condition effects on toxicity. This includes varied water or sediment quality parameters (e.g., temperature, pH, hardness, etc.), chemical mixtures, and bioavailability issues (e.g., DOC concentrations, OC or black carbon amendments, etc.). If a particular parameter appears to have a quantifiable effect on toxicity, compliance determination may be altered.

2.1.3.2 Multispecies ecosystem studies

Multispecies data are not used directly for criteria derivation but are collected for comparison to the criteria. These tests vary greatly and include multispecies laboratory, field, or semi-field exposures, such as mesocosms and microcosms (OECD 1995a; RIVM 2001). The resulting data may provide justification for adjustment of a final criterion if they indicate that the derived criteria will not be protective on an ecosystem scale (RIVM 2001; USEPA 1985, 2003a; Zabel and Cole 1999).

2.1.3.3 Terrestrial and human health data

Although these criteria are not intended for protection of human or terrestrial life, the risk of bioaccumulation or secondary poisoning in terrestrial organisms is assessed. Humans and terrestrial organisms may be indirectly exposed from feeding on aqueous species that have pesticide in their tissues. These data only need to be collected if the compound is likely to bioaccumulate, which can be described as having one or more of the following characteristics: $\log K_{OW} > 3$ (ECB 2003; OECD 1995a), molecular weight < 1000 (OECD 1995a), molecular diameter < 5.5 Ångstrom (OECD 1995a), molecular length < 5.5 nm (OECD 1995a), solid-water partition coefficient ($\log K_d$) > 3 , highly adsorbent (ECB 2003), belongs to a class of chemicals that are known to be bioaccumulative (ECB 2003), or if there are studies that demonstrate bioaccumulation

Dietary wildlife toxicity data are needed for bioaccumulation risk assessment, such as for mallard duck or a similar species with a significant food source in water. Long-term sublethal dietary exposures are preferable. Studies that report bioconcentration factors (BCF), bioaccumulation factors (BAF), and biomagnification factors (BMF) for various aquatic or benthic species should also be collected. For human health assessment, the United States Food and Drug Administration (USFDA) may have fish tissue action levels or dietary toxicity data available for the pesticide of interest. This assessment may have already been performed if there are existing WQC for the pesticide of interest; if this is the case, the user should search for updated toxicity values to revise the calculations, if none are available the bioaccumulation risk assessment does not need to be repeated.

2.1.3.4 Multipathway exposures

Sediment exposures are typically considered multipathway exposures because organisms can be exposed to freely dissolved chemicals from the overlying water and sediment interstitial water, as well as exposure by ingestion of sediment particles or direct contact with sediment. Incorporation of both aqueous and sediment exposure routes is inherent to SSTT protocols and is considered an advantage of using SSTT data to derive

BSQC. The principal path of exposure for hydrophobic nonionic compounds, such as pyrethroids, is considered to be via sediment interstitial water (Maund et al. 2001). Standard methods for conducting SSTT include daily feeding regimens during the exposure period; however, this may underestimate the ingestion exposure route because the food does not likely have time to equilibrate before it is ingested. If there is evidence that derived criteria are not protective, then dietary uptake studies for the particular compound and affected species are recommended to discern if exposure has been underestimated.

2.2 *Data estimation techniques*

The principal challenge in BSQC derivation is that very few usable data are available. This is of particular concern in the case of chronic toxicity data because sediment exposures are inherently chronic. This section presents approaches for estimation of acute and chronic toxicity. Some approaches such as acute-to-chronic ratios are widely used and accepted. Other approaches such as quantitative structure activity relationships (QSARs) are widely accepted for some kinds of toxicants, but are still under development for most toxicants. New approaches such as time-concentration-effect models have been validated for a large number of fish species, but are very data-intensive procedures that are not feasible with most currently available data. There are currently no available approaches to using interspecies correlations to predict sediment toxicity.

2.2.1 *QSARs*

If an experimentally determined K_{OC} is not available for a compound, it can be estimated from the K_{OW} using a quantitative structure activity relationship. The UCDSM applies the QSAR regression equations of Gerstl (1990):

Eq 8: $\log K_{OC} = a \cdot \log K_{OW} + b$

where a and b are constants for specific groups of chemicals (RIVM 2001). One reliable source of QSAR constants for pesticides is Sabljic et al. (1995); other reliable sources in the literature may be used based on professional judgment.

2.2.2 *Acute-to-chronic ratio estimation*

Due to the general lack of chronic SSTT data for pesticides, calculation of a default acute-to-chronic ratio based on SSTT data is not currently possible and as a result, the UCDSM has adopted the default ACR derived for use in the UCDM (TenBrook et al. 2010). In this report, the UCDM default ACR has been updated to include pyrethroid exposure data. A literature search was conducted to locate any ACR information derived from sediment exposures. Only one SSTT-based ACR was found in the literature. This

sediment-based ACR of approximately 7 was determined for cypermethrin and *C. dilutus* (formerly *C. tentans*; Giddings et al. 2006). The acute value in calculating this ACR was a 10-day LC₅₀ of 290 µg/g and the chronic values was a 60-day NOEC of 39 µg/g for the growth, reproduction, and percent emergence endpoints (which were all identical). These studies followed standard protocols and used approved spiking procedures that include applying the chemical to sand and evaporating off the solvent before mixing with natural sediment and equilibrating for at least 4 weeks. An alternate ACR using the 60-day MATC as the chronic value instead of the NOEC could also be calculated from the Giddings et al. (2006) data set. In this calculation, using the 60-d MATC for growth or reproduction of 57 µg/g as the chronic value would result in an ACR of approximately 5. An ACR of approximately 6 was calculated for *H. azteca* based on a USEPA database that included 1,657 field-collected samples of matching sediment toxicity and chemistry data for a variety of sediment contaminants, not solely pesticides (Ingersoll et al. 2001). Until more paired acute and chronic SSTT data are available for a variety of species, ACRs based on water-only exposure are recommended for estimating chronic values from acute data. The rationale behind this decision is that if benthic and water-column species have similar species sensitivity distributions, the distribution of acute and chronic effects concentrations in the organisms will also be similar, regardless of exposure media, which is consistent with the EqP approach (Di Toro et al. 2002).

2.3 Data evaluation

Once data are collected for the pesticide of interest, the data or studies are evaluated to determine if they follow standard protocols and meet quality guidelines. For physicochemical data, the preferred methods of data collection are given in section 2.3.1 to prioritize these data. Evaluation of single-species ecotoxicity data involves several steps described in section 2.3.2: 1) determine if data are relevant based on key study parameters, 2) if data pass relevance threshold, determine if data are reliable based on documentation and acceptability of test parameters, 3) calculate a study score for relevance and reliability, and 4) summarize the study parameters and scores in a table to document the evaluation process. The relevance and reliability scores are used to give the final rating (presented in Table 7 with individual scores presented in Table 8-Table 12). Toxicity values with a high final rating make up the acceptable data set used to calculate criteria, while toxicity values with lower ratings are considered supplemental and may be used to compare to criteria in the case that they represent species not included in the acceptable data set. Data with the lowest ratings are not used in any aspect of criteria derivation. Multiple species laboratory studies, mesocosm/microcosm studies, field or wildlife studies also have specialized evaluation tables because these types of data are used to compare to the criteria derived from single-species studies if they have moderate to high ratings for relevance and reliability.

2.3.1 Physicochemical data evaluation

As discussed in Phase I, it is extremely important to have accurate physicochemical data, especially K_{OW} , K_{OC} or K_{DOC} values, for highly insoluble chemicals (Fojut et al. 2011). These values are difficult to determine experimentally and K_{OC} and K_{DOC} will vary depending on the solid, thus, values in the literature can vary by orders of magnitude for such compounds. To address this issue, specific guidance for selection of high quality and reliable partition coefficients is provided by the USEPA (Di Toro et al. 2002) and The Netherlands (RIVM 2001). The USEPA EqP method recommends using newer experimental methodologies to determine the K_{OW} , such as the slow stir method (de Bruijn et al. 1989) and the generator column method (Woodburn et al. 1984). Experimentally determined values using these methods should take precedent over values based on other methodologies (Table 5) or estimated K_{OW} s based on QSARs.

Reliable physicochemical methods for generating data using acceptable standard protocols are summarized in Table 5 (first described by TenBrook et al. 2010; based on USEPA 2003a and RIVM 2001). The UCDM guidance for prioritizing recommended methods to determine K_{OW} s is given in Table 6, as well as expanded guidance for K_{OW} method selection for compounds with $\log K_{OW}$ greater than 6. Due to the highly insoluble nature of such compounds, the slow stir method is likely to produce an emulsion, leading to erroneous results. For this reason, Laskowski (2002) recommends that the generator column methods are used to determine pyrethroid K_{OW} s rather than the slow stir method, and this guidance also applies to other compounds with $\log K_{OW}$ greater than 6. The method most appropriate for the experimental determination of reliable K_{OW} s depends on the likely range of a compound's K_{OW} .

It should be noted that for physicochemical properties that are affected by temperature, it is important that the parameters were determined at relevant temperatures. If not, the parameters should be adjusted accordingly, as detailed in TenBrook et al. (2010). Physicochemical data that are not verifiable should not be used for criteria derivation and values taken from handbooks should be used with caution.

As stated in section 2.1.2, the collected physicochemical data should be placed in a table and presented as part of the criteria report. If multiple similar and acceptable values for a parameter are available, the geometric mean of these values should be reported and used in any subsequent calculations involving the specific parameter (e.g., K_{OC} , K_{DOC}), except when a partition coefficient was determined in the same study that the ecotoxicity information is reported.

It is important that quality physicochemical data are collected and systematically evaluated for reliability whether or not they are directly used in the calculation of BSQC. The acceptable methods for determination of physicochemical properties defined in the

UCDM are also adopted in the UCDSM. There are both nationally and internationally recognized protocols for the measurement of physicochemical properties and these standards are used to evaluate available data (OECD 1995b; RIVM 2001; USEPA 1985, 2003a). Yet, as discussed above, with partitioning coefficients of highly insoluble compounds, the use of standard protocols may not remove the large variation in published values resulting from the analytical challenges inherent to working with compounds with extremely low water solubilities.

2.3.2 Ecotoxicity data evaluation

The UCDM protocol developed to rank the relevance and reliability of an ecotoxicity study was based on the widely accepted ECOTOX (2006) rating system. The UCDSM further expands this protocol to incorporate data quality indicators for sediment toxicity studies. A systematic scoring approach to determine relevance and reliability of ecotoxicity data was developed so that data evaluation is more objective, although best professional judgment and knowledge of standard protocols are inherent to the process. The inclusion or exclusion of data can have a large effect on the resulting criteria, so the goal was to fully document the data evaluation process and include the documentation in the final criteria reports so that the process is transparent and available for review. The highest quality data are relevant to the purpose of this method, based on standard methods of testing, well-documented, and meet the acceptability criteria of the method employed. The meaning and intent behind the relevance and reliability parameters are described in more detail below. The relevance and reliability of a study are scored numerically based on acceptable design and execution of the study, which is based on documentation of study parameters. These numeric scores are translated into ratings as specified in Table 7. Studies with the highest scores are rated relevant and reliable (RR) are used directly for criteria derivation. Studies with moderate scores for either category are rated less relevant or less reliable (LL, RL, LR) and are used as supplemental data to compare to derived criteria (section 5). Studies with low scores for either category are rated as not relevant or not reliable (N, LN, RN) are not used for any purpose in the UCDSM.

2.3.2.1 Relevance

For single-species ecotoxicity studies, relevance corresponds to those studies that by design test a single species with a single pure chemical following standard test protocols. The relevance scoring is designed to require six key study parameters for a study to be considered relevant (R) for the purpose of this method, and therefore acceptable data for use in criteria derivation. The relevance parameters and associated point values are presented in Table 8 (adapted from TenBrook et al. 2010). The six key parameters are: 1) the endpoint is related to survival, growth, or reproduction; 2) it is a

freshwater spiked-sediment toxicity test; 3) the test chemical is >80% purity; 4) the test species is from a family found in North America; 5) a toxicity value is reported or one is calculable based on raw data (censored toxicity values are not considered relevant) and the toxicity value accounts for bioavailability¹; and 6) an acceptable control response is reported. If one or two of these six parameters is lacking, the data are less relevant (L) and are considered supplemental data that can be used to compare to criteria if they represent a species not included in the acceptable data set. If three or more parameters are lacking, the data are not relevant (N) and cannot be used for any aspect of criteria derivation or comparison. Each of the six parameters is worth 15 points of a total of 100 points. There are also 10 points given for citing and following a standard test protocol. This study parameter has a lower point value because many studies do follow standard protocols, but may not cite them, and if the study details are reported and are acceptable, then the quality of the data can be evaluated. These study parameters are considered in the reliability evaluation described next. Data with a relevance score of 90-100 are relevant (R) and are included in the acceptable data set, data with a relevance score of 70-89 are less relevant (L) and are included in the supplemental data set, and finally, data with a relevance score of 0-69 are not relevant (N) are not considered further. These scores are also given in Table 7. Studies that rate as relevant or less relevant are evaluated for reliability (Table 9, Table 10), while studies that rate as not relevant are not evaluated for reliability. The cutoff scores for the different relevance ratings were developed as part of the UCDM

2.3.2.2 Reliability

Reliability is divided into two main categories, documentation and acceptability. Adequate documentation of study design, test conditions, and data analysis methods improves the reliability of a sediment toxicity test and fitness for use in BSQC derivation. A documentation rating system for SSTTs, revised from the UCDM rating system for aquatic laboratory studies, is presented in Table 9. Sediment properties (particle size distribution, total organic carbon), sediment spike method, and spike equilibration time are now included as documentation parameters (ASTM 2013; USEPA 2001; USEPA1996c). Point values for the documentation of overlying water quality parameter values have been lowered by approximately half compared to the dilution water quality values in the UCDM (TenBrook et al. 2010). These points have been redistributed to the listed sediment characteristics in Table 9.

¹ As described in section 1.2.3, a toxicity value accounts for bioavailability if it is: 1) a whole sediment concentration normalized to the organic carbon content, 2) a whole interstitial water concentration that is normalized to the dissolved organic carbon content, 3) an interstitial water concentration estimated via solid-phase microextraction or another non-depleting equilibrium-based technique, or 4) an interstitial water concentration estimated via Tenax ® or another depletion technique.

The reliability of a SSTT depends not only on appropriate documentation, but acceptability of the test conditions in relation to an accepted standard. The acceptability rating system for single species SSTTs is presented in Table 10. This table is based on the UCDSM acceptability rating system for use in the evaluation of single-species aquatic laboratory data (TenBrook et al. 2010). The UCDSM acceptability rating system includes the following elements, which are related directly to sediment toxicity studies: sediment spiking method, solvent carrier amount and spike equilibration time. Recommended protocols for sediment spiking are provided by the USEPA (2001) and the CCME (1995) and are described in Appendix A. Briefly, regardless of the spiking technique, replicate subsamples of the spiked sediment should be analyzed to determine homogeneous mixing and confirmation of spike level. Wet sediment spiking is preferred over dry sediment spiking and the jar rolling method is considered more suitable for spiking large batches of sediment compared to hand mixing. It is recommended that spiked sediments are stored for at least one month before use in toxicity testing to ensure that the sediment and interstitial water are in chemical equilibrium, unless other information indicates otherwise, especially for highly hydrophobic compounds. It is noted that direct addition of solvent carrier to the sediment should be avoided and a shell coating method should be employed instead. The solvent carrier containing the chemical is added to sand or glassware surface and the solvent is allowed to evaporate completely before mixing with wet sediment. This procedure ensures that the solvent does not change the physicochemical properties of the sediment. Due to the importance of these factors in their potential to affect the validity of the sediment toxicity test results, these elements have been added to the acceptability rating table (Table 10).

In order to aid the user in determining whether a given test parameter is acceptable according to standard methods, a summary of acceptable test conditions have been compiled in Appendix A. Included is a summary table of the USEPA test conditions and acceptability requirements (or guidance) for *H. azteca* and *C. dilutus* given in OPPTS 850.1735 (USEPA 1996c). A summary table of the ASTM E 1706-05 test conditions and acceptability requirements (or guidance) for *H. azteca*, *C. dilutus*, *Chironomus riparius*, *Daphnia magna*, *Ceriodaphnia dubia*, *Hexagenia* spp., *Tubifex tubifex* and *Diporeia* spp. for various exposure durations is also included (ASTM 2013). A summary table of the EPA-823-B-01-002 protocol for sediment spiking and interstitial water handling procedures (USEPA 2001) is also included. Standard protocols are revised over time as new information becomes available; for this reason, the guidance in Appendix A is given as a snapshot in time of the current acceptable protocols, but is not meant to be a replacement for knowledge and familiarity with standard methods and updates and revisions that may alter how tests are performed and evaluated. There are currently efforts underway to update spiked-sediment toxicity test protocols for midges and amphipods and to finalize tests with longer exposure durations that include reproductive and other sublethal

endpoints. The most current standard test protocols should be taken into account for evaluating test reliability as the guidance in Appendix A will become outdated.

Although not directly included in the sediment or water criteria calculation, ecotoxicity studies based on multiple species laboratory studies, mesocosm/microcosm studies, field or wildlife investigations are still relevant for comparison to the derived criteria. It is important to evaluate the reliability of these types of data for acceptability and adequate documentation of the study, although there is a lack of standardized protocols for these tests. The UCDM rating scales developed for acceptability of these types of data are also considered relevant for evaluating sediment associated contaminants. A documentation and acceptability rating table for aquatic outdoor field data and indoor model ecosystems is presented in Table 11 (first adapted from ECOTOX 2006 by TenBrook et al. 2010). An acceptability rating table was also developed for terrestrial ecotoxicity data generated in the laboratory or field environment (Table 12, based on TenBrook et al. 2010). Table 11 and Table 12 have been updated to incorporate acceptability relevant to sediment.

2.3.2.3 Data summaries of ecotoxicity data

The UCDM details a procedure for systematically rating the relevance and reliability of an ecotoxicity study for use in the derivation of WQC (TenBrook et al. 2010). The UCDM expanded on the Dutch guidance in which components of an ecotoxicity study must be identified in order to evaluate the quality of a wide range of studies fairly (RIVM 2001). It is likely that the WQC and BSQC derivation methods will be used simultaneously or sequentially, and therefore it is beneficial to the user to follow a similar approach for summarizing and evaluating ecotoxicity data. The UCDSM has thus adapted the UCDM data summary sheet for use with sediment exposures. The list of components to be documented for an ecotoxicity study used to derive or support BSQC is presented in Table 14. Additions made to the UCDM data summary sheet include: documentation of the physicochemical properties of the sediment used in the toxicity test, if the sediment is formulated or natural, as well as information on the sediment spiking procedure, spike equilibration time and the sediment-to-solution ratio used in the toxicity test (Table 14). Documentation requirements of the procedures used for extracting interstitial water from sediment, interstitial water chemical extraction and instrumental analysis have also been added. Both water concentrations and sediment concentrations should be recorded, if these are reported in the study.

The intent of the documentation sheet is to extract relevant information while reviewing an ecotoxicity study. The documentation sheet should be completed with enough detail to provide support for assigning a numerical score for the overall relevance and reliability. To aid in the completion of the documentation sheet and rating of studies, a guidance document is presented in [Appendix A](#) that provides a summary of acceptable

test conditions gathered from standard methods. It should be noted that this is to serve as a guide and does not replace or take precedent over the original source information.

2.4 *Organization of data*

Single-species SSTT data should be separated into acute and chronic data, and each set should be summarized in a separate table. Within each table or in separate tables, data should also be separated into categories for different types of concentrations (i.e., OC-normalized sediment concentrations, DOC-normalized interstitial water concentrations, freely dissolved concentrations measured via passive sampling devices, or bioaccessible concentrations measured via Tenax). For ease of viewing, we also recommend summarizing the other types of available toxicity values in data tables, e.g., wildlife toxicity values, ecosystem-level studies, BCFs, etc. Recommended headings to include in the single-species data tables are: species name (binomial nomenclature); common name (optional); family name; life-stage, age, or size of organism; test endpoint; test type (i.e., static, static renewal, flow-through); exposure duration; test temperature; toxicity value(s) (e.g., LC₅₀, EC_x, NOEC, LOEC, MATC); OC content or % OC; the study reference or citation; and if applicable, the overall rating of the study (i.e., RL, LR, or LL) and the reason the study was excluded or rated lower than RR.

2.5 *Data prioritization*

For data that are rated relevant and reliable (i.e., RR, according to Table 7), the toxicity values are prioritized and combined to result in one toxicity value for each species, which will subsequently be used for criteria calculation. We recommend that this prioritization step should follow the data organization step described in section 2.4 because it only applies to toxicity values rated RR and having the data in a spreadsheet table allows for easy calculations and revisions to the table. The prioritization process should be done separately for the acceptable acute and chronic data sets to arrive at a single species mean acute value (SMAV) and species mean chronic value (SMCV) for each species in the respective data sets. The UCDSM data prioritization instructions to compute SMAVs and SMCVs have been adopted by the UCDSM and are listed here, with some additions specific to sediment:

- a) If a NOEC is not explicitly reported in chronic toxicity studies, but statistical analysis was done, the NOEC may be determined as the highest reported concentration not statistically different from the control ($p < 0.05$) that is below any other concentration producing significant adverse effects (ASTM 2013, RIVM 2001); the NOEC is not used in criteria derivation, but is needed for calculation of the MATC;

- b) Similarly, if a LOEC is not explicitly reported in chronic toxicity studies, it may be determined as the lowest reported concentration that is statistically different from the control ($p < 0.05$) that is above any other concentration not producing statistically significant adverse effects (ASTM 2013); the LOEC is not used in criteria derivation, but is needed for calculation of the MATC;
- c) If a MATC is not reported, it may be calculated as the geometric mean of the NOEC and LOEC;
- d) If no toxicity values were reported, but raw data are available, calculate toxicity values using appropriate statistical methods; calculate point estimates (EC_{10} or EC/LC_{50}) if the experimental design is appropriate, particularly the dilution factor must be ≥ 0.3 (ECB 2003);
- e) If a MATC is expressed as a range of values, recalculate the MATC as the geometric mean of the high and low values (RIVM 2001);
- f) If toxicity values are reported for the sediment matrix on a dry weight basis (e.g., $\mu\text{g}/\text{kg DW}$) and the organic carbon content of the sediment is also reported, the toxicity values should be normalized to the organic carbon content using Eq 1.
- g) If toxicity values are reported as an aqueous concentration in whole interstitial water and the fraction of DOC is also reported, the toxicity values should be normalized to the DOC content using Eq 4.
- h) Calculate SMAVs/SMCVs as the geometric mean of toxicity values from one or more acceptable tests with the same endpoints (ANZECC and ARMCANZ 2000; ECB 2003; OECD 1995a; RIVM 2001; USEPA 1985; 2003a);
- i) If data are available for life stages that are at least a factor of two more resistant than another life stage for the same species, use the data for the most sensitive life stage to calculate the SMAV or SMCV because the goal is to protect all life stages (RIVM 2001; USEPA 1985; 2003a);
- j) If data are available for multiple endpoints for one species, use the data for the most sensitive standard endpoint (ANZECC and ARMCANZ 2000; ECB 2003; OECD 1995a; RIVM 2001); standard endpoints are described in Section 2.1.3.1.3. Non-standard endpoints that measure survival, growth, or reproduction (e.g., instantaneous growth rate) can be used if standard endpoints are not available for a given species. If there are multiple endpoints that are equally sensitive, note all endpoints, but use only one value for criteria calculation;
- k) If differences between tests for the same species/endpoints are justified or known, then data may be grouped according to appropriate factors (e.g., pH or

temperature; ECB 2003). Selection of the appropriate value to use in criteria derivation should be based on standard test parameters. Tests conducted under non-standard conditions (vs. standard conditions as defined in standard test methods) may be used to derive quantitative relationships between those conditions and toxicity (as in USEPA 1985; 2003a). If such a relationship is established then toxicity values derived under non-standard conditions may be translated to standard conditions and added to the criteria derivation data set. If no quantitative relationship can be derived then tests conducted under non-standard conditions should not be used for criteria derivation, but may be used as supporting information;

- l) If data are available for multiple time points from crustacean or insect acute toxicity studies, use the longest exposure time (i.e., 10-d tests are preferred over tests of < 10-d for acute tests);
- m) Further prioritization may be needed in the course of SSD analysis. If data cannot be described by or fit to a distribution, then the set should be examined for outliers and/or bimodality as described in section 3.4.2.3. If data are bimodally-distributed (as determined visually), use only the lower of the two groups for criteria derivation (ANZECC and ARMCANZ 2000); the effects of data exclusions on the criteria must be explored and explained (ECB 2003).

2.6 *Graphical presentation of data*

If five or more toxicity values are available, it can be useful to plot the data to visually assess potential trends. As instructed in TenBrook et al. (2010), construct a histogram of the frequency distribution and examine the distribution for multimodality or outliers. Double-check toxicity values for errors, especially toxicity values that appear to be outliers. A multi-modal distribution may be more easily seen when graphing a cumulative frequency distribution. This can be done as part of the SSD fitting described in section 3.4, or a graph of cumulative frequency vs. log concentration can be constructed using Eq 9 below. If a distribution is used to calculate a final criterion, a graph of the distribution plotted with the actual toxicity values should be included in the final report.

Eq 9:
$$\text{Cumulative frequency} = (\text{rank}-0.5)/n$$

where

rank = position in set of ordered data (ranked from lowest to highest);

n = sample number.

Once data are collected, evaluated, selected and prioritized, criteria derivation may begin.

3 Criteria calculation

The criteria calculation procedure will depend on the number and type of data available. A flow chart of the sediment criteria derivation process is provided (**Figure 2**). A species sensitivity distribution is used to derive the criteria (section 3.4) if the data set contains the five required taxa (section 3.4.1.1). If less than the five taxa requirements are available, an assessment factor is used (section 3.5).

3.1 Defining numeric criteria

The different terms, definitions, and purposes of SQC used throughout the world were summarized in the Phase I report (Fojut et al. 2011). Numeric criteria in the UCDSM follow the same definition as given in the UCDM (TenBrook et al. 2009): science-based values, which are intended to protect aquatic life from adverse effects of pesticides, without consideration of defined water body uses, societal values, economics, or other non-scientific considerations. The UCDSM lays out a methodology to derive numeric criteria for pesticides associated with sediments according to this definition, which also corresponds to the USEPA numeric criterion definition.

Methods are presented below for derivation of numeric criteria from data sets of any size. The limitations of the derived values are discussed qualitatively, and, where possible, quantitatively, but no categorization is made as to what the values should be used for, as that decision lies in the realm of policy.

3.2 Protection goal

The protection goal of the UCDSM numeric BSQC is to protect all species in the ecosystem. Protection at the species-level is the goal because the disappearance of a single species could lead to the unraveling of community structure at the ecosystem level due to complex interactions among species (TenBrook et al. 2009). The strategy to achieve this goal is the same as that given in the UCDM, which is to extrapolate from available pesticide toxicity data for a limited number of species to a concentration that should not produce detrimental physiological effects in aquatic life. The protection goal and strategy should lead to achievement of the narrative objective of the CRWQCB-CVR, which is to maintain waters free of “toxic substances in concentrations that produce detrimental physiological responses in plant, animal, or aquatic life” (CRWQCB-CVR 2011). The development of the UCDM and the UCDSM focused on the Sacramento and

San Joaquin River watersheds of the California Central Valley and this ecosystem is specifically discussed in several instances. However, the UCDSM is generally appropriate for any freshwater ecosystem in the United States. Additionally, simple modifications could be made to adapt this method for saltwater criteria or other geographic areas.

3.3 Toxicity values

The UCDSM requires that separate acute and chronic criteria are derived. Acute criteria are derived from short-term LC₅₀ or EC₅₀ data, using either the SSD methodology (section 3.4) or the AF methodology (section 3.5), depending on the quantity and diversity of acute data available. For chronic criteria derivation, the procedure applied also depends on quantity and diversity of data available. The SSD procedure (section 3.4) is used if sufficient chronic data (EC_xs or MATCs) are available (see section 3.4.1.1), if not, the acute value is used with an acute-to-chronic ratio to calculate a chronic criterion (section 3.6). The definitions of acute and chronic exposures and the use of regression point estimates and hypothesis test results in criteria derivation were described in sections 2.1.3.1.1 and 2.1.3.1.2. Figure 2 is a flow-chart that illustrates how to proceed through the criteria calculation process depending on the quantity and diversity of data available.

3.4 SSD procedure and rationale

The species sensitivity distribution procedure of the UCDSM was also adopted for the UCDSM. The rationale behind the SSD procedure is presented in section 3.4.1, and the procedure is given in section 3.4.2. A SSD can be reliably fit to a toxicity data set if there are at least five data points, each representing an important taxon, given in section 3.4.1.1. Currently very few sediment toxicity data are available and the available data are primarily for two taxa (midge and amphipod). The SSD approach is therefore not likely to be used for calculating BSQC until there is a proliferation of high quality SSTT data and standard test protocols are developed for additional taxa. In the case that data for the five required taxa are met for a pesticide, instructions are given here for criteria calculation via SSD. Prior to fitting a SSD, toxicity data should be organized into separate categories (e.g., acute, chronic), as described in section 2.4, and a separate SSD should be fit to each data category that satisfies the taxa requirements. Either the Burr III distribution (3.4.2.1) or the log-logistic distribution (3.4.2.2) is fit to the data, depending on the number of species mean toxicity values in a given category.

3.4.1 SSD rationale

3.4.1.1 SSD ecotoxicity taxa requirements

The UCDSM requires data for five taxa to use a SSD, similar to the requirements of the UCDM (TenBrook et al. 2010). The UCDSM taxa requirements differ from the UCDM, however, in that UCDSM requirements emphasize more benthic organisms, representing various important taxa and varied habitats and feeding habits.

UCDSM SSD ecotoxicity taxa requirements:

- a) An epibenthic crustacean (e.g., *H. azteca*);
- b) A benthic insect (e.g., Chironomids);
- c) An infaunal invertebrate (e.g., *Hexagenia* spp., *Diporeia* spp.);
- d) A mollusk/amphibian/fish/other unrepresented phylum (e.g., *Rana* spp., oligochaetes, etc.);
- e) A benthic invertebrate from an unrepresented family.

H. azteca and *C. dilutus* (formerly *C. tentans*) are recommended as test organisms by MacDonald and Ingersoll (2002) because of the relative sensitivity of these organisms to contaminants, contact with sediment, ease of culture in the laboratory, inter-laboratory comparisons, tolerance of varying sediment physicochemical characteristics, and similarity to responses of natural benthos populations. The USEPA (2000) opted not to develop standard sediment test methods for mayflies, oligochaetes, other amphipods and midges because these did not meet all the criteria that *H. azteca* and *C. dilutus* met, although guidance on conducting tests with these species is available from ASTM (2013).

3.4.1.2 Percentile cutoff

To derive criteria using a SSD, a percentile from the distribution must be selected for use in the calculation. In selecting a percentile cutoff, the goal is to estimate a no-effect concentration. Thus, any percentile may be chosen as long as it can be validated against knowledge and understanding of ecosystem structure and function, as noted by Solomon et al. (2001). A common misconception regarding the percentile cutoff is that species below this point on the distribution will be harmed, while those above it will be protected, if the concentration remains below that of the selected percentile value. This interpretation is incorrect because the distribution is not likely a true representation of the sensitivity of all species in an ecosystem, but rather is a prediction based on available test species, and the percentile cutoff should be adjusted based on the certainty and reliability of a particular SSD (van Straalen and van Leeuwen 2002).

The median 5th percentile is recommended in many criteria derivation methodologies (ECB 2003; RIVM 2001, 2004; USEPA 1985, 2003a) and has been demonstrated to be a good predictor of no-effect concentrations in field studies (Emans et al. 1993; Hose and van den Brink 2004; Maltby et al. 2005; Okkerman et al. 1993; USEPA 1991; Versteeg et al. 1999). There is evidence that in some cases, the median 5th percentile is not protective of sensitive species. For example, Zischke et al. (1985) found that a laboratory-derived water quality criterion concentration of pentachlorophenol was not protective of invertebrates and fish in outdoor experimental channels. In addition, Maltby et al. (2005) determined that the median 5th percentile was generally protective of aquatic model ecosystems when there was a single application of an insecticide, but not when there were continuous or multiple applications. In these cases the estimate of the lower 95% confidence interval of the 5th percentile was protective. While using the lower estimates from the distribution may guarantee protection, the high uncertainty in the extreme tail of the distribution can actually contribute more to the resulting criterion than the data.

In order to give environmental managers more information regarding the uncertainty in these predictions, we recommend calculating the following estimates: concentrations that will protect 95% of species with 50% confidence (95:50 or median 5th percentile), 95% of species with 95% confidence (95:95), 99% of species with 50% confidence (99:50 or median 1st percentile), and 99% of species with 95% confidence (99:95). The estimates are referred to as either acute values or chronic values, depending on which data set was used. To calculate criteria, the median (50% confidence) 5th percentile is initially recommended because it is the most statistically robust. If there is evidence that the median 5th percentile may not be sufficiently protective based on data for sensitive, threatened or endangered species, or from multi-species, ecosystem-level tests, then the next lowest estimate may be used for criteria calculation, according to professional judgment.

3.4.2 SSD procedure

3.4.2.1 Burr Type III SSD (>8 toxicity values)

When the five SSD ecotoxicity taxa requirements are satisfied and there are >8 toxicity values available, the Burr Type III SSD should be fit to the data by following the SSD procedure described in ANZECC and ARMCANZ (2000). The Burr Type III distribution consists of three related distributions (Burr III, reciprocal Weibull, and reciprocal Pareto; Burr 1942). Any statistical package that is capable can be used to fit the distribution to the data (e.g., BurrliOZ v. 1.0.13 (CSIRO 2001). The software will state which of the three distributions best fit the data. The median 1st and 5th percentile

values should be generated by the statistical software, or they can be calculated using the following equations (values should be recorded to three significant figures):

For Burr III

$$\text{Eq 10: } PC(q) = \frac{b}{\left[\left(\frac{1}{1-q} \right)^{\frac{1}{k}} - 1 \right]^{\frac{1}{c}}}$$

where

$PC(q)$ = the protecting concentration that will protect $q\%$ of species;
 q = percentage of species to protect (e.g., to calculate the 5th percentile set $q = 95$);
 b , c and k are fit parameters.

For reciprocal Weibull (for cases when $k \rightarrow \infty$)

$$\text{Eq 11: } PC(q) = (-\alpha / \ln(1-q))^{\frac{1}{\beta}}$$

where α and β are fit parameters.

For reciprocal Pareto (for cases when $c \rightarrow \infty$)

$$\text{Eq 12: } PC(q) = x_0(1-q)^{\frac{1}{\theta}}$$

where x_0 and θ are fit parameters.

The lower 95% confidence interval should also be calculated using the BurrliOZ program (CSIRO 2001) or another capable statistical package. If the statistical package in use is not capable of generating the lower 95% confidence interval for the 1st and 5th percentile values, the following bootstrapping technique (CSIRO 2001) can be used:

- a) Resample the original data set, with replacement, to create a new data set the same size as the original set and calculate 1st and 5th percentile values from the new data set. Repeat this resampling and recalculation procedure 200-1000 times.

At least 501 resamplings are recommended (ANZECC and ARMCANZ 2000); fewer will give a less certain estimate; more will give a more certain estimate, but will require more calculation time;

- b) Order the bootstrapped estimates from lowest to highest (separately for the 1st and 5th percentile SSD estimates) and select the 5th percentile value; this represents the lower 95% confidence limit estimate of the 1st or 5th percentile of the SSD.

After the 1st and 5th percentile values and their associated confidence intervals have been generated, the fit test described below in section 3.4.2.3 should be performed to check for goodness of fit of the SSD to the data. The BurrliOZ software comes with a caution that for data sets of eight or fewer toxicity values there will be great uncertainty in the calculated values. The software authors provide a procedure to fit a log-logistic SSD to the data instead of the Burr Type III SSD in such cases. This procedure has been modified for the UCDM and UCDSM and is presented in section 3.4.2.2.

3.4.2.2 Log-logistic SSD (5-8 toxicity values)

When the five SSD ecotoxicity taxa requirements are satisfied and there are 5-8 toxicity values available, the log-logistic SSD should be fitted to the data. The log-logistic SSD has a lower likelihood of over-fitting data sets of this size because there are fewer fitting parameters in the cumulative distribution function. It should be noted that the log-logistic procedure given below is different than the procedure given in the BurrliOZ software (see readme file, Appendix C), and the BurrliOZ procedure should not be used.

A log-logistic SSD can be fit to the data using any statistics package capable of the analysis, e.g., ETX v.1.3 (Aldenberg 1993), which is documented in Appendix C and can be obtained from RIVM by contacting info@rivm.nl. Once the fit parameters (α and β) have been determined, the following equation can be used to determine median (50% confidence interval) 1st and 5th percentile values:

Eq 13:
$$p = 100 / (1 + \exp(-[\ln(x) - \alpha] / \beta))$$

where

p = percentage of species unaffected at x ; set $p = 1$ to calculate the 1st percentile;

$p = 5$ for the 5th percentile;

x = toxicity value (concentration threshold) at p ;

α = sample mean (of $\ln(x)$);

$\beta = k_L * S_n / C_5$;

and

k_L = extrapolation constant; dependent on sample size; selected for either median or lower 95% confidence interval estimate (see Table 23 of UCDM, Fojut et al.2011; TenBrook et al. 2009);
 s_n = sample standard deviation (of $\ln(x)$); n = sample size;
 C_5 = constant = 2.9444.

Note: some software, such as ETX v. 1.3, uses $\log(x)$ in place of $\ln(x)$ in Eq 13 and to calculate α and β . If using α and β calculated from $\log(x)$, be sure also to use $\log(x)$ in the Eq 13 instead of $\ln(x)$.

The lower 95% confidence intervals of the 1st and 5th percentiles can be calculated using a capable statistics package. If the ETX v. 1.3 software (Aldenberg 1993) is used, it is capable of calculating the lower 95% confidence interval for the 5th percentile, but not for the 1st percentile. The latter estimate may be omitted for the log-logistic SSD because the other three estimates are likely to be more useful because they have less uncertainty. Alternatively, the bootstrapping technique described for the Burr Type III SSD (section 3.4.2) can be used. After the 1st and 5th percentile values and their associated confidence intervals have been generated, the fit test described below in section 3.4.2.3 should be performed to check for goodness of fit of the SSD to the data.

3.4.2.3 Fit test

The following procedure checks that either the Burr Type III or log-logistic SSD fits the toxicity data. The BurrliOZ software chooses the best fitting SSD with a goodness of fit based on maximum likelihood estimation, whereas the fit test is a different approach based on cross-validation. The general process of the fit test is that a data point (x_i) is omitted from the data set and a distribution ($F_{\cdot i}$) is fit to that data set. Then the probability of the omitted point is estimated with that distribution ($F_{\cdot i}(x_i)$). This is done for each data point and the combined results for all points in the data set are examined for a significant lack of fit using Fisher's combined test, outlined below.

The distribution will have been fitted based on a sample of n species, each having one mean toxicity value (e.g., LC_{50} , EC_x , MATC), which can also be referred to as x values (i.e., plotting y vs. x). The type of SSD that was fit to the original data set (either the log-logistic or Burr Type III) should be refit to the data set that *omits* the point x_i . The resulting distribution function is called $F_{\cdot i}$. Then the probability of the omitted point (x_i) within this distribution is calculated by solving for $F_{\cdot i}(x_i)$, which is also called the y value. In the BurrliOZ software, after the distribution is refit, the results window allows entry of a concentration (x_i) and then provides the corresponding percentile, solving for $F_{\cdot i}(x_i)$. There is a similar option in the ETX v.1.3 software. The distribution $F_{\cdot i}(x_i)$ should be determined for each data point, which will result in n probabilities (y values).

Then let

$$\text{Eq 14: } p_i = 2 * \min(F_{-i}(x_i), 1 - F_{-i}(x_i))$$

where

p_i = p-value, i.e., the probability of obtaining a test statistic at least as extreme as the one that was actually observed, assuming the null hypothesis is true;

“min” indicates using the minimum of either $F_{-i}(x_i)$ or $1 - F_{-i}(x_i)$.

Next, apply Fisher’s combined test and calculate a chi-squared statistic of the form

$$\text{Eq 15: } \chi^2_{2n} \sim -2 \sum i \ln(p_i)$$

If any one of the data points is insufficiently fitted, then the test is capable of rejecting the hypothesis that the data point comes from the fitted distribution. Once all of the p_i values have been calculated, the chi-squared statistic (χ^2_{2n}) can be calculated. In Excel the significance of chi-squared statistic is calculated with the command:

$$\text{Eq 16: } \text{CHIDIST, with the fields } (x, df)$$

where

$$x = -2 \sum i \ln(p_i);$$

df = the degrees of freedom or n , the number of p_i values.

The closer the resulting value for χ^2_{2n} is to 1, the better the fit. When the result for $\chi^2_{2n} < 0.05$, there is a significant effect from the substitution and a 95% probability of a significant lack of fit. If there is a significant lack of fit, then the data should be critically examined and checked for multi-modality as described in section 3.4.2.3.1. If there is not a significant lack of fit ($\chi^2_{2n} \geq 0.05$), proceed to section 3.7 for criteria calculation.

3.4.2.3.1 Reasons for a significant lack of fit

If there is a significant lack of fit of the SSD to the data according to the fit test, then the data should be examined for multi-modality, outliers, and errors as outlined in the steps below. If the data set is altered based on this examination, criteria should be re-calculated using the appropriate procedure for the revised data set.

- a) Double-check the toxicity values to be sure they are not mistakes (i.e., typographical or transcriptional errors) and review the original studies again to be sure that all test conditions were appropriate. The need to remove outliers is considerably reduced using the Burr Type III distribution with the BurrliOZ software (CSIRO Biometrics, Campbell et al. 2000). If a fit cannot be obtained with a larger data set, critical examination of data is emphasized as any one point outlier that causes the SSD to not fit may represent an extreme difference that is erroneous (i.e., above the water solubility of the compound or below the analytical detection limit). If errors are found, remove the erroneous data from the data set and re-calculate the criteria using the remaining data. Removal of data from the SSD could also be justified if there is supporting information as to why the point(s) does not belong in the same SSD as the remaining data (e.g., a resistant strain of the species). This approach is reasonable because, as with all criteria derived from this methodology, criteria will be evaluated to determine if they will provide adequate protection (section 5).
- b) Examine data for multi-modality. Identifying multi-modality is only possible with a large enough data set that each mode could be characterized by multiple toxicity values. It is extremely unlikely that any SSTT data set would have enough data to identify multi-modality because SSTT data is so scarce and largely is only available for two taxa (midge and amphipod). However, in the case that a large data set ($n > 8$) were available a procedure for examining multi-modality is provided:

If a SSD cannot be fit and visual inspection indicates that the SSD is multi-modal, meaning the distribution has multiple peaks in the cumulative frequency distribution, and this occurs in a justifiable manner (such as by taxa), divide the data into subsets (such as vertebrates and invertebrates) and use the subset containing the lowest toxicity values (ANZECC and ARMCANZ 2000). This is easily done in conjunction with the data plotting step, described in section 2.6. A distribution can be fitted to a subset that does not contain the five taxa requirements, provided that the original data set fulfilled these requirements and the final subset contains at least five data points;

- c) If there is still a significant lack of fit after checking for multi-modality, outliers, and errors, proceed to section 3.4.2.3.2.

3.4.2.3.2 Procedure for an unsatisfactory fit

If the data set has been checked for errors, outliers, and multi-modality (or there is not enough evidence to determine if the SSD is multi-modal), and there is a significant lack of fit, then the following options are available:

- a) If there are more than eight original toxicity data:
If there is a significant lack of fit to the Burr Type III SSD, then the log-logistic SSD should be fit to the data, and the fit of the log-logistic SSD should be tested. If the log-logistic SSD fits the data, then it should be used for criteria calculation. If there is also a significant lack of fit to the log-logistic SSD, then the AF methodology (section 3.5) should be used to calculate the criteria.
- b) If there are 5-8 original toxicity data:
If there is a significant lack of fit to the log-logistic SSD, then the Burr Type III SSD should be fit to the data, and the fit of the Burr Type III SSD should be tested. If the Burr Type III SSD fits the data, then it should be used for criteria calculation. If there is also a significant lack of fit to the Burr Type III SSD, then the AF methodology (section 3.5) should be used to calculate the criteria. In this case it is likely that the data are multi-modal, but there are not enough data to clearly separate the lower subset.

3.5 Assessment factor procedure and rationale

When fewer than five data from an appropriate assortment of taxa are available, the SSD procedure cannot be used for criteria derivation. In such cases, an assessment factor procedure must be used (section 3.5.2). Background information on the use of AFs is given in section 3.5.1 so that users of the UCDSM can understand how the AFs were calculated. The rationale for determining the magnitude of assessment factors is discussed in section 3.5.1.1 and the detailed description of how the acute assessment factors used in the UCDSM were calculated is given in section 3.5.1.2. Users of the UCDSM do not need to re-calculate the AFs to use the method, but the description of how the AFs could be re-calculated is provided (section 3.5.1.2) for transparency and in the case that more data become available in the future, a user may want to re-calculate the AFs. The assessment factors used to calculate criteria in the UCDSM are provided in Table 18 and the procedure to calculate acute criteria using an assessment factor is described in section 3.5.2.

3.5.1 Assessment factor rationale

Assessment factors are recognized as a conservative approach for dealing with uncertainty in assessing risks posed by chemicals (Chapman et al. 1998). Assessment factors (also called safety factors, application factors, extrapolation factors, etc.) are usually applied to account for a wide range of possible effects and situations for which no data exist, including: lack of tests with relevant species, persistence or bioaccumulative potential of substances, genotoxic potential, laboratory to field extrapolation, acute-to-

chronic extrapolation, variations in mesocosm types for multispecies tests, absence of most sensitive species in multispecies tests, mixture effects, experimental variability, and lack of data (Fojut et al.2011; TenBrook and Tjeerdema 2006; TenBrook et al. 2009). Further factors may be applied in some cases based on the professional judgment of the risk assessor. In all cases, the more toxicity data are available for species of different trophic levels, different taxonomic groups, and different lifestyles, the smaller the applied factor.

An important point to keep in mind for using assessment factors is that application of AFs to toxicity data does not quantify uncertainty, but it does reduce the probability of underestimating risk, and can be thought of as a conservative approach for protecting ecosystem health. However, the use of AFs greatly increases the possibility of overestimating risk, which can be an important policy consideration (Chapman et al. 1998). A brief summary of points to keep in mind regarding the use of assessment factors in criteria derivation is given here (summarized from Fojut et al. 2013; TenBrook et al. 2009):

- a) Criteria must be protective of aquatic life, and therefore must err on the side of conservatism when effects data are lacking. When data are lacking, criteria will likely represent an overestimation of risk. More data will result in better estimates of risk, and therefore, better estimates of appropriately protective criteria;
- b) Assessment factors are used to fill gaps in scientific knowledge and existing criteria derivation methodologies use standardized factors of 10, 50, 100 and 1000, despite lack of supporting data (e.g., CCME 1995, ECB 2003, USEPA 2003a);
- c) Assessment factors are often based on policy rather than empirical science;
- d) All criteria are extrapolated values, and while those obtained by application of large factors to small data sets have a high level of conservatism and uncertainty, it is a policy decision whether or not to use them as threshold values;
- e) The AF procedure in the new methodology includes a range of factors and the factors get smaller as data sets get larger;
- f) Among the reasons for using larger factors are lack of data, persistence, bioaccumulative potential, mixture toxicity, and potential for genotoxic effects. The UCDM and UCDSM include other means of addressing bioaccumulative potential, bioavailability, and mixture toxicity and therefore do not incorporate these elements into assessment factors.

The AF procedure used in the UCDSM is the same as the approach used in the UCDM, which is modeled after the USEPA Great Lakes methodology. This AF procedure utilizes aqueous exposure data to generate empirically based assessment factors. These AFs generated from aqueous exposure data will be applied to limited sediment exposure data sets. Applying aqueous-based AFs to sediment data is not necessarily problematic, assuming the distribution of species sensitivities is similar in both aqueous and sediment exposures, and should be more reliable than arbitrary factor selection. AFs and ACRs are not statistically derived from sediment toxicity data because there is a dearth of sediment exposure studies.

The USEPA has recently begun to explore the use of AFs, also called extrapolation factors, that are specific to pesticides with a given mode of action (MOA), for example, acetylcholinesterase inhibitors (USEPA 2011). This work has identified that species sensitivity distributions can vary by MOA, and thus result in AFs that vary by MOA. The USEPA is also exploring ways of refining the AF derivation method used by Host et al. (1995) so that a complete criteria data set is not required for inclusion in the AF derivation data set; this would accommodate more pesticides so that robust MOA-specific AFs can be calculated. This methods are still be developed by USEPA, so they are not proposed for use in the UCDSM. It is recommended that when USEPA finalizes guidance on deriving MOA-specific AFs it should be evaluated to see if the proposed procedures are appropriate for use in the UCDM and the UCDSM.

3.5.1.1 Magnitude of assessment factors

A review of existing methods for SQC derivation revealed that magnitudes of AFs ranged from 1-1000 with little to no justification for the majority of AFs selected (Fojut et al. 2011). The UCDSM and UCDM used the procedure described by Host et al. (1995) to derive acute AFs and a default ACR that are designed to approximate the 5th percentile of the SSD using only pesticide data. Host et al. (1995) derived empirically-based and theoretically-supported final acute value factors, as well as default ACRs, that were used in the Great Lakes methodology (USEPA 2003a). For the UCDSM, acute AFs were derived from empirical aquatic pesticide effects data (Table 15) following the method of Host et al. (1995). The acute AFs for the UCDSM were calculated with the same data used to calculate AFs in the UCDM, with the addition of pesticide data for bifenthrin, cyfluthrin, cypermethrin, λ -cyhalothrin and permethrin.

3.5.1.2 Calculation of acute assessment factors

The procedure that was used to calculate the acute AFs presented in the UCDSM is outlined in this section. The AFs do not need to be re-calculated in order to use the UCDSM, but rather, the procedure is outlined here to provide background information on the AFs. If in the future more data are available a user would have the option of re-

calculating the AFs to incorporate this data. However, if a user is simply applying the UCDSM to calculate BSQC, the procedure to do so using AFs is given in the section 3.5.2.

As noted above, the procedure described by Host et al. (1995) was used with aquatic pesticide data to derive pesticide-specific acute AFs. The procedure used in the UCDSM (and UCDM) differs from what is described by Host et al. (1995) in that only pesticide data is used (vs. all kinds of contaminants) and Burr III or log-logistic distributions (vs. log-triangular distributions) are used. The only pesticide data available were for organic insecticides, but the AFs may also be relevant to herbicides, fungicides, molluscicides, and miticides, because some of these types of pesticides exhibit similar properties to insecticides. At this point it is only possible to derive acute (vs. chronic) factors because full data sets are required for several compounds to use the procedure. It was not possible to calculate AFs with spiked-sediment toxicity test data because no data sets were identified that were large enough to fit a SSD to; instead aqueous exposure data was used with the idea that SSDs for pesticide are expected to be similar for (epi)benthic organisms and those that reside in the water column (Di Toro et al. 2002). The goal of applying an AF is to estimate the 5th percentile of the SSD for a pesticide when there is not enough data to fit a SSD, so the assumption that the SSDs of benthic and water column organisms is key to the AF procedure of the UCDSM.

The goal of an AF is to estimate a 5th percentile value when data for fewer than the five required taxa are available. The magnitudes of the AFs need to be set to achieve this. To accomplish this, the AFs were used as divisors for the lowest value in data subsets containing 1-5 toxicity values. As per Host et al. (1995), the following procedure was applied to each individual pesticide data set (Table 15):

- a) Ninety-nine (99) subsets of five toxicity values were randomly selected with the restriction that the first value had to be for a benthic crustacean. These organisms were required because they are the most relevant for sediment criteria. Each successive sample had to fulfill a different requirement in the SSD minimum data set of the UCDM, which are 1) a benthic crustacean; 2) the family Salmonidae; 3) a warmwater fish; 4) a planktonic crustacean, of which one must be in the family Daphniidae in the genus *Ceriodaphnia*, *Daphnia*, *Simocephalus*; and 5) an insect (TenBrook et al. 2010). The selection of which family to use for the second and subsequent toxicity values in each subset was made randomly;
- b) For each subset of five toxicity values, subsets of 1-4 toxicity values were also created by subsampling the original 99 subsets of five values. This resulted in a total of 495 subsets of 1-5 toxicity values (i.e., 99 subsets of 5 values, 99 subsets of 4 values, 99 subsets of 3 values, 99 subsets of 2 values and 99 subsets of 1 value);

- c) The lowest acute value in each subset of size 1-5 toxicity values was used as the numerator for calculating the assessment factor;
- d) Each of the 99 five-sample subsets was used to generate 5th percentile values of whichever SSD was used on the original data set (Burr Type III or the log-logistic) following the procedures in section 3.4;
- e) The geometric mean of the 99 5th percentile values was used as the denominator for calculating the assessment factor;
- f) This procedure yielded 99AFs for each subset size;
- g) The 95th percentile of the 99 AFs was determined for each subset size.

This procedure was followed for aldrin, bifenthrin, chlordane, chlorpyrifos, cyfluthrin, cypermethrin, DDT, diazinon, dieldrin, endosulfan, endrin, heptachlor, λ -cyhalothrin, lindane, permethrin, and toxaphene for the UCDSM. Atrazine was not included in the UCDSM data set because the data was from a draft document, and it was also excluded from the UCDSM data set. Diazinon was not included by TenBrook et al. (2010) because the USEPA data set was bimodal and the Burr Type III SSD did not fit the data set. Diazinon was included in the AF re-calculation for the UCDSM using the diazinon data set gathered using the UCDSM, which did have a satisfactory Burr Type III SSD fit (Palumbo et al. 2012).

In accordance with USEPA (2003a), 95th percentile factors (from step g, above) for all pesticides were compiled and the median of those factors for each subset size was selected as the summary assessment factor for the UCDSM (i.e., a single factor to apply to any pesticide; Table 16). The summary AFs for each sample size are shown in Table 17 along with the estimated 5th percentile toxicity values obtained for each pesticide by dividing the geometric mean lowest value for each subsample size by the summary AFs. The geometric mean lowest value for each subsample size was not equal to the lowest value in the data set, because the subsamples were randomly selected and did not necessarily contain the lowest value in the overall data set. The lowest value varied among the 99 subsets within each subsample size, which is why the geometric mean of these values was calculated. The geometric mean lowest values for each subsample size are not reported in Table 17, but can be simply back-calculated as the median 5th percentile multiplied by the appropriate AF for reference.

The estimated median 5th percentile values in Table 17 were compared to the lowest values from the full data sets to check that the values estimated using AFs would be protective of the most sensitive tested species. The summary AFs produced an estimated 5th percentile value that is equal to or below the median 5th percentile value determined from applying the SSD procedure to the full data set in all but two cases. The

two exceptions were both less than a factor of 1.5 higher than the median 5th percentile and would both result in criteria lower than the lowest value in the data set. Thus, additional safety factors are not recommended for deriving criteria based on one datum, as was done in the UCDM. The final summary assessment factors are given in Table 18.

3.5.2 Assessment factor procedure

If data requirements for the SSD procedure are not met or an SSD cannot be fit, the assessment factor method is used to derive criteria. An AF is used to estimate the median 5th percentile, which is referred to as the acute value, by dividing the lowest species mean acute value from the acceptable data set by the appropriate AF (Table 18). The magnitude of the AF is dependent on the number of data requirements met, and at least one of the available, acceptable data must be a benthic crustacean, or a criterion cannot be calculated. Each of the additional data must satisfy a different required taxon of the SSD method (section 3.4.1), such that each additional value is building toward completion of the minimum SSD data set. The resulting acute value represents an estimate of the median 5th percentile value of the SSD, which can then be used to calculate the acute criterion, which is described in section 3.7.

Eq 17: Acute value = lowest value in data set/assessment factor

Because the assessment factors have been formulated with data from organic insecticides, these factors could also be relevant for some molluscicides, miticides and/or fungicides, which exhibit similar properties (TenBrook et al. 2010). Metal-based pesticides were not included in the derivation of the AFs, and therefore it is not known if the AFs will give reasonable estimates of the 5th percentile for these compounds. The AFs in Table 18 can be updated and re-calculated as more criteria are generated. Data sets that meet the five SSD taxa requirements may be added to the current data set in order to calculate new AFs.

3.6 Acute-to-chronic ratios

If at least five chronic data are available from five different families, the SSD method should be used to derive chronic criteria. Chronic ecotoxicity data are rarely available; thus an acute-to-chronic ratio is needed to extrapolate chronic toxicity from acute data. An ACR is calculated by dividing an acute value (LC/EC₅₀) by a chronic value (e.g., EC_x, MATC). Appropriate paired data to compute ACRs are acute and chronic toxicity values derived from the same test, or from tests conducted by the same laboratory under identical conditions (USEPA 1985; 2003a). There are three basic approaches to deriving ACRs (TenBrook et al. 2010): 1) derive chemical-specific, multispecies ACRs using acute and chronic values derived from the same tests

(ANZECC and ARMCANZ 2000; USEPA 1985, 2003a); 2) derive chemical-specific, multispecies ACRs using available chronic data, combined with one or more default ACR values (USEPA 2003a) and; 3) use a default ACR. It is preferable to use ACRs based on experimental data when available. When sufficient data are not available to calculate a chemical-specific ACR, a default ACR is used.

The UCDM provides a stepwise procedure to determine the ACR depending on data availability and is appropriate for incorporation into the UCDSM to extrapolate acute effects concentrations to chronic effects levels. Chronic criteria that are calculated using an ACR must be checked against available chronic water-only and sediment toxicity data to ensure adequate protection.

A review of the literature uncovered only one SSTT-based ACR for cypermethrin and *C. dilutus* (formerly *C. tentans*) that was determined to be approximately 7 (Giddings et al. 2006). Giddings et al. (2006) calculated this ACR based on data from 10-day LC₅₀ of 290 µg/g and a 60-day NOEC of 39 µg/g for growth, reproduction and emergence (the three endpoints resulted in identical toxicity values). These studies were conducted using standard protocols and approved spiking procedures that include applying the chemical to sand and evaporating off the solvent before mixing with natural sediment and equilibrating for at least 4 weeks. From the Giddings et al. (2006) data sets, other ACRs could also be calculated using different toxicity values, for example, using the 60-d MATC for growth or reproduction of 57 µg/g as the chronic value instead of the NOEC would result in an ACR of approximately 5. An EC₅₀ for growth of 220 µg/g was also reported that could serve as the acute value in the ACR calculation, if used with the 60-day MATC for growth or reproduction of 57 µg/g as the chronic value would result in an ACR of approximately 4. Until more acute and chronic SSTT data are available for a variety of species, ACRs based on water-only exposure concentrations are recommended for calculating chronic criteria from acute data as discussed in section 2.2.2.

3.6.1 Multispecies ACR based on measured data

The UCDM guidance on ACR derivation is based on that provided in detail by the Great Lakes water quality guidance document (USEPA 2003a). The procedure for aqueous exposures requires acute and chronic data from organisms in at least three different families that include a fish, an invertebrate, and at least one other acutely sensitive species. If insufficient freshwater data are available to fulfill the ACR data requirements, saltwater species may be used, as freshwater and saltwater ACRs have been shown to be comparable (USEPA 1985). This approach has been accepted in numerous water criteria derivations (Siepmann and Finlayson 2000; USEPA 1980a, b, c, d, 2003b, 2005a). To adapt these requirements to sediment exposures, the UCDSM requires acute and chronic data from organisms in at least three different families of

benthic organisms, but particular taxa groups are not specified, and saltwater data for benthic species are also acceptable. In reality, because SSTT data are so scarce, particularly for chronic exposures, this procedure is unlikely to be used and default ACRs will be employed. However, in the case that such data are available, the guidance provided by USEPA (1985) has been adapted for sediment and are described below.

For each chronic value (EC_x or MATC) having at least one corresponding appropriate acute value, an ACR is calculated by dividing the geometric mean of all acceptable acute toxicity values (in the case that multiple acute tests were conducted in one study) by the chronic value. For all species, the acute test(s) should be part of the same study and use the same dilution water or sediment as the chronic test. If acute tests were not conducted as part of the same study, but were conducted as part of a different study in the same laboratory and sediment, then they may be used. If no such acute tests are available, results of acute tests conducted in the same sediment in a different laboratory may be used. If no such acute tests are available, an ACR is not calculated.

The ACR calculation procedure steps described here should be followed in order. The species mean acute-to-chronic ratio (SMACR) is calculated for each species as the geometric mean of all ACRs available for that species. For some materials, the ACR seems to be the same for all species, but for other materials the ratio seems to increase or decrease as the SMAV increases. Thus the multispecies ACR can be obtained in one of three ways, depending on the data available:

- a) If the SMACR seems to increase or decrease as the SMAVs increase, calculate the ACR as the geometric mean of the ACRs for species whose SMAVs are close to the acute 5th percentile value (this includes species whose SMACRs are within a factor of 10 of the SMACR of the species whose SMAV is nearest the 5th percentile value);
- b) If no major trend is apparent and the ACRs for all species are within a factor of 10, the ACR is calculated as the geometric mean of all of the SMACRs;
- c) If SMACRs are less than 2.0, and particularly for SMACRs less than 1.0, acclimation has probably occurred during the chronic test. In this situation, the final ACR should be assumed to be 2.0, so that the chronic criterion is equal to the acute criterion.

If the available SMACRs do not fit one of these cases, use the procedure described in section 3.6.2 to derive an ACR based partially on measured values and partially on default values. This procedure generally applies if the data requirements of this section cannot be met, or if the ACR cannot be obtained by one of methods a, b or c above.

3.6.2 Multispecies ACR based on measured and default values

If insufficient data are available for calculation of an ACR according to the procedure in section 3.6.1, the ACR can be derived by calculating the geometric mean of any available experimental ACRs, plus enough default ACRs of 11.4 (described in the next section) to give a total of three ACRs (USEPA 2003a). For example, if no experimental ACRs are available, three default ACRs should be used. If two experimental ACRs are available, one default value is sufficient to make up the total of three ACRs.

3.6.3 Default ACRs

The default ACR used in the Great Lakes guidance was recalculated for the UCDM to include only pesticide data from Host et al. (1995), data from the California Department of Fish and Wildlife (CDFW) diazinon criteria document (Siepmann and Finlayson 2000) and a new chlorpyrifos data set (TenBrook et al. 2010). For the UCDSM, this data set was expanded to include pyrethroid data (cyfluthrin and λ -cyhalothrin; Table 19) and the CDFW diazinon data was replaced with UCDM diazinon data (Palumbo et al. 2012). The default ACR was calculated as the 80th percentile of all of the chemical-specific ACRs; the Great Lake guidance uses the 80th percentile to determine the default ACR and this percentile was also adopted for the UCDM and UCDSM (Host et al. 1995). This data set produced a default ACR of 11.4, which comes with the same caveats presented in the UCDM: 1) if data sets collected according to the UCDM or UCDSM lead to different ACRs, those values should be substituted into this table and the default ACR should be recalculated, especially if it can be calculated based on SSTT data; 2) if previously calculated ACRs are shown to be invalid based on data sets collected according to the UCDM or UCDSM, then those values should be removed and the default ACR should be recalculated; and 3) if additional pesticide ACRs become available, the default ACR should be recalculated (TenBrook et al. 2010). In any of these events, the default ACR should be recalculated as the 80th percentile value of the new set of ACRs. Any future revisions of the value should start with the ACRs in Table 18.

The default ACR was formulated with data from organic insecticides and thus, could be relevant to some molluscicides, miticides and/or fungicides that have similar properties. The default ACR was not derived with any data for metal-based pesticides so it is not known if using the default ACR for these compounds would give reasonable estimates of the chronic toxicity.

3.7 Final criteria calculations

3.7.1 Acute and chronic criteria calculations

Acute criteria are derived using EC₅₀ or LC₅₀ data, while chronic criteria are derived using EC_x or MATC data. To calculate an acute criterion, an acute value is derived from an SSD or AF, which is an estimated percentile of the true distribution. The recommended acute value is the 5th percentile value at the 50% confidence value, but other acute percentiles can be used if other ecotoxicity data indicate that the criterion calculated with this percentile is not protective. The acute value is divided by a safety factor of 2 because a 50% effect is not acceptable. The safety factor of 2 was originally calculated across 219 acute aqueous toxicity tests with various chemicals, which showed that the mean concentration that did not cause mortality greater than the control was 0.44 times the LC₅₀ (USEPA 1978). The inverse of 0.44 (2.27) was rounded to 2 for use in EPA WQC derivation methods (USEPA 1985, 2003a). The chronic value from either a SSD or an AF is used as the chronic criterion. The criteria can be expressed as the OC-normalized sediment concentration (e.g., µg/g OC), the DOC-normalized interstitial water concentration (e.g., µg/g DOC), or the freely dissolved interstitial water concentration (e.g., ng/L).

For the acute criterion:

Eq 18: Acute criterion = acute value/2

The recommended criterion = (5th percentile value at 50% confidence level)/2

For the chronic criterion:

The recommended criterion = 5th percentile value at the 50% confidence level

Alternatively, more conservative criteria may be derived from other percentile or confidence levels.

The number of significant digits in the final criterion should be consistent with known variability in the calculated criteria. Calculated criteria should not be expressed with more significant figures compared to the original toxicity data. If using the median estimate as the criteria, the 95% confidence limit can be used as a guide. The digit in the median estimate that is different from the 95% confidence limit will indicate the last significant digit. Also, the 5th percentile values generated from omitting data sets during the fit test (section 3.4.2.3) can be used to estimate the uncertainty in the calculated

criteria. The last digit that is relatively variable among these estimates indicates the last significant digit.

If toxicity is quantitatively related to a water quality parameter (i.e., pH, temperature dependence), follow procedures in section 4.3 for appropriate calculation of the criterion. Criteria should be checked against the individual toxicity values in the data sets used in the SSD, to ensure protection of all represented species.

3.7.2 Averaging periods

Criteria derived according to any of the above methods are stated in terms of the magnitude of a chemical that may be in the bioavailable fraction without causing harm, but without consideration of the duration and/or frequency that this level may be exceeded without harm. Section 3.7.3 addresses the frequency component. This section explores the question of duration. The goal in setting an averaging period or exposure duration is to choose a duration long enough that toxicity might occur due to an exceedance and short enough that the effects of concentration fluctuations on the average concentration are minimized (TenBrook et al. 2010).

Ideally, data or models that could account for various exposure durations would be available to determine criteria compliance for a given set of samples. This would account for both exposures of constant concentrations and pulse exposures. The criteria are derived from studies conducted under constant exposure scenarios that do not account for the possibility of pulsed, or otherwise uneven, exposures. New inputs from storm or irrigation events, degradation or dissipation, or transformation due to biologic activity in the sediments are some of the reasons that sediment exposures to current-use pesticides may be uneven. Time-to-event models could potentially provide a way to express criteria for any given exposure duration, but such models are not currently feasible for use in criteria derivation and will not likely be feasible in the future, given that time course of sediment toxicity has not been evaluated in past studies and will not likely be evaluated in future studies.

Because neither data nor models are currently available to account for various exposure durations for criteria compliance, the following types of information were reviewed in the literature to inform the selection of averaging periods: 1) concentration fluctuations in field sediments and interstitial water, 2) degradation rates in sediment, 3) differences in durations and concentrations causing mortality and sublethal effects in spiked-sediment toxicity tests, and 4) life cycles of benthic organisms.

Fojut et al. (2013) noted in their review of sediment quality criteria derivation methods that none of the existing methods discussed the duration component of the criteria. One explanation for this was that sediment contaminant concentrations are

expected to be relatively stable over time, with constant chronic exposures (USEPA 2003c, d). This explanation may be true for legacy compounds, such as the organochlorine pesticides, but is not necessarily true for current-use pesticides, which tend to be less persistent. Spatial heterogeneity is also a critical factor in exposure; if there are differences in concentration on a microscale important to the organism, this factor will affect exposure more than the persistence of a contaminant in a fixed point. Thus, the literature was surveyed to determine the timescale on which current-use pesticide concentrations fluctuate in sediments and interstitial water and the spatial heterogeneity of pesticide concentrations in sediments and interstitial water. In the field it is difficult to determine whether concentration changes over time are due to degradation and/or new inputs, or whether they are simply caused by spatial heterogeneity.

Several studies analyzed sediments from an individual site on a biweekly to quarterly basis and demonstrated that concentrations of current-use pesticides, such as pyrethroids, organophosphates, and herbicides, vary significantly on these time scales, but the cause of variation cannot be clearly identified (Domagalski et al. 2010; Long et al. 1998; Moore et al. 2002; Phillips et al. 2012; Weston et al. 2009a). For example, Domagalski et al. (2010) reported bifenthrin sediment concentrations from an agricultural creek (Hospital Creek, California) varied by a factor of 5 between samples from July and September. When these concentrations were converted to toxicity units (TUs) based on 10-day LC₅₀s for the amphipod *Hyaella azteca*, the TUs varied by a factor of 3.9. They reported that pyrethroid detections in sediments and their concentrations were highly variable and that these changes might be attributable to new pesticide inputs or re-suspension and deposition from previous applications, as well as degradation processes. Domagalski et al. (2010) collected integrated samples over a 100-m reach, and the variation in concentrations may in part be due to spatial heterogeneity of pesticide deposits within each reach.

Weekly sediment sampling was conducted from June to September 2003 in several creeks and ditches in an agricultural area (Monterey County, California) where flows are dominated by agricultural field runoff (Kelley and Starner 2004, Starner et al. 2008). In this study, pyrethroid sediment concentrations were relatively stable and typically fluctuated by less than a factor of 2 from one week to the next, when they were detected. However, there were three occasions in which the pyrethroid sediment concentration changed by a factor of 2-4 from one week to the next. These larger fluctuations were likely the response from recent field applications to crops, unrelated to storm events. Monthly or biweekly sampling of pyrethroids in sediment from two California agricultural creeks (Del Puerto and Orestimba) conducted from December 2007-June 2008 showed similar concentration fluctuations of a factor of 1-4 between sample dates (Ensminger et al. 2011). When these sediment concentrations were converted to TUs based on LC₅₀s for *H. azteca*, the TUs ranged from <1-15 over the 7

month period, emphasizing the large range of potential sediment toxicity over this relatively short time scale. Again, it should be emphasized that the causes of variations in sediment concentrations over time may in fact be due to spatial heterogeneity, and not accumulation or degradation processes.

Interstitial water concentrations of pesticides have not been widely monitored in the field and there is limited data to survey. Physicochemical parameters including partitioning coefficients and solubility, as well as environmental conditions, will govern equilibration times between sediments and overlying or interstitial water, and highly hydrophobic pesticides will have longer equilibration times (≥ 30 days). Thus, interstitial water concentrations may vary with contact time, particularly for compounds with large partition coefficients (Bondarenko et al. 2006). Sediment and interstitial water samples from the San Diego Creek watershed, California were analyzed for pyrethroids in the wet (April-May) and dry (August) seasons of 2005 (Budd et al. 2007). Concentrations in both matrices varied widely, with interstitial water concentrations differing by up to a factor of 20 when detected, and ranging as widely as 1.5-31.2 ng/L at an individual site. These differences likely reflect the heterogeneity of the sediments and the streambed. In the same study, the OC-normalized sediment concentrations differed by up to a factor of 10 at an individual site. In a study in the Salinas River, California, chlorpyrifos concentrations measured in interstitial water followed the same trends as chlorpyrifos measured in the overlying water (Anderson et al. 2003). Other studies have also shown correlations between interstitial water and overlying water concentrations, indicating that bioavailable concentrations of current-use pesticides are not constant and fluctuate similarly to water column concentrations (Giesy et al. 1999, Bacey et al. 2004).

Degradation rates of current-use pesticides are varied, with half-lives ranging from days to months to years, and are particularly dependent on whether environmental conditions are aerobic or anaerobic (Bondarenko and Gan 2004, Nillos et al. 2009, Qin et al. 2006). Compounds that degrade very quickly ($t_{1/2} < 3$ days) are less likely to be identified as pesticides of concern in sediment, but if criteria were derived for such a pesticide, the degradation rate may be considered when determining the final criteria statement with clear justification if any adjustment is recommended. However, many pesticides have relatively constant sources in the Sacramento-San Joaquin watershed that may negate the effect of degradation, particularly those used in urban and residential areas because those uses are less seasonal than agricultural uses.

Spiked-sediment toxicity testing has demonstrated that various invertebrate species experience mortality and sublethal toxic effects due to sediment-associated pesticides in the standard test duration of 10 days. In one study testing *C. dilutus* and *H. azteca*, growth NOECs for nine pesticides were approximately one half of their LC_{50} values in 10-day exposures (Ding et al. 2011). Maul et al. (2008) examined lethal and multiple sublethal endpoints in spiked-sediment toxicity tests with *C. dilutus* (formerly *C.*

tentans) and four current-use pesticides, as well as two pesticide metabolites in 10-day exposures. The most sensitive sublethal endpoints were related to growth and immobility and were up to a factor of 9 lower than LC₅₀s for a given constituent. There are few pesticide SSTT data available for other taxa, such as amphibians or fish, but other types of studies indicate that these taxa are sensitive to pesticides (Hayes et al. 2006, Wojtaszek et al. 2005).

The standard 10-day exposure duration in spiked-sediment toxicity testing presents a conundrum because it is typically thought of as an acute test, but the duration may be a significant portion of an organism's life cycle for some species (ASTM 2007, 2013). The goal of acute criteria is to protect against adverse effects due to chemical concentrations that occur on a short time scale, which is defined as a duration that is not a significant portion of an organism's life cycle. The goal of chronic criteria is to protect against adverse effects due to exposure over a significant portion of an organism's life cycle. For many invertebrate species 10 days is a significant portion of their life cycles, which are one month or less for many benthic organisms (ASTM 2013, Dussault et al. 2008, Phillips et al. 2010, Tassou and Schulz 2012). The 10 day duration appears to be somewhere between a short-time scale and a significant portion of the life cycle for many benthic invertebrates, thus, not particularly well-suited for derivation of either type of criteria, yet 10-day toxicity values constitute the vast majority of the few available SSTT data for pesticides. If the goal is to use SSTT data to derive the criteria, the 10-day data must be used and it would be an oversight to ignore either the mortality or growth endpoint. Chronic data are sparsely available, in part because standard guidance has only been developed for a few test species (*H. azteca*, *C. dilutus*; ASTM 2013). These SSTT methods measure growth and/or reproductive effects and have exposure durations ranging from 28 to 42 days, but they are still considered in the development phase and are not yet considered standard protocols.

Considering all of the above factors, including available data and standard test methods, the UCDSM proposes a 10-day averaging period for acute BSQC and a 28-day averaging period for chronic BSQC. The authors recognize that there is no information available to know whether acute effects occur on a shorter time-scale than 10 days and the potential magnitudes of concentrations that could cause them. Because standard acute test methods have 10-day exposure durations, it is unlikely that more information on acute effects from shorter duration exposures will be generated in the near future. In reality, environmental monitoring for compliance is typically performed monthly or even less frequently, and in these cases, the chronic BSQC will need to be protective of both acute and chronic environmental exposures.

3.7.3 Allowable frequency of exceedance

In setting an allowable frequency of exceedance of a BSQC, the question is how much time it would take for organisms at various organizational levels to recover from pulse exposures to contaminants. TenBrook et al. (2010) conducted a thorough review on this subject and concluded that an allowable frequency of exceedance of three years should allow for full recovery of aquatic ecosystems from the effects of an excursion above criteria and the following is a brief summary of this review. It appears that ecosystem recovery from pulse exposures generally occurs in less than three years, and often in less than one year (Yount and Niemi 1990). Species that are slowest to recover are those with the longest life cycles. Most ecosystems are able to recover from disturbances in less than three years except in cases where the physical habitat was altered, the system was isolated, or residual pollutant remained (Niemi et al. 1990). The majority of reviewed studies that consider community, population, or species-level effects indicate that recovery occurs in three years or less. Based on this review of over 30 studies, three years between exposure events should allow full recovery from effects of an excursion above BSQC. This is in agreement with the UCDSM and USEPA WQC methodologies (USEPA 1985, 2003a). The UCDSM includes a statement that BSQC exceedances should not occur more than once every three years.

4 Water quality effects

4.1 Bioavailability

Bioavailability is directly incorporated in the UCDSM by using either the freely dissolved interstitial water concentration, the DOC-normalized interstitial water concentration, or the OC-normalized sediment concentration to derive criteria for a wide range of sediment types. The rationale for the bioavailability approach to BSQC derivation is discussed in section 1.2.3.

For compliance monitoring, the same types of concentrations should be used to compare field measurements to BSQC: OC-normalized sediment concentrations, DOC-normalized interstitial water concentrations, the freely dissolved interstitial water concentration, or the bioaccessible concentration. Whole interstitial water or whole dry-weight sediment concentrations are not recommended for compliance monitoring for hydrophobic compounds ($\log K_{ow} > 3$) because sorption to DOC or TOC will likely overestimate the bioavailable fraction. Measuring pesticides in interstitial water is not a common practice in environmental monitoring, so it is likely that most monitoring data will consist of sediment concentrations. If sediment samples are analyzed for pesticides, the sediment OC content must also be measured so that the dry weight sediment

concentrations can be OC-normalized. It is not appropriate to compare dry-weight sediment concentrations to OC-normalized BSQC.

4.2 *Mixtures*

As recommended in Phase I (Fojut et al. 2011), the UCDSM includes the concentration-addition model for pesticides with similar modes of action (Plackett and Hewlett 1952) and the non-additive interaction model for chemicals that display antagonistic or synergistic interactions (Finney 1942). Two approaches to using the concentration-addition model are presented in section 4.2.1 and the non-additive interaction model is presented in section 4.2.2. The application of all of these mixture models requires that each pesticide that is considered in the model has a numeric BSQC.

4.2.1 *Additivity –compounds with similar modes of action*

The concentration-addition model can be used for mixtures of similarly-acting pesticides. The toxic unit approach is recommended for estimating the combined toxicity of compounds with the same mode of action. The toxic unit (TU) approach has been described by Pape-Lindstrom and Lydy (1997), and this approach was adapted to determine criteria compliance by the CRWQCB-CVR (2011) as follows:

Eq 19:
$$\sum_{i=1}^n \frac{C_i}{O_i} < 1.0$$

where:

C_i = concentration of toxicant i (in sediment or interstitial water);

O_i = interstitial water quality objective/criterion for toxicant i (in the same matrix as C_i).

This approach is also adopted for the UCDSM. As long as the sum is < 1.0 , the water body is considered to be in compliance with respect to the mixture.

4.2.2 *Non-additivity*

Chemical mixtures may display non-additive toxicity in the form of either antagonistic or synergistic effects. This indicates an interaction between chemicals such that the response observed for a mixture is either less than (antagonism) or greater than (synergism) that predicted by additivity models. The concept of synergy is often used in reference to cases where one chemical present at non-toxic concentrations increases the toxicity of a second chemical, but it can be applied to mixtures in which both chemicals are at toxic levels. Mu and LeBlanc (2004) utilized the coefficient of interaction (K) to define this relationship. First described by Finney (1942), the basic equation is:

Eq 20:
$$K_x = \frac{EC50_0}{EC50_x}$$

where:

K_x = coefficient of interaction at synergist/antagonist concentration x ;

$EC50_0$ = EC_{50} of chemical in absence of synergist/antagonist;

$EC50_x$ = EC_{50} of chemical in presence of synergist/antagonist at concentration x .

When a measured concentration of a chemical is multiplied by K_x for a given concentration of a synergist/antagonist, the result is an adjusted, or effective, concentration of the chemical in presence of the synergist/antagonist. Mathematically, this is expressed as:

Eq 21:
$$C_a = C_m(K_x)$$

where:

C_a = adjusted, or effective, concentration of chemical;

C_m = measured concentration.

For application to compliance determination, Eq 21 can be used to determine the effective concentration for comparison to the BSQC. Additionally, the effective concentration can be used in the compliance model for additivity described in section 4.2.1 (Eq 19). The difficulty is in determination of an appropriate K value. Although, logistic functions have been used to describe the relationship between K values and piperonyl butoxide (PBO) concentrations (Rider and LeBlanc 2005), K values derived in that manner are not generally applicable to a wide range of pollutants or species.

As discussed in the UCDM, Equation 22 can be modified, in theory, for mixtures containing both synergists and antagonists, or multiple synergists/antagonists (TenBrook et al. 2010):

Eq 22:
$$C_a = C_m * (K_1 K_2 \dots K_n)$$

where:

K_1, K_2, K_n = K values for synergist/antagonist 1, 2... n .

It is cautioned that the error of the adjusted concentration will increase as more K values are strung together, and as a result, this approach should not be used for compliance determination, but may be used to assess research needs.

4.2.3 Combined models

Environmental samples are usually complex mixtures and the models discussed so far apply to only one type of mixture effect. Combined mixture effects models account for additivity with similar modes of action, additivity with different modes of action, and interactions leading to synergism or antagonism. A model was developed that combines concentration-addition and response-addition models, including an interaction component (Olmstead and LeBlanc 2005; Rider and LeBlanc 2005):

$$R = 1 - \prod_{I=1}^N \left\{ 1 - \frac{1}{1 + \frac{1}{\left(\sum_{i=1}^n \frac{k_{a,i}(C_a) \times C_i}{EC50_i} \right)^{\rho'}}} \right\}$$

Eq 23:

where:

R = response of the mixture (percent of individuals responding);

N = number of cassettes (cassette = group of chemicals of similar mode of action);

I = I^{th} cassette;

n = number of chemicals;

i = i^{th} chemical;

$k_{a,i}$ = interaction coefficient for chemical a (synergist/antagonist) interacting with chemical i ;

C_a = concentration of chemical a in the mixture;

C_i = concentration of chemical i in the mixture;

$EC50_i$ = EC_{50} for chemical i alone;

ρ' = average power (slope) of dose-response curves of chemicals in cassette I .

A thorough discussion regarding the use of the combined toxicity model in WQC derivation was provided by TenBrook et al. (2010). Briefly, the model integrates all aspects of mixture toxicity, but can only be applied to one species at a time. Although the model could be adapted to determination of criteria compliance, it may not be possible to derive a reliable multi-species K value. The concern is that since K is a mechanism-dependent value, the assumption of a common value across species is equivalent to assuming similar toxicity mechanisms across species (TenBrook et al. 2010). TenBrook et al. concluded that before it can be used for compliance assessment, the Rider and LeBlanc (2005) model needs to be validated for use across species. However, K values for individual species could be used to assess the potential harm from non-additive toxicity on a species by species basis (TenBrook et al. 2010).

4.2.4 Summary of mixtures

The concentration-addition model and the non-additive interaction model are included in the UCDSM. The non-additive interaction model is presented with the caveat that it can only be applied in cases where a valid coefficient of interaction (K) is available (either a multispecies K value, or individual species K values). Without multispecies K values, this technique should not be used to assess compliance with BSQC, but K values for individual species could be used to assess the potential harm from non-additive toxicity on a species by species basis. The application of all of these mixture models requires that each pesticide considered in the model has a numeric BSQC.

4.3 Environmental factors affecting toxicity (temp, pH, etc.)

Environmental conditions, such as temperature, can significantly affect the toxicity of pollutant chemicals on living organisms. As such, relevant environmental characteristics should be included in ecological risk assessments. The UCDM provides a procedure to assess if the toxicity of two or more species has a similar relationship to the water quality characteristic tested (e.g., temperature, pH dependence; USEPA 1985, 2003a). To apply this procedure to the UCDSM, SSTT data at both standard and non-standard conditions for two or more species would be required. The UCDM also provides a procedure to recalculate toxicity values from tests conducted under non-standard conditions to transform them to standard conditions so that these recalculated values could be added to the acceptable data set, in the case that a statistical relationship between toxicity and a water quality characteristic is demonstrated. Neither of these procedures is described in any more detail the UCDSM because it is very unlikely that the data necessary to determine a statistical relationship between toxicity and the water quality parameter would be available. However, if such data were available, the procedures are appropriate to apply to SSTT data, and section 9.5.3 of the UCDM can be consulted to examine these relationships (TenBrook et al. 2012).

5 Comparing derived criteria with ecotoxicity data

A comparison must be made between the derived criteria and acceptable ecotoxicity data values to ensure protection of sensitive species, threatened and endangered species (TES) and the ecosystem as a whole. If the toxicity values indicate that the criteria may not be protective of all species, then downward adjustment may be recommended. Guidance on downward adjustment of criteria is given in section 5.5. If sediment concentrations and aqueous concentrations are being compared, refer to section **Error! Reference source not found.** to convert between the two media.

5.1 Sensitive species

The BSQC should be compared to all toxicity values that were rated as acceptable (RR) or supplemental (RL, LR, LL) to ensure that the criteria are protective of the most sensitive species in the data sets. If any toxicity values are below the derived criterion, the criterion could be adjusted downward to be protective of the most sensitive tested species (see section 5.5).

5.2 Threatened and endangered species

Threatened and endangered species may be particularly sensitive to stressors in the environment, thus it should be ensured that BSQC will be protective of these species. TES typically have a limited range, thus in setting BSQC for the Sacramento and San Joaquin River basins or any specific area, only local species should be considered. There are various benthic organisms currently on the CDFW list of TES, including eight crustaceans (CDFW 2013), which could potentially be affected by sediment contaminants. Generally, few SSTT data are available, and toxicity data for TES are likely to be even scarcer, thus being able to predict toxicity values for TES would be very valuable. Unfortunately, interspecies correlations based on surrogate species have not been developed for sediment toxicity, as they have for aqueous toxicity. It may be difficult to incorporate specific procedures to ensure protection of benthic TES at this time because of the few toxicity data likely to be available. However, any available TES data should be used to evaluate the derived criteria to assess protection of listed species.

The UCDM provides a framework to evaluate both acute and chronic TES toxicity data against the derived acute and chronic criteria and this procedure is adopted in the UCDSM. The procedure is detailed as follows (TenBrook et al. 2010):

1. Obtain the list of California TES available from the CDFW web site: http://www.dfg.ca.gov/wildlife/nongame/t_e_spp/ (CDFW 2013).
2. Compare the acceptable acute and chronic toxicity values to the acute and chronic TES values.
3. If no acute or chronic surrogate values are available and the chemical of interest has a narcotic mode of action, select a QSAR that can be used to estimate toxicity to the TES or to a surrogate based on a log K_{ow} . QSARs from the RIVM (2001) and OECD (1995a) are presented in the UCDM (TenBrook et al. 2010) but do not preclude the use of other acceptable QSARs.

If there is a lack of TES, surrogate or QSAR toxicity values available for comparison, the comparison cannot be made. Although the specific protection of TES cannot be evaluated under these circumstances, the criteria are derived with the intention of protecting all species. If the most sensitive species is protected, it is likely TES will be

included, assuming that the most sensitive species in the ecosystem is known. The criteria may be adjusted downward if the criterion is found to not be protective of TES.

5.3 Multispecies studies

The derived BSQC should be compared to the acceptable multispecies laboratory, field or semi-field studies to ensure protection of the ecosystem as a whole. Acceptable multispecies studies are those that rate R or L according to Table 7. Guidance for this comparison has been adopted from the UCDM; BSQC should be compared to reported ecosystem NOECs, or on NOEC, EC_x, IC_x or LC_x values for individual species within a system (TenBrook et al.2010). Criteria may be adjusted downward if they exceed toxicity values from multispecies studies.

5.4 Comparison to water quality criteria

The acute and chronic sediment criteria are based on bioavailable concentrations, which can be expressed as freely dissolved interstitial water concentrations. As such, the BSQC expressed as freely dissolved interstitial water concentrations should be compared to both acute and chronic water quality criteria. This comparison ensures that the BSQC are protective of the entire aquatic ecosystem and not just those organisms living in the benthic zone.

5.5 Adjusting criteria based on ecotoxicity data

It is recommended that criteria are only adjusted downward, not upward, based on ecotoxicity data because the available single-species data has indicated that the criteria are protective and upward adjustment may result in toxicity to sensitive species. Typically, the most sensitive species in a given ecosystem may not be known and most sensitive species are not suitable for laboratory toxicity testing. Thus, upward adjustment of criteria may lead to underprotective BSQC. Criteria should only be adjusted downward based on toxicity values from either acceptable (RR) or supplemental (RL, LR, LL) studies that use of the acceptable methods for accounting for bioavailability; toxicity values based on nominal concentrations or concentrations that do not account for bioavailability (e.g., dry weight sediment concentrations) are not an acceptable basis for adjustment. Criteria should only be adjusted based on supplemental toxicity values if the species is found in North America and the species is not represented in the acceptable data set. Best professional judgment should also be used to determine whether the study is an appropriate basis for criteria adjustment. If a SSD was used to calculate the criteria, then the next lowest estimate (e.g., lower 95% confidence interval or 1st percentile) should be used in criteria calculation to adjust the criteria downward. If an AF and/or

ACR were used for criteria calculation, then the acute value can be divided by a factor of 2 for downward adjustment.

6 Harmonization between media

The potential for pollutants in the sediment and/or interstitial water to partition into other environmental compartments (i.e., water column, air, biota) should be assessed during criteria derivation because it is of concern to environmental managers. Specifically, the potential for sediment pollutants equal to the BSQC to exceed levels of concern established for other compartments should be assessed. This is defined as harmonization between media and was discussed in TenBrook et al. (2009). If BSQC are found to be in conflict with existing guidelines for other compartments, this fact should be flagged for review by environmental managers, but the BSQC should not be adjusted.

6.1 Environmental media

Pesticides in the sediment and/or interstitial water may partition to the water column and may cause toxicity in this environmental compartment. Steady-state environmental models may be used to assess harmony of chronic criteria across all environmental media. These models are based on equilibrium partitioning, thus only chronic BSQC will be used; acute criteria are not appropriate because they are short-term transient pesticide concentrations (non-equilibrium). Levels of concern and/or toxicity data for the water column are necessary for this assessment and they are typically available. Harmonization with air is not directly addressed in this report because partitioning would occur between water and air, not between sediment and air. If BSQC are harmonized with WQC, then air is accounted for because WQC are harmonized with available air criteria.

There are freely available acceptable models for this analysis, 1) the Exposure Analysis Modeling System (EXAMS; Burns 2004), available from the USEPA Center for Exposure Assessment Modeling (CEAM; <http://www.epa.gov/ceampubl/swater/index.htm>) and 2) Mackay's (2001) Fugacity-Based Environmental Equilibrium Partitioning Models Levels I, II, and III, available as free downloads from the Canadian Environmental Monitoring Center (CEMC; <http://www.trentu.ca/cemc/welcome.htm>). These models vary in complexity and require the use of default environmental parameters when measured values are not available, but they can provide rough estimates of equilibrium concentrations in all environmental compartments based on a given sediment or interstitial water concentration and some pesticide physicochemical properties.

The Level I fugacity model is recommended as an initial evaluation because of its relative simplicity. The EXAMS and Level II and III fugacity models can be used, but are

more complex and require more site-specific input. The Level I fugacity model requires several input parameters including water solubility, vapor pressure, melting point, and $\log K_{OW}$ for prediction of steady-state concentrations of chemicals in air, water, suspended sediment, bed sediment, and biota. In using this model, the pesticide concentration in sediment or interstitial water is set at the chronic BSQC by adjusting the total mass of pesticide in the system. The model should be run over a range of parameter values that may affect equilibria, for example, OC levels or fish lipid levels; changing the concentration of solids or volume of water or air will not alter equilibrium. If there are no exceedances of criteria or levels of concern in other compartments in a series of Level I analyses, then no further analysis is necessary. However, if any potential exceedances are identified, then site-specific data should be obtained to allow for more refined modeling.

For all models used in this analysis, it is important to state all input parameters, conditions, and assumptions. The model outputs can then be compared to appropriate levels of concern established for the non-water compartments (e.g., WQC, USFDA action levels). If the steady-state concentrations in all compartments are acceptable, then the BSQC is acceptable. If the concentration in another compartment is projected to exceed a concentration of concern, then this should be indicated in the final criteria statement.

6.2 *Biota (bioaccumulation)*

Chemicals that accumulate in the sediments often also have a propensity to accumulate in organisms; this accumulation may lead to secondary poisoning effects as contaminants magnify up the food chain. This section provides guidance in order to address bioaccumulation in the derivation of BSQC.

This evaluation predicts whether sediments meeting chronic criteria could possibly lead to secondary poisoning of wildlife or human health effects resulting from the bioaccumulation of pesticides in fish or other prey. Only chronic criteria are evaluated because bioaccumulation occurs over an extended period of time and bioaccumulation potential is calculated with the assumption that the system is at apparent equilibrium. This evaluation requires toxicity values that demonstrate adverse effects from dietary intake of pesticides for wildlife and the availability of US Food and Drug Administration (USFDA) action levels for human health.

The first step is to evaluate whether the chemical of concern has the potential to bioaccumulate. According to the OECD (1995a), substances with a $\log K_{OW}$ or $K_d > 3$, a molecular weight < 1000 , a molecular diameter $< 5.5 \text{ \AA}$ and/or a molecular length $< 5.5 \text{ nm}$ may bioaccumulate, and these are similar properties as described for chemicals likely to accumulate in sediments. If a chemical has one or more of the above characteristics or the chemical has been shown to bioaccumulate in well conducted studies, the potential

for bioaccumulation and secondary poisoning should be evaluated (TenBrook et al. 2010).

The risk of secondary poisoning to terrestrial wildlife from sediment contaminants can be assessed by calculating the sediment concentration that would not lead to adverse effects on terrestrial predator organisms, referred to as the $NOEC_{\text{sediment}}$. The $NOEC_{\text{sediment}}$ is calculated with the assumption of a simplified food web in which the pesticide transfers from sediment to a fish or benthic species (prey) to a terrestrial animal (predator). The parameters required for this calculation are a biota-sediment accumulation factor for a fish or benthic species ($BSAF_{\text{prey}}$), a biomagnification factor for a fish or benthic species (BMF_{prey}), and an oral (ingestion) NOEC for a terrestrial predator ($NOEC_{\text{oral_predator}}$).

Wildlife toxicity studies for species that have significant food sources in water, such as mallard ducks, should be evaluated using Table 12. Studies that rate relevant/reliable (R) or less relevant/less reliable (L) may be used in the calculation of $NOEC_{\text{sediment}}$. A chronic NOEC is the preferred wildlife toxicity value, but sub-acute toxicity values may be used if a NOEC is not available. If multiple studies reporting the same type of toxicity value are available for a single species, the geometric mean of the values should be computed and used in the final calculation. The most sensitive species should be used if data for multiple species are available. Three common oral wildlife toxicity values are described below:

- 1) Acute (LC_{50}): one time dose, usually force fed (oral gavage/intubation), and the toxicity value is reported as mg/kg body weight. Since this value is expressed per body weight, rather than as a feed concentration, it is not recommended for use in this section.
- 2) Sub-acute (LC_{50}): in which the compound is administered in the feed to the animals for 2 weeks to several months, and the toxicity value is usually reported as mg/kg in feed.
- 3) Chronic (NOEC and LOEC): similar to the exposure conditions in the sub-acute study, but effects on reproduction parameters are monitored.

The BSAF, which is equal to the ratio of the tissue concentration to the sediment concentration, should be based on lipid-normalized tissue concentrations and OC-normalized sediment concentrations (Schwarzenbach et al. 2003). It is preferred that measured concentrations in the two media are reported in BSAF studies rather than estimated concentrations, but estimated BSAFs may be used if measured BSAFs are not available. If multiple BSAFs are available for a single species, the geometric mean should be computed. A biomagnification factor accounts for accumulation of the pesticide in the prey organism and is equal to the ratio of pesticide concentration in a

predator to pesticide concentration in a prey species. If an appropriate BMF is not available, default values based on the log K_{ow} are available (Table 13).

The following equations, adapted from the EU risk assessment technical guidance document (ECB 2003), can be used to calculate the $NOEC_{\text{sediment}}$ for wildlife:

$$\text{Eq 24: } NOEC_{\text{sediment}} = NOEC_{\text{oral_predator}} / (BSAF_{\text{prey}} \times BMF_{\text{prey}})$$

or

$$\text{Eq 25: } NOEC_{\text{sediment}} = LC_{50,\text{oral_predator}} / (BSAF_{\text{prey}} \times BMF_{\text{prey}})$$

A similar equation is used to calculate the $NOEC_{\text{sediment}}$ for human health protection:

$$\text{Eq 26: } NOEC_{\text{sediment}} = \text{USFDA action level} / (BSAF_{\text{food item}} \times BMF_{\text{prey}})$$

where

$BSAF_{\text{food item}}$ is the biota-sediment accumulation factor for a benthically-coupled fish.

Examples of benthically-coupled fish include channel catfish (*Ictalurus punctatus*), fathead minnow (*Pimephales promelas*), medaka (*Oryzias latipes*), weakfish (*Cynoscion regalis*), spot (*Leiostomus xanthurus*), croaker (*Micropogonias undulatus*), white perch (*Morone americana*), summer flounder (*Paralichthys dentatus*), and scup (*Stenomus chrysops*) (Tracey and Hansen 1996).

The $NOEC_{\text{sediment}}$ is compared to the chronic BSQC; if it is below the criterion, this information should be indicated in the final criteria statement to alert environmental managers that the BSQC may not be protective of all beneficial uses based on the bioaccumulation potential and additional review may be needed. If the $NOEC_{\text{sediment}}$ exceeds the chronic BSQC, then this assessment indicates there is little potential for harm to terrestrial predators if the chronic BSQC is not exceeded in sediments.

7 Criteria Summary

7.1 Final criterion statement

The final statement should be accompanied by a short summary of the derivation process used to calculate the criteria and include any important considerations that should be made by policy makers. Criteria should be stated as follows (based on USEPA 1985, 2003a; ASTM (2013):

Aquatic life should not be affected unacceptably if the 28-day average concentration of (1) does not exceed (2) $\mu\text{g/g}$ OC in sediment or (3) $\mu\text{g/L}$ in interstitial water more than once every three years on average and if the 10-day average concentration does not exceed (4) $\mu\text{g/g}$ OC in sediment or (5) $\mu\text{g/L}$ in interstitial water more than once every three years on average.

where:

- (1) - insert name of chemical
- (2) - insert the chronic criterion given as an OC-normalized sediment concentration
- (3) – insert the chronic criterion given as an interstitial water concentration
- (4) - insert the acute criterion an OC-normalized sediment concentration
- (5) – insert the acute criterion given as an interstitial water concentration

Depending on the magnitude of the criteria and if interstitial water toxicity values were analyzed using SPME or Tenax, the units of the criteria may need to be adjusted. For example, if there were DOC-normalized interstitial water concentrations available to calculate the criteria, the units may be ng/g DOC. If a K_{OC} was used to convert between OC-normalized sediment concentrations and interstitial water concentrations, it should be reported along with the criteria statement, as well as all associated citations if a geometric mean of multiple K_{OC} s was calculated. These averaging periods and the frequency of exceedance may be modified if data and/or models become available that can scientifically defend altering them.

7.2 *Assumptions and Limitations*

The assumptions, limitations and uncertainty involved in criteria derivation should be included in the criteria methodology and associated reports so that environmental managers are able to make informed decisions based on the accuracy and confidence in the criteria. In the criteria report, any data limitations that affected the criteria calculation, such as missing taxa requirements, should be summarized. The goal is to make the derivation process and reasoning behind the process transparent. A list of assumptions associated with using a SSD are provided in the UCDM (TenBrook et al. 2010).

7.3 *Comparison to existing criteria*

The derived BSQC should be compared to any other available sediment quality criteria from other methods or jurisdictions and summarized in the criterion report.

8 Derivation of bifenthrin BSQC

8.1 Introduction

Bifenthrin is a pyrethroid insecticide that has been detected in sediments throughout the Sacramento and San Joaquin Rivers watershed and linked to sediment toxicity in both urban and agricultural drainages (Amweg et al. 2006, Holmes et al. 2008, Weston et al. 2004). Pyrethroids are widely used in agricultural and urban settings for control of invertebrate pests. The pyrethroid insecticides are hydrophobic compounds that quickly partition to sediments and particulates in the environment and are moderately persistent. These compounds are nerve agents that cause over-excitation of the neurons, leading to paralysis and ultimately death. Aquatic invertebrates are particularly sensitive to pyrethroids because they disrupt osmoregulation (Clark and Matsumura 1982). In addition to lethality, sublethal toxic effects of pyrethroids, such as reduced growth, altered behavior and endocrine reproductive effects have also been documented, which may contribute to a decrease in an organism's survival, growth or reproduction (Werner and Moran 2008).

Bifenthrin sediment criteria are calculated and presented as an illustration of the BSQC derivation methodology outlined in this report. Current limitations to the criterion calculation are discussed and rationale is provided as to how to best proceed under such conditions. Acute and chronic water quality criteria calculated via the UC Davis method are available for bifenthrin (Fojut et al. 2012, Palumbo et al. 2010). The first sections (8.2 - 8.5) summarize information that was gathered for the WQC report: basic information about bifenthrin, physicochemical property data, environmental and metabolic fate, and human and wildlife dietary values. The literature was reviewed for current information not included in these sections and updated where appropriate. Following these introductory sections, sediment exposure data is summarized (sections 8.6 and 8.7) and the criteria calculations are described (sections 8.8 and 8.9). The remaining sections describe potential water quality effects (section 8.10) and compare other types of ecotoxicity data to the derived criteria (section 8.11) and check that the BSQC will not lead to adverse effects in other phases (section 8.12). Finally, the bifenthrin BSQC and the major assumptions and limitations inherent in the criteria are summarized (section 8.13).

8.2 Basic information

This section summarizes the basic information for bifenthrin, as identified in the bifenthrin WQC report (Fojut et al. 2012, Palumbo et al. 2010). In the future, if a pesticide has the potential to partition to sediments, it would be most efficient to derive

both water and sediment criteria simultaneously to prevent repeated summaries of information that are relevant to both types of criteria. The chemical structure of bifenthrin and its stereoisomers is presented in **Figure 3**.

Bifenthrin is identified by the following CAS and IUPAC names, and with the following trade names identified in the WQC report (Palumbo et al. 2010):

CAS: (2-methyl[1,1'-biphenyl]-3-yl)methyl (1*R*,3*R*)-rel-3-[(1*Z*)-2-chloro-3,3,3-trifluoro-1-propenyl]-2,2-dimethylcyclopropanecarboxylate

IUPAC: 2-methyl-3-phenylbenzyl (1*RS*)-cis-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

Trade names: Bifenthrin, bifenthrine, Bifentrin, Bifentrina, Biflex, Biphenthrin, Brigade, Capture, cyclopropanecarboxylic acid, FMC 54800, FMC 54800 Technical, Talstar, Tarstar, DeterMite, Biphenate, Torant (with Clofentezine), Zipak (with Amitraz) (EXTOXNET 1995; Kegley et al. 2008)

8.3 *Physicochemical data*

The physicochemical data presented in the bifenthrin WQC report (Palumbo et al. 2010) are summarized in Table 20. Calculation of geometric mean values for various physicochemical properties is detailed in the WQC report and not repeated here, except for the source and calculation of the geometric mean of the K_{OC} , as this has particular relevance to calculation of BSQC.

The updated acceptable source data used to calculate the geometric mean of the organic carbon – water adsorption coefficient and the dissolved organic carbon – interstitial water adsorption coefficient are presented in Table 21. The K_{OC} is used in the UCDSM to estimate interstitial water concentrations from OC-normalized sediment concentrations where necessary. The K_{DOC} may be used to estimate freely dissolved interstitial water concentrations from total interstitial water concentrations. Studies that determined the bifenthrin K_{OC} in marine sediments and marine interstitial waters were excluded in the data sets used to calculate the geometric means, as salt and fresh water data are to be treated separately in the UCDSM.

The reader is referred to Palumbo et al. (2010) for a complete summary of BCFs and environmental half-life values. No new values were found.

8.4 *Environmental and metabolic fate*

Bifenthrin is a nonpolar compound with low aqueous solubility, high lipid solubility (i.e., octanol-water partition coefficient; K_{OW}) and a high K_{OC} (Table 21). The

aqueous insolubility of bifenthrin predisposes it to partition out of water and sorb with strong affinity to sediment, soil particles, suspended matter and solids in general. Off-site movement of bifenthrin after application is unlikely unless bound to suspended particles or DOM in runoff water (Gan et al. 2005; Weston et al. 2004). Aquatic toxicity has been shown to decrease as a result of the presence of suspended particles, which have been suggested to limit the bioavailability of pyrethroids (Hill 1989; Muir et al. 1985).

Bifenthrin is stable to hydrolysis and very slowly undergoes photolysis in water (408 d; Laskowski 2002). Bifenthrin was shown to be more persistent under anaerobic soil conditions (half-life = 425 d, 179.5 d) compared to aerobic conditions (half-life = 96 d, 123 d) (Laskowski 2002; Kegley et al. 2008). Bifenthrin sediment half-lives ranged from 8 to 17 months at 20°C (Gan et al. 2005). Degradation of bifenthrin can occur under both biotic (microbe-mediated degradation) and abiotic (i.e., photolysis) conditions (Laskowski 2002; Lee et al. 2004).

8.5 *Human and wildlife dietary values*

There are currently no USFDA action levels for bifenthrin (USFDA 2000), but food tolerances are provided for human consumption of the meat of cattle, goat, hogs, horses, and sheep at 0.5 ppm (USEPA 2006a). There are currently no food tolerances for human consumption of fish.

Toxicity data for the mallard duck were used in the bifenthrin WQC report to assess if the derived criteria would be protective of wildlife (Fojut et al. 2012). The mallard duck toxicity values are also relevant for comparison to the derived BSQC for bifenthrin; as such, the toxicity values for the mallard duck are summarized here. An eight-day dietary LC₅₀ of 1280 mg/kg feed was reported for mallard ducklings (Fletcher 1983a) and a NOEC of 2150 mg/kg body weight has been reported for adult mallards (Fletcher 1983b). No effects to mallard ducks were observed 21 days after a single dose of pure bifenthrin was administered (Fletcher 1983b). A dietary NOEC of 75 mg/kg feed was reported based on no observed reproductive effects in 26 week old mallards, but this NOEC is likely an underestimation because it was the highest tested dose and no effects were observed at any exposure level over the 22-week dietary exposure (Roberts et al. 1986).

8.6 *Ecotoxicity data*

Fifteen original single species spiked-sediment toxicity tests with bifenthrin were identified and reviewed. Each study was rated for relevance and reliability. Relevance was rated according to Table 8 of the UCDSM. If the study rated relevant (R) or less relevant (L) then it was further evaluated for reliability. The reliability evaluation was based on a combination of documentation and acceptability scores calculated according

to Table 9 and Table 10 of the UCDSM. Studies that were rated relevant or less relevant and reliable or less reliable (RR, RL, LR, or LL) according to the method were summarized in the data summary sheets (formatted according to Table 14). Copies of completed summaries for all studies are included in [Appendix B](#) of this report. Data rated as acceptable (RR) and used directly in the acute criterion derivation are presented in Table 22. Studies that were rated RR but that were excluded in the prioritization process are presented in Table 23, including the reason for data exclusion. Supplemental studies rated as RL, LR or LL are used to evaluate the criteria to check that they are protective of particularly sensitive species and threatened and endangered species (summarized in Table 24). There were no studies identified that rated as N, LN, or RN.

Based on the data evaluation procedures, eight acute toxicity studies, yielding 17 toxicity values from two taxa, were judged reliable and relevant (RR; Table 22 and Table 23). No relevant and reliable chronic sediment toxicity studies were identified. Eleven studies reported toxicity values that were rated RL, LL, or LR and were used as supplemental information for evaluation of the derived criteria in sections 8.11.1 and 8.11.3 (Table 24).

Mesocosm and field studies evaluated for derivation of the bifenthrin WQC are also relevant to BSQC derivation for bifenthrin. Five mesocosm, microcosm and ecosystem (field and laboratory) studies were rated R or L according to Fojut et al. (2012) and are summarized in Table 25. Three relevant studies on effects of bifenthrin on wildlife were identified and reviewed for consideration of bioaccumulation in section 8.12.2.

8.7 *Data prioritization*

Multiple toxicity values for bifenthrin for the same species were reduced to one species mean toxicity value according to procedures described in the UCDSM (section 2.5). The final acute data set contains two SMAVs and is shown in Table 22. Acceptable acute data were prioritized and some were excluded for reasons including: standard endpoints are preferred over non-standard endpoints, more sensitive endpoints were available for the species, and tests conducted at standard conditions are preferred over those conducted at non-standard conditions (Table 23). There are currently no chronic SSTT data available for bifenthrin.

8.8 *Acute criterion calculation*

Two of the five taxa required to construct a species sensitivity distribution were available for bifenthrin, thus an assessment factor was used to calculate the acute BSQC. The epibenthic crustacean requirement is represented by the amphipod *H. azteca*, and the benthic insect category is represented by *C. dilutus*. The three missing taxa are an

infaunal invertebrate, a mollusk/amphibian/other unrepresented phylum, and a benthic invertebrate from an unrepresented family.

The acute criterion is calculated by first dividing the lowest SMAV in the acceptable (RR) data set by an assessment factor, which results in an estimate of the 5th percentile of the SSD (section 3.5.2). The lowest SMAV for bifenthrin was 0.65 µg/g OC, which is equal to the geometric mean of seven 10-d *H. azteca* LC₅₀s (Table 22). The AF is chosen based on the number of taxa in the data set; the AF for a data set with 2 taxa is 12 (Table 18). This 5th percentile is the recommended acute value, which is divided by two to derive the acute BSQC.

Interim Acute BSQC Calculation

Acute value = lowest SMAV ÷ assessment factor

$$= 0.65 \mu\text{g/g} \div 12$$

$$= 0.054 \mu\text{g/g OC}$$

Interim Acute BSQC = acute value ÷ 2

$$= 0.054 \mu\text{g/g OC} \div 2$$

$$= 0.027 \mu\text{g/g OC}$$

Interim Acute BSQC = 0.027 µg/g OC

$$= 27 \text{ ng/g OC}$$

8.9 *Chronic criterion calculation*

Due to the dearth of chronic data in both the acceptable and supplemental data sets for bifenthrin, no SMCVs could be calculated and thus the ACR procedure is used to calculate the chronic criterion for this compound (section 3.6.3). The lack of chronic sediment toxicity data for bifenthrin also prevents the calculation of an ACR by pairing appropriate acute and chronic spiked sediment toxicity studies. Because an experimental ACR cannot be calculated for bifenthrin, the chronic criterion is calculated with the default ACR of 11.4 (Table 19) and the acute value as follows:

Interim Chronic BSQC Calculation

Chronic BSQC = acute value ÷ ACR

$$= 0.054 \mu\text{g/g OC} \div 11.4$$

$$= 0.005 \mu\text{g/g OC}$$

$$\text{Interim Chronic BSQC} = 0.005 \mu\text{g/g OC}$$

$$= 5 \text{ ng/g OC}$$

8.10 Water Quality Effects

8.10.1 Bioavailability

Bioavailability is directly incorporated into the UCDSM by using bioavailability-based toxicity values to derive criteria. The rationale for the bioavailability approach to BSQC derivation is discussed in section 1.2.2. The BSQC are expressed OC-normalized sediment concentrations, and may be converted to freely dissolved interstitial water concentrations if desired to compare to interstitial water concentrations. If site-specific partition coefficients are available they can be used to convert between phases via Eq 2. If a site-specific partition coefficient is not available, then the geometric mean of acceptable partition coefficients can be used. To compare the OC-normalized sediment BSQC to relevant aqueous concentrations, the BSQC were converted to interstitial water concentrations using the K_{OC} of 524,000, which is the geometric mean of 10 values (Table 21). The resulting acute and chronic interstitial concentrations were 0.05 ng/L and 0.01 ng/L, respectively.

8.10.2 Mixtures

In general, additive mixture effects can be incorporated in criteria compliance using the concentration-addition model when it has been established that it is reasonable to assume additivity (section 4.2.1). When it is demonstrated or can be assumed that mixture effects will be additive, toxic unit analysis (section **Error! Reference source not found.**) is a simple way to check for compliance as long as there are BSQC available for each compound in the mixture. For non-additive mixture effects, interaction coefficients can be used if ample data are available (section 4.2.2). More complex mixtures, involving both synergists and antagonists cannot be incorporated into compliance determination at this time, although some complex models do exist to predict effects in these situations (section 4.2.3).

Bifenthrin often occurs in the environment with other pyrethroid pesticides in sediments of both urban and agricultural waterways in the Central Valley of California

(Amweg et al. 2005, 2006; Weston et al. 2005, 2008). All pyrethroids have a similar mode of action and several studies have demonstrated that pyrethroid mixture toxicity is approximately additive (Barata et al. 2006, Brander et al. 2009, Trimble et al. 2009). In a review paper that included derivation of water quality criteria for pyrethroids, Fojut et al. (2012) concluded that additivity of pyrethroid mixture toxicity is well-described in the literature and recommended that the concentration-addition method should be used for compliance determination to account for multiple pyrethroids in a sample. This is also the recommendation to determine BSQC compliance.

Although PBO is known to synergize the toxic effects of pyrethroids (Weston et al. 2006; Brander et al. 2009), no interaction coefficients (K) have been derived with relevant species to describe synergism between bifenthrin and PBO. Consequently, there is no accurate way to account for the interaction of bifenthrin and PBO in compliance determination.

Several other studies have tested mixture toxicity with various constituents, but interaction coefficients are not available for these combinations so they cannot be included in criteria compliance. One mesocosm study tested mixtures of atrazine and bifenthrin and found that the two compounds did not act synergistically and that if one pesticide was present at a high concentration, community-level effects of the other pesticide were masked (Hoagland et al. 1993). There is evidence that the presence of KCl does not affect partitioning or bioavailability of bifenthrin, but there appears to be a slight antagonistic mixture effect based on testing with *Hyaella azteca* and *Chironomus dilutus* (Trimble et al. 2010). This effect is likely caused by a physiological or toxicodynamic interaction. Carbon nanomaterials are becoming more common in consumer products, and joint toxicity of bifenthrin and a functionalized fullerene was investigated with *Daphnia magna* (Brausch et al. 2010). The researchers reported that the fullerene significantly increased bifenthrin acute toxicity but did not affect chronic endpoints; further study on these interactions is needed to incorporate them for criteria compliance.

8.10.3 Temperature, pH, and other water quality effects

The effects of temperature, pH, and other water quality parameters on the toxicity of bifenthrin were examined to determine if these are described well enough in the literature to incorporate into BSQC compliance (section 4.3). The effects of temperature and pH on pyrethroid toxicity were discussed previously in the bifenthrin WQC report (Fojut et al. 2012) and this discussion is also applicable to sediment toxicity. To summarize, there is an inverse relationship between temperature and the toxicity of pyrethroids (Miller and Salgado 1985; Werner and Moran 2008), and this relationship is likely the result of an increased sensitivity of the organism's sodium channel at lower temperatures (Narahashi et al. 1998). Pyrethroid contaminated sediments were more than twice as toxic to *H. azteca* when tested at 18°C compared to 23°C in the laboratory

(Weston et al. 2008). Weston et al. (2008) found that temperatures required in standard methods are likely higher than environmental temperatures and toxicity may be underestimated as a result of colder habitats. These results are not directly applicable for use in BSQC compliance because environmental samples were used, instead spiked sediment toxicity tests.

Despite the known effect of temperature on pyrethroid toxicity, there is not enough information to incorporate temperature effects in BSQC or compliance at this time. Also, no studies could be found that addressed pH or other water quality effects on bifenthrin toxicity in sediment or interstitial water. As a result, information is insufficient at this time to be able to incorporate the effects of water quality parameters into BSQC compliance.

8.11 Comparison of ecotoxicity data to derived criteria

8.11.1 Sensitive species

A data comparison was conducted to assess if the derived criteria for bifenthrin are protective of the most sensitive species. In the following, the derived BSQC are compared to toxicity values for the most sensitive species in both the acceptable (RR) and supplemental (RL, LR, LL) data sets as described in section 5.1.

The lowest reported acute sediment toxicity value in the RR data set is a 10-d LC₅₀ of 0.18 (0.16-0.20) µg/g OC for *H. azteca* (Picard 2010a). The interim acute BSQC of 0.027 µg/g OC is a factor of 7 below this toxicity value and the BSQC very protective based on this toxicity value.

The lowest toxicity value in the supplemental data set is a 10-d LC₅₀ of 0.0008 µg/g OC *Eohaustorius estuarius* (Anderson et al. 2008; Table 24). This value is below the interim acute BSQC of 0.027 µg/g OC by a factor of 34, however, it is an estuarine species so the criterion will not be adjusted downward.

The lowest species mean acute value in the RR data set is 0.65 µg/g OC for *H. azteca* (Table 22) and it was used directly in criteria derivation. Many of the SSTT studies used to calculate the acute BSQC also reported NOEC or LOEC values for the 10-day study. Since 10-day NOEC/LOECs do not meet the requirements for inclusion in the acute data set (which requires LC/EC₅₀s) or the chronic data set (which requires ≥ 28-d full or partial life cycle tests), these values were not used for derivation of BSQC, but are compared to the derived BSQC. The lowest MATC reported for *H. azteca* is 0.03 µg/g OC based on a 10-d growth endpoint (Picard et al. 2010a). The acute BSQC is very similar to this value, but is slightly below it. Because the MATC is presumed to be an

approximation of a no-effect concentration, the similarity of the values may indicate that the interim acute BSQC is reasonably protective and not overprotective.

The only available chronic SSTT data are for the saltwater species *Leptocheirus plumulosus* (Table 24). The 28-day growth MATC for bifenthrin was 3.7 µg/g OC (Putt 2005b). This value is well above the interim chronic BSQC and would be protective of *L. plumulosus*.

8.11.2 Ecosystem and other studies

In this section, the derived bifenthrin criteria are compared to acceptable laboratory, field, or semi-field multispecies studies (rated R or L), to determine if the criteria will be protective of ecosystems (section 5.3). Five bifenthrin microcosm, mesocosm and pond studies were identified and evaluated and all five studies rated R or L (Table 25).

Pond mesocosm studies performed by Drenner et al. (1993) and Hoagland et al. (1993) examined the effects of sediment-bound bifenthrin on zooplankton, phytoplankton, and fish. In these related studies, sediment was dosed with a formulation containing bifenthrin, but only water column concentrations of bifenthrin were reported. The 8-d LC₅₀ for gizzard shad, a sediment filter-feeder fish, was 207 ng/L based on the average concentration of the exposure duration (Drenner et al. 1993). Bluegill mortality (33%) occurred at an average maximum bifenthrin concentration of 3,150 ng/L (Hoagland et al. 1993). Gizzard shad may be more sensitive to bifenthrin than bluegills, or their feeding habits may have led to a higher exposure. In both studies, effects on the zooplankton community were reported, with higher bifenthrin concentrations resulting in a shift from crustaceans (copepods and/or cladocerans) to rotifers at concentrations of 90 ng/L and 39 ng/L (Drenner et al. 1993 and Hoagland et al. 1993, respectively).

A pond mesocosm study by Surprenant (1988) examined effects of sediment-bound bifenthrin on fathead minnows, daphnids, clams, and isopods (*Asellus* spp). At sediment treatment levels of 0.1, 0.3, and 1.0 mg/kg, no adverse effects were observed for fathead minnows or clams. Daphnid survival and reproduction was not affected at the lowest treatment, but catastrophic mortality was observed at the highest treatment level, which corresponded to a water column concentration of 1.86 µg/L. Isopods were the most sensitive taxon examined, with reduced survival at a treatment corresponding to a water column concentration of 0.30 µg/L and complete mortality a concentrations of 0.72 µg/L and 2.58 µg/L.

In a large-scale natural pond study, effects of bifenthrin spray drift and storm runoff from nearby aerial applications to cotton fields were reported for several taxa (Sherman 1989). Calanoid copepods were eliminated in the summer application period

and did not recover the following spring. Mayflies were also eliminated immediately following bifenthrin applications, and demonstrated only limited recovery the following season. Water striders (gerridae) and water beetles (gyrinidae) were also affected negatively. Other organisms were affected, but showed signs of recoveries in the year following bifenthrin application. Concentrations in water ranged 1.3-695 ng/L and in sediment ranged 13-8,173 µg/kg over the course of 14 months.

Auber et al. (2011) tested two pesticide application regimes that included multiple pesticides corresponding with wheat production in France. Bifenthrin was applied to the outdoor pond mesocosms in one of the regimes, along with eight other pesticides, over a 28 week exposure. Bifenthrin concentrations were measured in the water column, with an average exposure concentration of 46 ng/L. After a single application, bifenthrin was above the detection limit in the water column for an average of 5.5 ± 0.7 d in one treatment group and 9.2 ± 0.4 d in a second treatment group. Presumably the bifenthrin partitioned to the sediment and vegetation, was degraded or metabolized within 5-10 days of treatment, although other matrices were not analyzed. Decreased abundance of isopods (*Asellus aquaticus*) and amphipods (*Gammarus pulex*) was attributed to the bifenthrin treatment, and their decreased abundance was correlated to a decreased rate of leaf litter breakdown. The abundance of *A. aquaticus* recovered at 25 weeks post-treatment, while *G. pulex* abundance did not recover by the end of observation, 40 weeks post-treatment, although it should be noted that there were several applications of other pesticides during this period that may have also affected this taxon.

Sensitive taxa are relatively consistent in all of the mesocosm or pond studies available for bifenthrin; copepods, isopods, amphipods, and mayflies appear to be the taxa most severely affected by bifenthrin exposure, and recovery by these taxa may be limited or take months or years. All reported effect levels or measured concentrations are higher than the UCDM chronic WQC of 0.6 ng/L, as well as the interim chronic BSQC of 0.005 µg/g OC. The BSQC are considerably lower than the reported effects concentrations in the few studies that measured sediment concentrations. However, none of the reviewed studies report NOECs for any matrix or report effects concentrations as measured in sediment or interstitial water, so we cannot be certain that no effects would occur in mesocosms or aquatic ecosystems at criteria concentrations.

8.11.3 Threatened and endangered species

In this section, the derived criteria for bifenthrin are compared to toxicity values for threatened and endangered species to ensure that the criteria will be protective of these species (sections 5.2, TenBrook et al. 2009). Current records of state and federally listed threatened and endangered animal species in California were obtained from the

CDFW web site (<http://www.dfg.ca.gov/biogeodata/cnddb/pdfs/TEAnimals.pdf>; CDFW 2013).

No listed threatened or endangered species are included in the acceptable and supplemental data sets used for bifenthrin BSQC derivation (Table 22 and Table 24). Similar to the WQC report (Palumbo et al. 2010), no data were found for effects of bifenthrin on federally endangered crustaceans and insects, or acceptable surrogates (i.e., in the same family). In the WQC report, the lowest toxicity value for a threatened or endangered species was an LC₅₀ of 0.15 mg/L for *Oncorhynchus mykiss* that was used in the bifenthrin WQC derivation calculation (Palumbo et al. 2010). The acute and chronic BSQC were converted to interstitial concentrations of 0.05 ng/L and 0.01 ng/L, respectively, to compare to this aqueous value. The acute and chronic BSQC are far below this toxicity value. Based on the little available data, there is no evidence that the interim acute and chronic bifenthrin BSQC will be under-protective of threatened or endangered species but this assessment lacks chronic data and data for crustaceans and insects, which are considered the most sensitive species.

8.12 Harmonization with other environmental media

8.12.1 Water

The BSQC were converted from OC-normalized sediment concentrations to interstitial water concentrations to compare them to existing water quality criteria. The K_{OC} of 524,000, which is the geometric mean of 10 values (Table 21), was used as the partition coefficient. The resulting acute and chronic BSQC interstitial concentrations were 0.05 ng/L and 0.01 ng/L, respectively. The bifenthrin acute and chronic WQC are 4 ng/L and 0.6 ng/L, respectively, which are above the BSQC concentrations. Therefore, if the BSQC were attained it would be unlikely that the WQC would be exceeded due to desorption from sediment, if equilibrium conditions are assumed.

8.12.2 Biota

Based on the mean log K_{OW} of bifenthrin of 6.0 and its molecular weight of 422.87 g/mol, bifenthrin has the potential to bioaccumulate (section 6.2). In the UCDM WQC report, the accumulation of bifenthrin in food items to levels that are known to cause harm to their predators was examined to ensure WQC were protective (Fojut et al. 2012). To assess the risk of secondary poisoning, the BAF (28,000 L/kg, McAllister 1988) and the NOEC values for mallard (75 mg/kg feed; Roberts et al. 1986) and humans (0.5 mg/kg; USEPA 2006a) were used to roughly estimate water concentrations that would equate to no-effect levels for consumption of fish by terrestrial wildlife or by humans (Fojut et al. 2012). The estimated NOECs were 267 ng/L for mallard duck and

23 ng/L for humans. The chronic bifenthrin WQC and interstitial water BSQC (0.6 ng/L and 0.01 ng/L, respectively) are below these values, indicating that compliance with the BSQC should not conflict with other efforts to protect wildlife or human health from bifenthrin exposure.

8.13 Bifenthrin Criteria Summary

8.13.1 Assumptions, limitations and uncertainties

The assumptions, limitations and uncertainties involved in criteria derivation should be available to inform environmental managers of the accuracy and confidence in the derived criteria (section 7.2). This section summarizes any data limitations that affected the procedure used to determine the final bifenthrin criteria.

For the bifenthrin acute BSQC, a major limitation was the lack of acute SSTT data for freshwater species other than *H. azteca* and *C. dilutus*. Three of the five taxa requirements of the UCDSM were not met, and as such, an assessment factor approach was used to calculate the acute BSQC. The major limitation for the bifenthrin chronic BSQC derivation was the lack of any freshwater species in the chronic toxicity data set. None of five taxa requirements were met, which precluded the use of a SSD; therefore, an ACR was used to derive the chronic criterion. Since no acceptable experimental ACRs were available for bifenthrin in the literature, the default ACR of 11.4 was used. Particularly of concern was the lack of chronic data for *H. azteca*, which was the most sensitive species in the acute toxicity data set. Uncertainty cannot be quantified for either the acute or chronic criteria because they were not derived with a SSD.

To compare the OC-normalized sediment BSQC to relevant aqueous concentrations, the BSQC were converted to interstitial water concentrations using the K_{OC} of 524,000, which is the geometric mean of 10 values (Table 21). The resulting acute and chronic interstitial concentrations were 0.05 ng/L and 0.01 ng/L, respectively.

As concluded in the bifenthrin WQC report, increased bifenthrin toxicity as a result of lower temperatures still cannot be accounted for quantitatively (Fojut et al. 2012). An additional safety factor is not recommended to adjust criteria at this time but environmental managers should keep this factor in mind if derived criteria are not protective in colder water bodies.

Although greater than additive effects have been observed for mixtures of pyrethroids and PBO, there is insufficient data to account for this interaction in compliance determination. This is a significant limitation because formulations that contain both pyrethroids and PBO are available on the market. When additional highly rated data are available, the criteria should be recalculated to incorporate new research.

8.13.2 Comparison to existing criteria

To date, no USEPA sediment criteria or benchmarks are available for bifenthrin. The USEPA proposes an EqP-based approach, through which, the chronic WQC is used to predict the corresponding sediment concentration using the K_{OC} (Di Toro et al. 2002). The lowest SMAV in the acceptable sediment data set was converted to an interstitial water concentration to compare it to existing WQC. The lowest SMAV in the RR data set of 0.65 $\mu\text{g/g OC}$ for *H. azteca* (Table 22) was converted to an interstitial concentration of 1.24 ng/L using the geometric mean of K_{OC} s of 524,000. This value is compared to the chronic WQC for bifenthrin of 0.6 ng/L, which is approximately a factor of 2 lower than the lowest SMAV. Thus, the chronic WQC would likely be protective of short-term effects from sediment-associated bifenthrin. However, no chronic bifenthrin effects data are available, so it is unclear as to whether the chronic WQC would also be protective of long-term sublethal effects.

8.13.3 Bifenthrin interim criteria statement

The interim criteria statement is:

Aquatic life should not be affected unacceptably if the 28-day average concentration of bifenthrin does not exceed 0.005 $\mu\text{g/g OC}$ (5 ng/g OC) in sediment more than once every three years on average and if the 10-day average concentration does not exceed 0.27 $\mu\text{g/g OC}$ (27 ng/g OC) in sediment more than once every three years on average.

Although the criteria were derived to be protective of aquatic life in the Sacramento and San Joaquin Rivers, these criteria would be appropriate for any freshwater ecosystem in North America, unless species more sensitive than are represented by the species examined in the development of the present criteria are likely to occur in the ecosystems of interest.

The final acute criterion was derived using the AF procedure and the acute data used in criteria calculation are shown in Table 22. The chronic criterion was derived by use of a default ACR.

9 UCDSM Summary

After an extensive review of approaches used worldwide to derive SQC, the SSTT approach was used to develop the UCDSM because it has a strong technical foundation as the data clearly link cause and effect. It was noted, however, that there are very few SSTT data available for pesticides and experimental uncertainties may hinder

the use of what little data there are. The UCDSM represents a new approach to the derivation of SQC in that it uses single species, single chemical SSTT data that are based on the bioavailable sediment and/or interstitial water concentrations. Other jurisdictions that use the SSTT approach (CCME 1995) are not based on freely dissolved interstitial water concentrations, and instead use OC-normalized sediment concentrations; both types of concentrations are utilized in the UCDSM.

The UCDSM is based on the UCDM for deriving water quality criteria (TenBrook et al. 2010). Acute BSQC are derived using an assessment factor approach (< 5 taxa) or a species sensitivity distribution (> 5 taxa), depending on the number of taxa represented in the data set. The AFs derived for the UCDM have been updated and recalculated to include additional pesticide data for use in the UCDSM. Chronic BSQC are derived using an acute-to-chronic ratio (< 5 taxa) or a SSD (> 5 taxa), depending on data availability. The default acute-to-chronic ratio derived in the UCDM has also been updated with additional pesticide data and recalculated for inclusion in the UCDSM. Guidance on the collection, evaluation and prioritization of collected data for use in the UCDSM is adapted from the UCDM (TenBrook et al. 2010), as many procedures are applicable to both water column and sediment exposures. The derived BSQC are compared to effects data for sensitive species, ecosystem-level exposures, and threatened and endangered species to determine if the derived BSQC are adequately protective. Both the UCDSM and the UCDM provide guidance to assess the bioaccumulation of nonionic organic contaminants. As in the UCDM, the UCDSM also provides guidance to determine if water quality (pH, temperature) and mixture effects on toxicity can be incorporated into criteria compliance.

Since many jurisdictions worldwide incorporate sediment quality guidelines into an aquatic assessment framework, similar documents were evaluated to collect and gather quality data for use in the development of sediment and water quality criteria (RIVM, 2001; CCME 1995). The efforts by TenBrook et al. (2009, 2010) represent a comprehensive and robust protocol based on international guidance for collecting and evaluating physicochemical data that is applicable to both aquatic and sediment criteria derivation. As a result, the UCDSM is based on the same data collection and evaluation procedures as TenBrook et al. (2010).

Although SSTT data for a variety of taxa are not currently available, the UCDSM provides a framework to include current research on the estimation of the bioavailable fraction of chemicals as the most robust means of deriving sediment quality criteria. However, at this point the UCDSM is still just a framework because larger more diverse data sets must be tested with the method before BSQC should be used as firm regulatory values. For this reason, we have termed the resulting bifenthrin BSQC as interim values. There is a high degree of uncertainty in the values because they are based on so few data and species, so the qualifying term is appropriate.

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Tables

Table 1 Data sources for derivation of bioavailable sediment quality criteria.
Updated from TenBrook et al. (2010).

Source	Details/Notes
US Environmental Protection Agency (EPA) re-registration eligibility decision (RED) or interim RED (IRED)	Review compound RED or IRED and EPA Office of Pesticide Programs (OPP) database (www.ipmcenters.org/Ecotox/). Submit a Freedom of Information Act (FOIA) request for relevant studies by completing an 'affirmation of non-multinational status form' (www.epa.gov/pesticides/foia/affirmation.htm). Send the form with a list of the study master record identification numbers (MRIDs) and information about yourself and your employer to: hq.foia@epa.gov
California Department of Pesticide Regulation (CDPR)	Find relevant study numbers in the CDPR pesticide database: http://apps.cdpr.ca.gov/ereglib/ To retrieve studies, contact the registration branch of CDPR: Jacquelyn Rivers: jrivers@cdpr.ca.gov
California Department of Fish and Wildlife - Aquatic Toxicity Laboratory	Contact or check online for laboratory or criteria reports that may be available through CDPR
University Libraries	
Electronic databases	See Table 2 of this report for list and details
Handbooks:	
ECETOC 1993	Technical report no. 56 - Aquatic toxicity data evaluation
Mackay et al. 1997 (CD-ROM 1999)	Illustrated handbook of physical-chemical properties and environmental fate for organic chemical. Volume V. Pesticide chemicals
MITI 1992 (MITI = Ministry of International Trade and Industry, Japan)	Biodegradation and bioaccumulation data on existing data based on the CSCL Japan (CSCL = Chemical Substances Control Law)
Verschueren 2009 (book and CD-ROM)	Handbook of environmental data on organic chemicals, 5 th edition
Other sources	
Review articles including, e.g.; Laskowski 2002	Physical and chemical properties of pyrethroids
Biological effects database for sediments (BEDS)	BEDS may include spiked-sediment toxicity testing data: See Table 2 of this report
Internal databases	

Source	Details/Notes
International criteria documents/ government reports:	Often available via the Internet
Laboratory reports	
Manufacturer data	May be listed in RED/IREDD, EPA OPP database and available from EPA, information may be proprietary
Memos	May be listed in RED/IREDD, EPA OPP database and available from EPA
Registration packets	Studies used for pesticide registration may be listed in RED/IREDD, EPA OPP database and available from EPA, packets can be difficult to obtain

Table 2 Web addresses for various electronic resources used in derivation of bioavailable sediment quality criteria.
Updated from TenBrook et al. (2010).

Database	Description/contents	URL
BEDS (Biological effects database for sediments)	Biological effects databases including SSTT data from NOAA NSTP and EIM (previously FSEDQUAL but includes all WA monitoring data)	http://ccma.nos.noaa.gov/stressors/pollution/nsandt/ ; http://www.ecy.wa.gov/eim/index.htm
CLOGP (Calculated log P (estimated log K_{ow}))	K_{ow} calculator available through Bio-Loom	www.biobyte.com/bb/prod/bioloom.html
Biosis	Bibliographic database; multidisciplinary	http://thomsonreuters.com/products_services/science/science_products/a-z/biosis/
ChemFinder	Chemical database; chemical structures and names	www.chemfinder.com
Chemical Abstracts	Bibliographic database; primarily chemistry, life sciences	http://www.cas.org/
Current Contents	Bibliographic database; multidisciplinary	http://thomsonreuters.com/products_services/science/science_products/a-z/current_contents_connect/
ECOTOX (was AQUIRE)	Single chemical toxicity information for aquatic and terrestrial life	http://www.epa.gov/ecotox/
EFDB (Environmental Fate Data Base)	Access to Datalog, Biolog, Chemfate and Biodeg databases (below)	http://www.srcinc.com/what-we-do/efdb.aspx
Datalog	Bibliographic database; environmental fate	
Biolog	Microbial toxicity and biodegradation	
Chemfate	database	

Database	Description/contents	URL
Biodeg	Environmental fate and chemical-physical properties database Biodegradation database	
EXTOXNET (Extension Toxicology Network)	Pesticide profiles and toxicology information	http://extoxnet.orst.edu/
Estimation Program Interface (EPI) Suite	USEPA tools for estimation of numerous physical-chemical parameters	http://www.epa.gov/opptintr/exposure/pubs/episuite.htm
<i>K_{OW}</i> Win	<i>K_{OW}</i> program; Syracuse Research Corporation, New York, NY. Available at USEPA EPI Suite	http://www.epa.gov/opptintr/exposure/pubs/episuite.htm
LOG <i>K_{OW}</i>	<i>K_{OW}</i> database; Sangster Research Laboratories	http://logkow.cisti.nrc.ca/logkow/index.jsp
Pesticide Action Network	Bibliographic database; toxicity and regulatory information for pesticides	http://www.pesticideinfo.org/Index.html
PHYSPROP	Physical properties database including chemical structures and names	http://www.srcinc.com/what-we-do/product.aspx?id=133
OPP Pesticide Ecotoxicity Database	USEPA OPP toxicity database for registered pesticides, mostly unpublished studies, see EPA entry in Table 1 of this report	http://www.ipmcenters.org/Ecotox
POLTOX	Bibliographic database; Ovid; pollution and toxicology, plants, animals, and humans.	http://www.ovid.com
PubMed	Bibliographic database; medicine, life	http://www.ncbi.nlm.nih.gov/entrez/query.fcgi?DB=pubmed

Database	Description/contents	URL
	sciences, molecular biology, genetics, others	
TOXNET	Access to HSDB, TOXLINE, IRIS (below)	http://toxnet.nlm.nih.gov/
HSDB (Hazardous Substances Data Bank)	Toxicology database	
TOXLINE	Toxicology literature database	
IRIS (Integrated Risk Information System)	Database over hazard identification and dose-response assessments	
TSCATS (Toxic Substances Control Act Test Submission database)	Bibliographic database	http://www.srcinc.com/what-we-do/databaseforms.aspx?id=384
Web of Science	Bibliographic database; access to Institute for Scientific Information (ISI) Citation Databases	http://thomsonreuters.com/products_services/science/science_products/az/web_of_science/

EIM = Environmental Information Management (WA), FSEDQUAL = freshwater sediment quality database (WA), K_{ow} = octanol-water partition coefficient, NOAA = National Oceanic and Atmospheric Administration, NSTP = National Status and Trends Program, OPP = Office of Pesticide Programs, SSTT = spiked-sediment toxicity test, WA= Washington State

Table 3 Physicochemical data to be collected for derivation of bioavailable sediment quality criteria.

Based on TenBrook et al. (2010).

Parameter
BCF (bioconcentration factor)
BMF (biomagnification factor)
CAS (chemical abstract service number)
Chemical formula
Density
IUPAC (International Union of Pure and Applied Chemistry) name
K_H (Henry's law constant)
Log K_d (solid–water partition coefficient)
Log K_{DOC} (dissolved organic carbon–water partition coefficient)
Log K_{OC} (organic carbon–water partition coefficient)
Log K_{OW} (octanol–water partition coefficient)
Melting point
Molecular weight
pK _a (acid dissociation constant)
S (aqueous solubility)
Structure
$t_{1/2}$ (half-life), hydrolysis, photolysis, biotic degradation
Vapor pressure

Table 4 Ecotoxicity data to be collected for derivation of bioavailable sediment quality criteria.

Based on TenBrook et al. (2010).

Desired type of toxicity threshold/organism/experiment
Acute toxicity threshold concentrations (survival, immobilization)
Aquatic, infaunal, benthic and epibenthic insects
Aquatic plants
Bioavailability
Chemical mixtures
Chronic toxicity threshold concentrations (survival, growth, reproduction, embryonic/shell development, hatching, germination, behavior effects, enzyme inhibition, endocrine disruption, other physiological effects, insect control, changes in species diversity or abundance)
Field experiments
Fish
Insects
Laboratory experiments
Mesocosm experiments
Microcosm experiments
Multi-species
Non-insect aquatic, infaunal, benthic or epibenthic invertebrates
Single chemical
Single-species
Wildlife (mallard duck)
US Food and Drug Administration action levels (human health)

Table 5 Acceptable methods for determination of physicochemical parameters, other than the octanol-water partition coefficient, K_{ow} .
From TenBrook et al. (2010).

Constant	Method	Notes	Reference (method ID)
Bioconcentration factor, BCF	Flow-through; fish	Determines the apparent steady state BCF	OECD 1996 (305)
	Flow-through; fish and mollusks	Determines the apparent steady state BCF	ASTM E 2002a (1022-01)
Acid dissociation constant, pK_a	Conductometric	Onsager (1927) Equation must hold; acid/base dissociations, non-acid/base dissociations	OECD 1981 (112)
	Spectrophotometric	Solubility: low to high; differential UV/VIS absorption for ionized vs. unionized species, acid/base dissociations, non-acid/base dissociations	OECD 1981 (112)
	Titration	Solubility: moderate to high	OECD 1981 (112)
Hydrolysis rate constant, $t_{1/2}$, hydrolysis	Tiered approach	Determines the rate under acidic, basic and neutral conditions	ASTM 2001a (E 895 89)
	Tiered approach	Determines the rate under acidic, basic and neutral conditions	OECD 2004 (111)
Solid-water partition coefficient, K_{OC}	Batch equilibrium	Colloidal binding can reduce accuracy	ASTM 2001b (E 1195 01)
K_d , K_{OC}	Batch equilibrium	Colloidal binding can reduce accuracy	OECD 2000 (106)
Coefficient	Batch equilibrium co-solvent	Corrects for colloid binding	Evers & Smedes 1993
K_{OC}	HPLC	Estimation technique	OECD 2001 (121)
Solubility, S	Column elution	Solubility < 10 ⁻² g/L	OECD 1995b (105)
	Flask	Solubility > 10 ⁻² g/L	OECD 1995b (105)
	Flask	Solubility ≥ 1 mg/L	ASTM 2002b (E 1148 02)
	Generator column	Solubility < 1 mg/L	ASTM 2002b (E 1148 02)
	Nephelometric	Solubility ≥ 1 mg/L	ASTM 2002b (E 1148 02)

HPLC = high performance liquid chromatography, K_d = solid-water partition coefficient, K_{OC} = organic carbon-normalized solid-water partition coefficient.

Table 6 Acceptable experimental and computational techniques for determination of the octanol-water partition coefficient, K_{ow} , and the priority of use. Modified from TenBrook et al. 2010, based on Laskowski 2002; USEPA 2003a.

Log K_{ow} < 4		
Method	Reference (method ID)	Priority ^a
Slow stir	de Bruijn et al. 1989	1
Generator-column	USEPA 1996a	1
Shake-flask	USEPA 1996b	1
HPLC w/ extrapolation to 0% solvent	ASTM 1997 (E 1147-92)	2
HPLC w/o extrapolation to 0% solvent	ASTM 1997 (E 1147-92)	3
CLOGP program	Through Bio-Loom at www.biobyte.com	4
4 < Log K_{ow} < 6		
Method	Reference	Priority ^a
Slow stir	de Bruijn et al. 1989	1
Generator-column	USEPA 1996a	1
HPLC w/ extrapolation to 0% solvent	ASTM 1997 (E 1147-92)	2
HPLC w/o extrapolation to 0% solvent	ASTM 1997 (E 1147-92)	3
Shake-flask	USEPA 1996b	4
CLOGP program	Through Bio-Loom at www.biobyte.com	5
Log K_{ow} > 6		
Method	Reference	Priority ^a
Generator-column	USEPA 1996a	1
Slow stir	de Bruijn et al. 1989	2
HPLC w/ extrapolation to 0% solvent	ASTM 1997 (E 1147-92)	3
HPLC w/o extrapolation to 0% solvent	ASTM 1997 (E 1147-92)	4
Shake-flask	USEPA 1996b	5
CLOGP program	Through Bio-Loom at www.biobyte.com	6

CLOGP = calculated log P (estimated octanol-water partition coefficient), HPLC = high performance liquid chromatography.

^aPriority of 1 indicates highest priority.

Table 7 Data categories based on relevance and reliability scores for application in derivation of bioavailable sediment quality criteria.
 N = not relevant/not reliable; L = less relevant/reliable; R = relevant, reliable. Unshaded category is acceptable for criteria derivation, light shaded category is supplemental to criteria derivation and the dark shaded category is not acceptable.

		Reliability		
		0-59	60-73	74-100
Relevance	0-69	NN	NL	NR
	70-89	LN	LL	LR
	90-100	RN	RL	RR

Table 8 Relevance evaluation for single-species spiked sediment toxicity test data.
Based on ASTM 2013; ECOTOX 2006; TenBrook et al. 2010; USEPA 1996a, 2001.

Parameter	Score
Acceptable standard (or equivalent) method used	10
Endpoint linked to survival/growth/reproduction	15
Freshwater spiked-sediment toxicity test	15
Chemical \geq 80% pure	15
Species is in a family that resides in North America	15
Toxicity value calculated or calculable (e.g., LC ₅₀) that accounts for bioavailability*	15
Acceptable control response	15
Total	100

LC₅₀ = exposure concentration that is lethal to 50% of a test population.

*The toxicity value accounts for bioavailability if it is 1) a measured sediment concentration normalized to the organic carbon content, 2) a measured interstitial water concentration that is normalized to the dissolved organic carbon concentration, 3) an interstitial water concentration estimated via solid-phase microextraction or another non-depleting technique, or 4) a bioaccessible concentration estimated via Tenax ® or another depletion technique.

Table 9 Documentation evaluation for single-species spiked sediment toxicity test data. Full score is given if parameter is reported; 0 score is given if not. Based on ASTM 2013; ECOTOX 2006; TenBrook et al. 2010; USEPA 1996a, 2001.

Parameter	Score
Results published or in signed, dated format	6
Exposure duration	8
Control type (e.g., solvent, dilution water)	8
Organism information (i.e., age, life stage, etc.)	
Source	4
Age/life stage/size/growth phase	4
Chemical	
Grade or purity	5
Analytical method (if measured)	4
Nominal concentrations in interstitial water and/or sediment	2
Measured/estimated concentrations in interstitial water and/or sediment	10
Exposure type & renewal frequency	4
Overlying water	
Source	2
Hardness	1
Alkalinity	1
Conductivity	1
pH	1
Dissolved oxygen	2
Temperature	3
Photoperiod and/or light intensity	1
Sediment	
Particle size distribution	1
TOC	3
Sediment spike method	4
Sediment spike equilibration time	4

Parameter	Score
Statistics	
Methods identified	5
Hypothesis tests	
Statistical significance	2
Significance level	2
Minimum significant difference	2
% of control at NOEC and/or LOEC	2
Point estimates (e.g., LC ₅₀ , EC _x , etc.)	8
Total	100

EC₂₅ = exposure concentration that causes effect in 25% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, NOEC = no observed effect concentration, TOC = total organic carbon.

Table 10 Acceptability evaluation for single-species spiked sediment toxicity test data. Score is given if the parameter meets standard test guidance; a score of 0 is given if the parameter is not reported or does not meet test guidance.

Adapted from ECOTOX 2006; TenBrook et al. 2010.

Parameter	Score
Acceptable standard (or equivalent) method used (e.g., ASTM, USEPA, OECD, Environment Canada)	5
Test was of appropriate duration	2
Control	
Appropriate (e.g., solvent control included, if carrier was used)	5
Response within test guidance	10
Chemical	
Purity > 80% pure	10
Measured concentrations within 20% of nominal	4
Sediment	
Sediment spike method	4
Spike equilibration time adequate (≥ 1 month)	6
Carrier solvent fully evaporated; score 4 if no solvent applied to sediment	4
Organisms	
Appropriate size/age/growth phase	3
No prior contaminant exposure	3
Organisms randomly assigned to test containers	1
Adequate number per replicate/appropriate cell density	2
Feeding appropriate to standard methods	3
Organisms properly acclimated and disease-free prior to testing	1
Exposure type and renewal frequency appropriate to chemical	2
Overlying water source acceptable	2
Hardness within organism tolerance and/or overlying water specifications	1
Alkalinity within organism tolerance and/or overlying water specifications	1
Dissolved oxygen $\geq 60\%$	5
Temperature within organism tolerance (3 pts) and/or test guidance and held to $\pm 1^\circ\text{C}$ (3 pts)	6

Parameter	Score
Conductivity within organism tolerance and/or overlying water specifications	1
pH within organism tolerance and/or overlying water specifications	1
Photoperiod and light intensity within organism tolerance and/or test guidance	1
Statistics	
Adequate number of concentrations	3
Random or random block design employed	2
Adequate replication (≥ 4 reps)	2
Appropriate spacing between concentrations (dilution factor ≥ 0.3)	2
Appropriate statistical method used	2
Hypothesis tests	
Minimum significant difference (MSD) below recommended upper bound	1
NOEC response reasonable compared to control	1
LOEC response reasonable compared to control	1
Point estimates	
LC/EC values calculable (i.e., no < or > results)	3
Total	100

ASTM = American Society for Testing and Materials, EC = effective concentration, LC = lethal concentration, LOEC = lowest observed effect concentration, NOEC = no observed effect concentration, OECD = Organisation for Economic Co-operation and Development, USEPA = United States Environmental Protection Agency.

Table 11 Documentation and acceptability evaluation for data derived from aquatic outdoor field and indoor model ecosystems experiments.

Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

Parameter ^a	Score ^b
Results published or in signed, dated format	5
Exposure duration and sample regime adequately described	6
Unimpacted site (Score 7 for artificial systems)	7
Adequate range of organisms in system (1° producers, 1°, 2° consumers)	6
Chemical	
Grade or purity stated	6
Concentrations measured/estimated and reported	8
Analysis method stated	2
Habitat described (e.g., pond, lake, ditch, artificial, lentic, lotic)	6
Water quality	
Source identified	2
Hardness reported	1
Alkalinity reported	1
Dissolved oxygen reported	2
Temperature reported	2
Conductivity reported	1
pH reported	1
Photoperiod reported	1
Organic carbon reported	2
Chemical fate reported	3
Geographic location identified (Score 2 for indoor systems)	2
Pesticide application	
Type reported (e.g., spray, dilutor, injection)	2
Frequency reported	2
Date/season reported (Score 2 for indoor systems)	2

Parameter ^a	Score ^b
Test endpoints	
Species abundance reported	3
Species diversity reported	3
Biomass reported	2
Ecosystem recovery reported	2
Statistics	
Methods identified	2
At least 2 replicates	3
At least 2 test concentrations and 1 control	3
Dose-response relationship observed	2
Hypothesis tests	
NOEC determined	4
Significance level stated	2
Minimum significant difference reported	2
% of control at NOEC and/or LOEC reported or calculable	2
Total	100

LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

^aCompiled from RIVM 2001, USEPA 1985 and 2003a, ECOTOX 2006, CCME 1995, ANZECC and ARMCANZ 2000, OECD 1995a, and van der Hoeven et al. 1997.

^bWeighting based on ECOTOX 2006 and on data quality criteria in RIVM 2001 and OECD 1995a.

Table 12 Documentation and acceptability rating for data derived from terrestrial laboratory/field experiments.

Score is given if the parameter is reported.

Adapted from ECOTOX 2006; Table from TenBrook et al. 2010.

Parameter ^a	Score ^b
Exposure duration	20
Control type	7
Organism information (i.e., age, life stage, etc.)	8
Chemical grade or purity	5
Chemical analysis method	5
Exposure type (i.e., dermal, dietary, gavage, etc.)	10
Test location (i.e., laboratory, field, natural, artificial)	5
Application frequency	5
Organism source	5
Organism number and/or sample number	5
Dose number	5
Statistics	
Hypothesis tests	
Statistical significance	5
Significance level	5
Minimum significant difference	3
% of control at NOEC and/or LOEC	3
Point estimates (i.e., LC ₅₀ , EC _x , etc.)	4
Total	100

EC_x = exposure concentration that causes effect in x% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, NOEC = no observed effect concentration.

^a Compiled from ECOTOX 2006 and van der Hoeven et al. 1997.

^b Weighting based on ECOTOX 2006.

Table 13 Default biomagnification factors (BMF) (ECB 2003).

$\log K_{ow}$	BMF
< 4.5	1
4.5 - < 5	2
5 - 8	10
> 8 - 9	3
> 9	1

Table 14 Toxicity Data Summary.

Developed from ASTM 2013; MacDonald and Ingersoll 2002; TenBrook et al. 2010; USEPA 1996a, 2001. If a parameter is not reported, NR should be specified as the value.

Study reference:

Relevance

Score:

Rating:

Reliability

Score:

Rating:

Parameter	Value	Notes
Results published or in signed, dated format (Y/N)		
Test method (e.g., ASTM E 1706-05)		
Phylum		
Class		
Order		
Family		
Genus		
Species		
Family present in North America (Y/N)		
Age/size at start of test/growth phase		
Source of organisms		
Have organisms been pre-exposed to contaminants? (Y/N)		
Were animals acclimated and disease-free? (Y/N)		
Were animals randomized? (Y/N)		
Were test vessels randomized? (Y/N)		
Test duration		
Additional test durations		
Effect 1		

Parameter	Value	Notes
Control response 1		
Effect 2		
Control response 2		
Effect 3		
Control response 3		
Temperature		
Exposure type (note renewal frequency)		
Photoperiod/light intensity		
Overlying water source		
pH		
Hardness		
Alkalinity		
Dissolved oxygen		
Conductivity		
TOC/DOC		
Ammonia-N		
Sediment source		
Organic carbon content		
Particle size distribution (sand, silt, clay)		
Sediment spike method		
Carrier solvent addition		Evaporated? (Y/N)
Sediment spike equilibration time		
Sediment chemical extraction/analysis method		
Was interstitial water monitored? (Y/N)		
Interstitial water isolation method (if performed)		

Parameter	Value	Notes
Interstitial water chemical extraction/analysis method		
DOC of interstitial water		
Feeding		
Purity of test chemical		
% Measured compared to nominal (for measured matrix)		
Were toxicity values calculated based on nominal or measured/estimated concentrations?		
Concentration of carrier (if any) in test solutions		
Concentration 1 nom/meas (units)		# of reps & # of individuals per vessel:
Concentration 2 nom/meas (units)		# of reps & # of individuals per vessel
Concentration 3 nom/meas (units)		# of reps & # of individuals per vessel
Concentration 4 nom/meas (units)		# of reps & # of individuals per vessel
Concentration 5 nom/meas (units)		# of reps & # of individuals per vessel
Concentration 6 nom/meas (units)		# of reps & # of individuals per vessel
Control description (e.g., blank, solvent)		# of reps & # of individuals per vessel
LC ₅₀ (units)		Method:
EC ₅₀ (units)		Method:
EC _x (units)		
NOEC (units)		Method: p: MSD:
LOEC (units)		Same as above
MATC (GeoMean NOEC,LOEC) (units)		

Parameter	Value	Notes
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% of control at NOEC

% of control at LOEC

ASTM = American Society for Testing and Materials, DOC = dissolved organic carbon, EC₅₀ = exposure concentration that causes effect in 50% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, MATC = Maximum acceptable toxicant concentration, meas = measured, MSD = minimum significant difference, NOEC = no observed effect concentration, nom = nominal, reps = replicates, p = p-value for statistical significance, TOC = total organic carbon, USEPA = United States Environmental Protection Agency.

Notes:

Reliability points taken off for:

Documentation (Table 9):

Acceptability (Table 10):

Table 15 Acute aquatic toxicity concentration data set used to derive updated assessment factors for the UC Davis Sediment Method. Toxicity concentrations ($\mu\text{g/L}$) are species mean acute values and are ordered from low to high (rank); expanded from TenBrook et al. 2010.

Rank	Ald ^a	Atr ^a	Bif ^b	Chl ^a	Cpf ^b	Cyf ^b	Cyp ^b	DDT ^a	Dia ^b	Diel ^a	Endos ^a	End ^a	Hept ^a	Lcy ^b	Lin ^a	Per ^b	Txp ^a
1	4	3000	0.0065	3	0.035	0.002	0.002	0.36	0.34	2.5	0.34	0.15	0.9	0.002	2	0.0211	0.8
2	4.5	5300	0.0843	6.3	0.0427	0.062	0.05	1.1	0.52	4.5	0.83	0.32	1.1	0.002	10	0.0896	1.3
3	6.1	6300	0.105	15	0.06	0.155	0.07	1.4	1.79	5	2.3	0.33	1.8	0.005	10.5	0.189	1.962
4	7.4	6700	0.15	26	0.0654	0.16	0.1	1.6	4.15	6.1	3.2	0.41	2.8	0.013	32	0.21	2
5	8	14700	0.35	26	0.1	0.251	0.147	1.7	4.3	8	3.7	0.44	7.8	0.026	32	0.21	2.3
6	9	20000	0.405	37	0.15	0.5	0.2	1.7	10.7	8.1	3.8	0.46	13.1	0.03	40	0.32	3
7	10	27000	1.6	40	0.22	0.998	0.2	1.9	16.82	10.8	5.8	0.47	23.6	0.038	44	0.664	3.1
8	16	49000	2.615	45	0.25	2.49	0.2	1.9	460	15	6	0.54	24	0.047	44	1.5	3.446
9	21	60000		56	1		0.6	2.4	723	20	88	0.69	26	0.078	45	1.58	3.7
10	27			57	4.7		0.683	2.6	1643	22	261	0.75	29	0.15	48	1.7	3.822
11	27			58	6		0.7	3	3198	24		0.76	42	0.16	55.6	1.71	4.874
12	28			59	8		0.9	3	4441	39		0.78	47.3	0.16	64	2.5	5.782
13	32			82	10		1	3.2	7804	41		0.85	61.3	0.2	67.1	2.71	6
14	34			190	15.96		2	3.5		130		1	78	0.27	68	3.34	6.7
15	42				178			3.9		213		1.1	81.9	0.3	83	4.16	10
16	45.9				806			4		250		1.2	101	0.4	90	5.4	10.12
17	50				2410			4.3		567		1.3	148	0.5	138	5.95	10.8
18	143							4.9		620		1.5	320	0.64	141.1	7.0	11.85
19	180							5		740		1.8		2.3	207	9.38	12
20								7.3				2.1		3.3	460		13
21								7.8				3.1			485		13
22								7.8				4.7			676		13.78
23								8				5.9					14.59
24								8.5				32					14.6
25								9.3				34					15.68
26								10				60					16.71

Rank	Ald ^a	Atr ^a	Bif ^b	Chl ^a	Cpf ^b	Cyf ^b	Cyp ^b	DDT ^a	Dia ^b	Diel ^a	Endos ^a	End ^a	Hept ^a	Lcy ^b	Lin ^a	Per ^b	Txp ^a
27								12				64					17.61
28								14				352					18
29								17									20
30								18									24
31								25									26
32								33									31.75
33								40									40
34								48									73.48
35								48									140
36								54									210
37								67									500
38								68									
39								175									
40								192									
41								362									

Ald = aldrin, Atr = atrazine, Bif = bifenthrin, Chl = chlordane, Cpf = chlorpyrifos, Cyf = cyfluthrin, Cyp = cypermethrin, DDT=dichlorodiphenyltrichloroethane, Dia = diazinon, Diel = dieldrin, Endos = endosulfan, End= endrin, Hept = heptachlor, Lcy = λ-cyhalothrin, Lin = lindane, Per = permethrin, Txp = toxaphene, UCDSM = University of California Davis sediment method, USEPA = United States Environmental Protection Agency.

^aUS Environmental Protection Agency water quality criteria documents (USEPA 1980a – g, 1986a-b, 2003c, 2005a; summarized in TenBrook et al. 2010)

^bUC Davis Method data (Palumbo et al. 2012, Fojut et al. 2012)

Table 16 Compilation of the 95th percentile of factors for subsets of 1-5 samples.
 Median values in the last row are the summary assessment factors for each sample size.

Subset size	5	4	3	2	1
Aldrin	5.55	5.76	6.58	10.28	10.28
Bifenthrin	2.45	2.45	2.45	2.45	2.45
Chlordane	1.79	4.42	4.78	4.78	4.78
Chlorpyrifos	8.70	12.66	237.33	237.33	237.33
Cyfluthrin	7.84	7.84	7.84	7.84	7.84
Cypermethrin	8.74	8.74	8.74	8.74	8.74
DDT	3.07	4.13	4.72	9.21	63.74
Diazinon	10.46	26.97	42.40	42.40	42.40
Dieldrin	3.42	6.97	12.36	79.26	234.60
Endosulfan	1.20	11.28	21.15	21.15	21.15
Endrin	3.30	4.73	5.16	137.70	137.70
Heptachlor	4.36	20.41	37.38	73.68	73.68
Lambda-cyhalothrin	31.96	31.96	134.57	134.57	43.25
Lindane	6.94	7.36	7.36	7.36	7.36
Permethrin	4.57	5.08	5.08	5.08	5.08
Toxaphene	5.61	7.73	8.18	13.46	117.80
Median (summary)	5.1	7.5	8	12	32

Table 17 Median 5th percentile toxicity value estimates for sample sizes of 1-5 acute toxicity values using summary assessment factors (AFs) from Table 16 Compilation of the 95th percentile of factors for subsets of 1-5 samples. Table 16.

All median species sensitivity distribution (SSD) 5th percentiles are Burr Type III unless otherwise noted. All values have units µg/L. Bold values are larger than the median SSD 5th percentile for the chemical.

Sample size	5	4	3	2	1	SSD 5 th percentile (median)	Lowest value in data set ^a
Summary AFs	5.1	7.5	8	12	32		
Aldrin	1.23	1.01	1.08	1.03	0.58	4.86	4
Bifenthrin	0.0013	0.00086	0.00081	0.00055	0.00020	0.0027 ^b	0.0065
Chlordane	1.95	1.46	1.65	1.31	0.64	8.37	3
Chlorpyrifos	0.014	0.011	0.020	0.029	0.025	0.025	0.035
Cyfluthrin	0.0029	0.0020	0.0019	0.0012	0.00047	0.0079 ^b	0.0023
Cypermethrin	0.0056	0.0039	0.0037	0.0027	0.0010	0.023	0.0027
DDT	0.23	0.18	0.21	0.21	0.15	0.85	1.1
Diazinon	0.13	0.13	0.22	0.29	0.30	0.40	0.34
Dieldrin	0.87	0.76	0.94	1.16	4.41	3.15	2.5
Endosulfan	0.067	0.08	0.12	0.18	0.19	0.28	0.34
Endrin	0.074	0.058	0.067	0.091	0.12	0.23	0.15
Heptachlor	0.27	0.23	0.30	0.37	0.28	0.64	0.9
Lambda-cyhalothrin	0.0013	0.0011	0.0012	0.00098	0.00051	0.0037	0.0023
Lindane	2.27	1.72	1.85	1.28	0.56	6.52	2
Permethrin	0.0135	0.0097	0.0097	0.0072	0.0029	0.041	0.0211
Toxaphene	0.48	0.40	0.51	0.64	1.22	1.78	0.8

^aFrom Table 15.

^bThe median 5th percentile value is from a log-logistic distribution.

Table 18 Assessment factors used to calculate interim acute BSQC.

Number of required taxa ^a	Assessment Factor (AF)
1	32
2	12
3	8
4	7.5
5	5.1

^aRequired taxa for use of a species sensitivity distribution (section 3.4.1.1), which are 1) an epibenthic crustacean, 2) a benthic insect, 3) an infaunal invertebrate, 4) a mollusk, amphibian, fish, or other unrepresented phylum, and 5) a benthic invertebrate from an unrepresented family.

Table 19 Calculation of default acute-to-chronic ratio (ACR).

Chemical	ACR
Chlordane ^a	14
Chlorpyrifos ^b	2.2
Cyfluthrin ^b	10.27
Diazinon ^b	2.3
Dieldrin ^a	8.5
Endosulfan ^a	3.9
Endrin ^a	4.0
Lambda-cyhalothrin ^b	4.73
Lindane ^a	25
Parathion ^a	10
Default ACR (80th percentile)	11.4

^aFrom Host et al. 1995; originally from US Environmental Protection Agency criteria documents.

^bFrom UC Davis criteria reports (Fojut et al. 2012; Palumbo et al. 2012)

Table 20 Physical-chemical properties of bifenthrin.

Property	Bifenthrin
Chemical formula	C ₂₃ H ₂₂ ClF ₃ O ₂
Chemical Abstract Service (CAS) number	82657-04-3
California Department of Pesticide Regulation chemical code	2300
Classification	EPA Class C Carcinogen ^a
Molecular weight	422.87
Density (g/mL)	1.24 (geomean, n=2)
Water solubility (mg/L)	0.001 (geomean, n=2)
Melting point (°C)	69.3 (geomean, n=2)
Vapor pressure (Pa)	2.41x10 ⁻⁵ (geomean, n=2)
Henry's law constant (K_H) (Pa m ³ mol ⁻¹)	0.24 (geomean, n=2)
Log-normalized organic carbon-water partition coefficient (log K_{OC})	5.40 (geomean, n=9)
Log-normalized octanol-water partition coefficient (log K_{OW})	6.00 ^b

^aEXTOXNET 1995

^bSangster Research Laboratories 2010

Table 21 K_{OC} and K_{DOC} geometric mean calculations for bifenthrin using acceptable values.

K_{OC}	K_{DOC}	Reference
237,000	-	Laskowski 2002
380,000	190,000	Xu et al. 2007
650,000	170,000	Xu et al. 2007
700,000	180,000	Xu et al. 2007
980,000	350,000	Xu et al. 2007
236,610	-	FOOTPRINT 2010
110,000	250,000	Bondarenko et al. 2006
700,000	2,740,000	Bondarenko et al. 2006
4,265,795	-	Maul et al. 2008
501,187	-	Maul et al. 2008
524,118	334,225	Geometric mean
524,000	334,000	Rounded geometric mean (3 significant figures)
5.72	5.52	Log geometric mean

Table 22 Final acute toxicity data used to calculate bifenthrin bioavailable sediment quality criteria.
All studies were rated relevant and reliable (RR).

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/ size	Sediment LC/EC ₅₀ (95% CI) (µg/g OC)	% OC	Reference
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (AFDM)	3 rd instar	14.2 (10.9-16.5)	5.5	a
“	“	“	10	23	Growth (AFDM)	3 rd instar	6.96 (6.09-7.83)	2.3	b
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	Geometric mean				9.9		
<i>Hyalella azteca</i>	Amphipod	Hyal.	10	23±1	Survival	7 d	0.18 (0.16-0.20)	2.1	c
“	“	“	10	23±0.1	Survival	7-10 d	0.76 (0.69-0.85)	0.56	d
“	“	“	10	23±0.1	Survival	7-10 d	0.73 (0.62-0.84)	1.77	d
“	“	“	10	23±0.1	Survival	7-10 d	0.76 (0.69-0.85)	1.77	d
“	“	“	10	23±0.1	Survival	7-10 d	0.89 (0.74-1.04)	1.77	d
“	“	“	10	23±0.1	Survival	7-10 d	0.97 (0.82-1.10)	1.77	d
“	“	“	10	23±0.1	Survival	7-10 d	0.73 (0.62-0.84)	4.43	d
<i>Hyalella azteca</i>	Amphipod	Hyal.	Geometric mean				0.65		

LC₅₀ = exposure concentration lethal to 50% of a test population, EC₅₀ = exposure concentration that causes effect in 50% of a test population, CI: confidence interval, OC = organic carbon, Chir. = Chironomidae, SR = static renewal, IGR = instantaneous growth rate, Hyal. = Hyalellidae.

^aPutt 2005a, ^bPicard et al. 2010b, ^cPicard 2010a, ^dWeston & Jackson 2009.

Table 23 Acceptable acute toxicity data excluded in the data prioritization process for bifenthrin.
All studies were rated relevant and reliable (RR).

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	Sediment LC/EC ₅₀ (µg/g OC)	% OC	Converted interstitial water LC/EC ₅₀ (ng/L)	Measured/estimated interstitial water LC ₅₀ (ng/L)	Ref	Excl
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (IGR)	3rd instar	1.5 (1.2-1.6)	0.97	-	-	a	1
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Immobility	3 rd instar	2.2 (1.9-2.4)	0.97	-	-	a	1
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	3 rd instar	15.2	2.3	-	-	b	
"	"	"	10	23	Survival	3 rd instar	6.2 (5.1-8.7)	0.97	-	-	a	
"	"	"	10	23	Survival	2 nd -3 rd instar	29.0 (20.1-40.8)	1.44	-	48	c	
"	"	"	10	23	Survival	2 nd -3 rd instar	29.8 (19.5-50.8)	1.88	-	53	c	
"	"	"	10	23	Survival	2 nd -3 rd instar	18.3 (12.1-28.7)	5.03	-	48	c	
"	"	"			Geometric mean		11.7			50		2
<i>Hyalella azteca</i>	Amphipod	Hyal.	10	19.85 ±1	Survival	14-21 d	0.105	0.69	0.025 [†]	-	d	3

[†]A site-specific K_{OC} of 4,265,795 was available for this study to calculate the converted interstitial water concentration.

LC₅₀ = exposure concentration lethal to 50% of a test population, EC₅₀ = exposure concentration that causes effect in 50% of a test population, OC = organic carbon, Ref = reference, Excl. = reason for exclusion, Chir. = Chironomidae, Hyal. = Hyalellidae, SR = static renewal.

^aMaul et al. 2008a, ^bPicard et al. 2010b, ^cXu et al. 2007, ^dMaul et al. 2008b.

¹Nonstandard endpoint

²Data with more sensitive (standard) endpoint available

³Data at standard temperature available.

Table 24 Supplemental studies excluded from bifenthrin bioavailable sediment quality criteria derivation.
 Studies rated less relevant and/or less reliable: RL, LR, or LL.

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	LC/EC ₅₀ (µg/g OC)	% OC	Measured/estimate d interstitial water LC ₅₀ (ng/L)	MATC (ng/L)	Ref	Rating, Excl.
<i>Ampelisca abdita</i>	Amphipod	Ampeliscidae	10	20	Survival	NR	0.18	0.78	-	-	a	LL, 1
“	“	“	10	20	Survival	NR	0.067	0.78	-	-	a	LL, 1
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Immobility	3 rd instar	5.46	0.56	4.97	-	b	RL, 5
“	“	“	10	23	Immobility	3 rd instar	4.02	1.77	4.17	-	b	RL, 5
“	“	“	10	23	Immobility	3 rd instar	1.87	4.43	1.49	-	b	RL, 5
“	“	“	10	23	Immobility	3 rd instar	2.2 (1.9-2.4)	0.97	-	-	c	LR, 2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (AFDM)	3 rd instar	2.4 (1.6-2.8)	0.97	-	EC ₂₀ : 1.0 (0.7-1.3)	c	LR, 2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	2 nd instar	8.1	0.983	-	-	d	LR, 4
“	“	“	10	23	Survival	3 rd instar	9.0	0.56	8.84	-	b	RL, 5
“	“	“	10	23	Survival	3 rd instar	6.1	1.77	6.35	-	b	RL, 5

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	LC/EC ₅₀ (µg/g OC)	% OC	Measured /estimate d interstitial water LC ₅₀ (ng/L)	MATC (ng/L)	Ref	Rating, Excl.
“	“	“	10	23	Survival	3 rd instar	4.3	4.4 3	3.28	-	b	RL, 5
“	“	“	10	21-24	Survival	2 nd -3 rd instar	>2500	5.5	-	-	e	LR, 3
<i>Eohaustorius estuarius</i>	Amphipod	Haustori dae	10	15	Survival	NR	0.0012	00. 78	-	-	a	LL, 1
“	“	“	10	15	Survival	NR	0. 0008	00. 78	-	-	a	LL, 1
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23±1	Growth	7 d	> 0.37	2.1	-	-	f	RL. 3
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Immobility	7-10 d	0.631	0.5 6	0.400	-	b	LR, 5
“	“	“	10	23	Immobility	7-10 d	0.625	1.7 7	0.458	-	b	LR, 5
“	“	“	10	23	Immobility	7-10 d	0.391	4.4 3	0.448	-	b	LR, 5
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	18	Survival	7-10 d	0.450	0.0 187	-	-	g	RL, 5
“	“	“	10	23	Survival	7-14 d	0.197	0.9 83	-	-	d	LR, 4
“	“	“	10	23	Survival	7-10 d	0.26	1.7- 2.1	-	-	h	RL, 5
“	“	“	10	23	Survival	7-14 d	0.22	2.0 0	-	-	i	RL, 5
“	“	“	10	23	Survival	6-10 d	0.37	6.5	-	-	j	LL. 4,5
“	“	“	10	23	Survival	6-10 d	0.57	1.1	-	-	j	LL. 4,5

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	LC/EC ₅₀ (µg/g OC)	% OC	Measured /estimate d interstitial water LC ₅₀ (ng/L)	MATC (ng/L)	Ref	Rating, Excl.
“	“	“	10	23	Survival	6-10 d	0.63	1.4	-	-	j	LL, 4,5
“	“	“	10	23	Survival	7-10 d	0.99	0.0 187	-	-	g	RL, 5
“	“	“	10	23	Survival	7-10 d	0.784	1.7 70	0.540	-	b	RL, 5
“	“	“	10	23	Survival	7-10 d	0.829	0.5 60	0.563	-	b	RL, 5
“	“	“	10	23	Survival	7-10 d	0.592	4.4 30	0.594	-	b	RL, 5
<i>Leptocheirus plumulosus</i>	Amphipod	Aoridae	28	24-27	Survival	Neonates	5.9	4.1	-	2.0	k	LR, 1
<i>Leptocheirus plumulosus</i>	Amphipod	Aoridae	28	24-27	Growth	Neonates	3.7	4.1	-	2.0	k	LR, 1

Aor. = Aoridae, Chir. = Chironomidae, EC₅₀ = exposure concentration that causes effect in 50% of a test population, Excl. = reason for exclusion, Hyal. = Hyalellidae, K_{OC} = organic carbon-normalized solid-water partition coefficient, LC₅₀ = exposure concentration lethal to 50% of a test population, OC = organic carbon, Ref = reference, S = static, SR = static renewal.

^aAnderson et al. 2008, ^bHarwood et al. 2013a, ^cMaul et al. 2008a, ^dTrimble et al. 2010, ^ePutt 2005a, ^fPicard et al. 2010a, ^gWeston et al. 2009b, ^hAmweg & Weston 2007a, ⁱAmweg & Weston 2007b, ^jAmweg et al. 2005, ^kPutt 2005b.

¹Saltwater

²Control response not reported or not acceptable

³Effects reported as > value

⁴Toxicity value not based on measured bioavailable concentration

⁵Low reliability score

Table 25 Multispecies field, semi-field, laboratory, microcosm and/or mesocosm studies for bifenthrin.

Those rated R or L are used as supplemental information for bifenthrin bioavailable sediment quality criteria calculation. Same as for bifenthrin water quality criteria (Fojut et al. 2012).

Reference	Habitat design	Rating
Auber et al. 2011	Outdoor pond mesocosm	L
Drenner et al. 1993	Outdoor tank mesocosm	R
Hoagland et al. 1993	Outdoor tank mesocosm	R
Sherman 1989	Outdoor ponds	R
Surprenant 1988	Indoor laboratory microcosm	R

R = reliable.

Figures

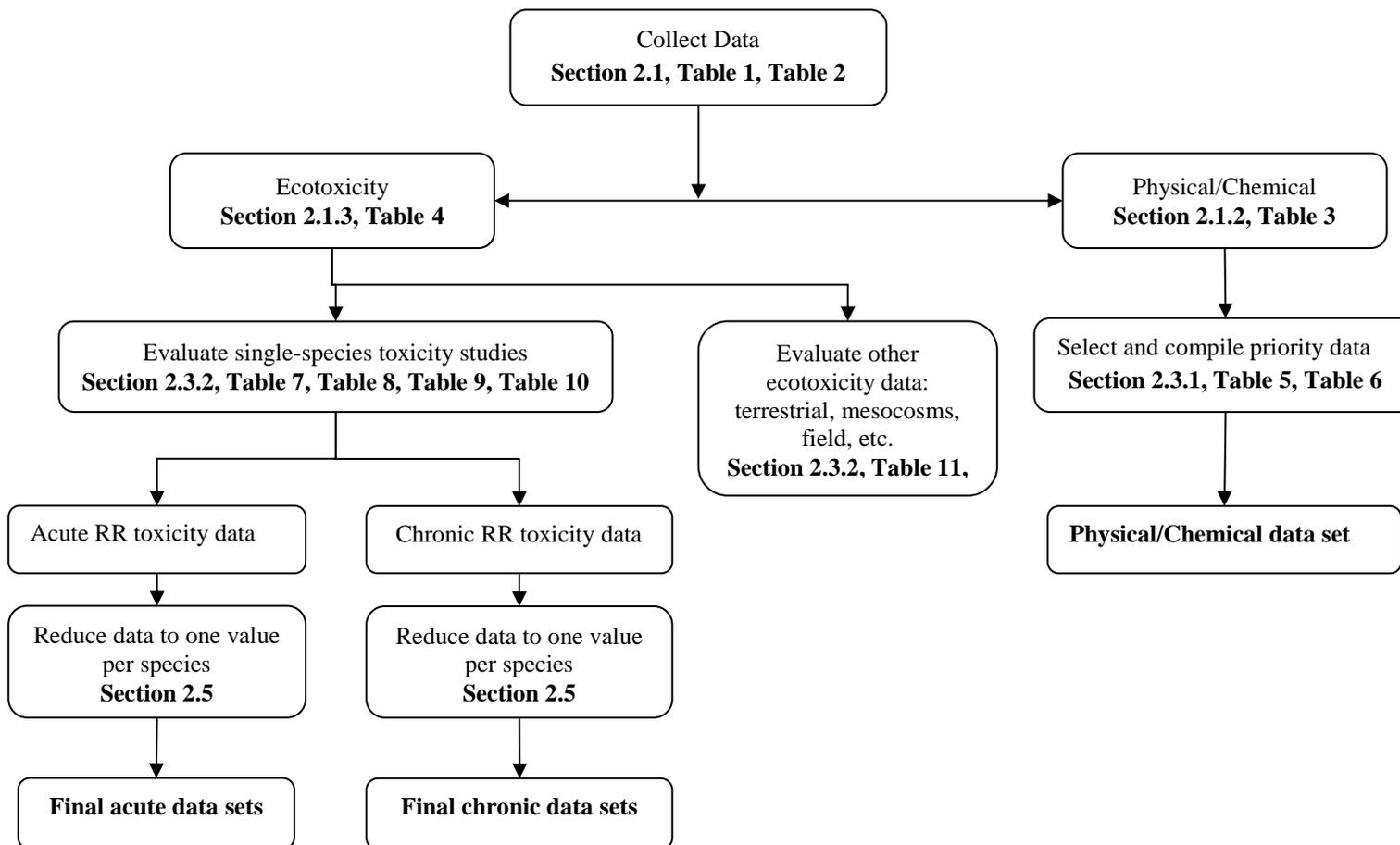


Figure 1 Flow chart for the collection, organization, and prioritization of data used to derive the bioavailable sediment quality criteria. Details on the process are found in the sections and tables listed in bold.

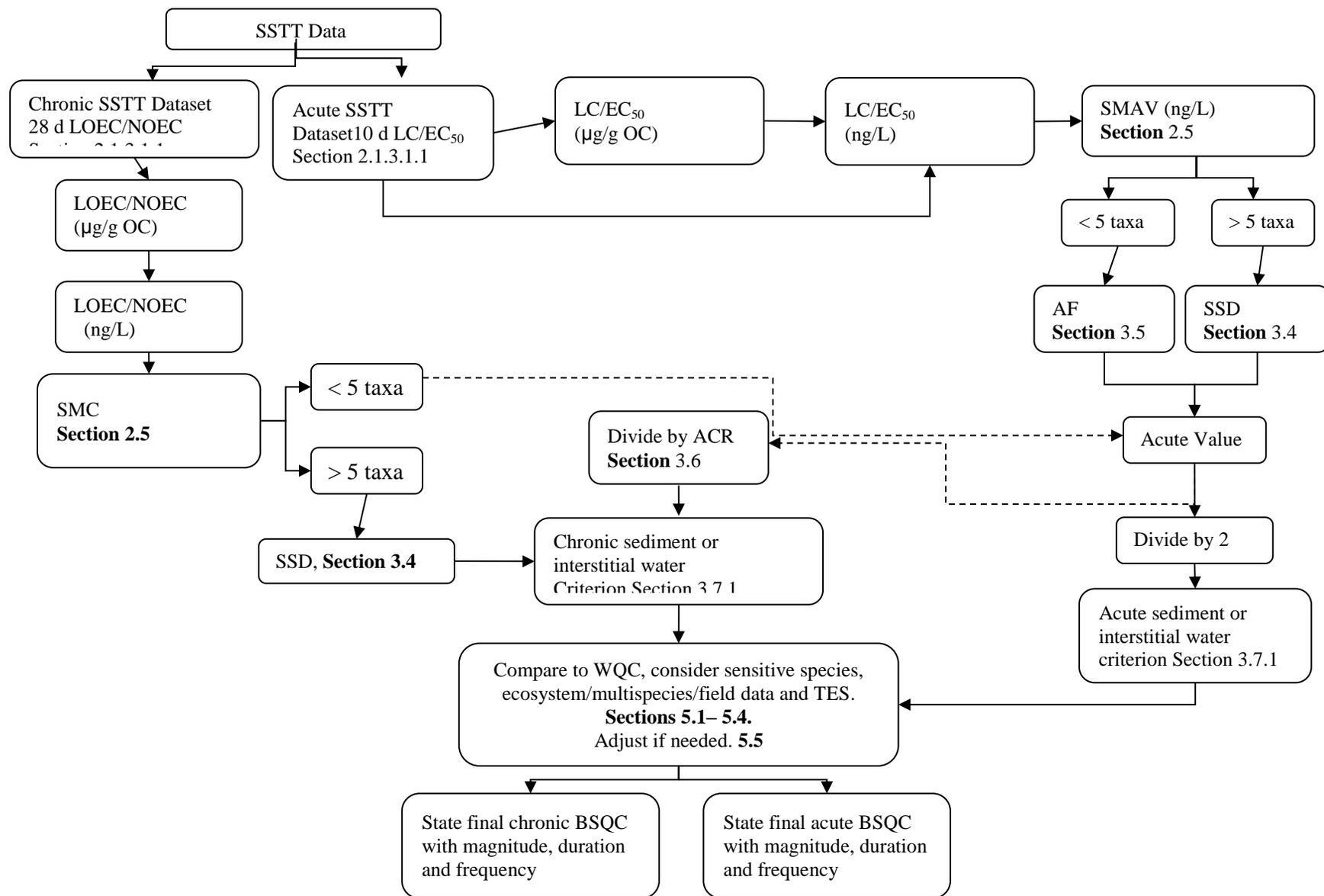


Figure 2. Criteria derivation flow chart for the UCDSM. Details on the process are found in the sections and tables listed in bold. Modified from TenBrook et al. (2010).

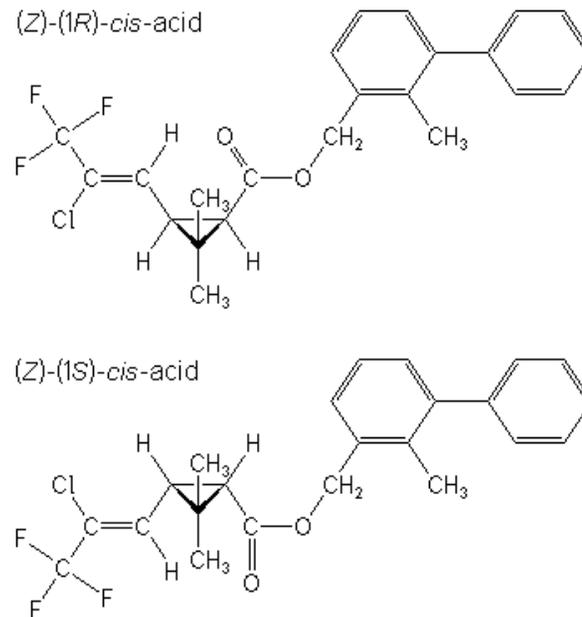


Figure 3 Structure of bifenthrin and stereoisomers (Wood 2008).

References

- Adams WJ, Kimerle RA, Mosher RG (1985) Aquatic safety assessment of chemicals sorbed to sediments. In: Cardwell RD, Purdy R, Bahner RC, eds, Aquatic Toxicology and Hazard Assessment: Seventh Symposium. STP 854. American Society for Testing and Materials, Philadelphia, PA, pp. 429-453
- Aldenberg T (1993) ETX 1.3a. A program to calculate confidence limits for hazardous concentrations based on small samples of toxicity data. National Institute for Public Health and the Environment (RIVM), Bilthoven, The Netherlands
- Amweg EL, Weston DP (2007a) Whole sediment toxicity identification evaluation tools for pyrethroid insecticides: I. Piperonyl butoxide addition. *Environ Toxicol Chem* 26:2389-2396
- Amweg EL, Weston DP (2007b) Whole sediment toxicity identification evaluation tools for pyrethroid insecticides: II. Esterase addition. *Environ Toxicol Chem* 26: 2397-2404.
- Amweg EL, Weston DP, Ureda N (2005) Use and toxicity of pyrethroid pesticides in the Central Valley, CA, USA. *Environ Sci Technol* 24:966-972
- Amweg EL, Weston DP, You J, Lydy MJ (2006) Pyrethroid insecticides and sediment toxicity in urban creeks from California and Tennessee. *Environ Sci Technol* 40:1700-1706
- Anderson BS, Hunt JW, Phillips BM, Nicely PA, Gilbert KD, de Vlaming V, Connor V, Richard N, Tjeerdema RS (2003) Ecotoxicologic impacts of agricultural drain water in the Salinas River, California, USA. *Environ Toxicol Chem* 22:2375-2384
- Anderson B, Lowe S, Phillips BM, Hunt JW, Vorhees J, Clark S, Tjeerdema RS (2008) Relative sensitivities of toxicity test protocols with the amphipods *Eohaustorius estuarius* and *Ampelisca abdita*. *Ecotoxicology and Environmental Safety* 69:24-31.
- Anderson BS, Phillips BM, Hunt JW, Connor V, Richard N, Tjeerdema RS (2006) Identifying primary stressors impacting macroinvertebrates in the Salinas River (CA, USA): Relative effects of pesticides and suspended particles. *Environ Poll* 141:402-408

- ANZECC and ARMCANZ (2000) Australian and New Zealand guidelines for fresh and marine water quality. Australian and New Zealand Environment and Conservation Council (ANZECC) and Agriculture and Resource management Council of Australia and New Zealand (AMRCANZ), Canberra, Australia
- ASTM (1997) Standard test method for partition coefficient (n-octanol/water) estimation by liquid chromatography. Annual book of standards, E 1147-92. American Society for Testing and Materials (ASTM), West Conshohocken, PA
- ASTM (2001a) Practice for determination of hydrolysis rate constants of organic chemicals in aqueous solutions. Annual book of standards, E 895-89. American Society for Testing and Materials (ASTM), West Conshohocken, PA
- ASTM (2001b) Test method for determining a sorption constant (K_{OC}) for an organic chemical in soil and sediments. Annual book of standards, E 1195-01. American Society for Testing and Materials (ASTM), West Conshohocken, PA
- ASTM (2002a) Guide for conducting bioconcentration tests with fishes and saltwater bivalve mollusks. Annual book of standards, E 1022-01. American Society for Testing and Materials (ASTM), West Conshohocken, PA
- ASTM (2002b) Test method for measurements of aqueous solubility. Annual book of standards, E 1148-02. American Society for Testing and Materials (ASTM), West Conshohocken, PA
- ASTM (2007) Standard guide for conducting whole sediment toxicity tests with amphibians. American Society of Testing and Materials (ASTM). ASTM designation: E 2591-07
- ASTM (2013) ASTM Standard E1706-05(2010). Standard tests method for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates. American Society of Testing and Materials (ASTM) International, West Conshohocken, PA
- ASTM (2011) Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode, D7363-11. American Society for Testing and Materials (ASTM), West Conshohocken, PA
- Auber A, Roucaute M, Togola A, Caquet T (2011) Structural and functional effects of conventional and low pesticide input crop-protection programs on benthic macroinvertebrate communities in outdoor pond mesocosms. *Ecotoxicol* 20:2042-2055

- Bailey HC , Deanovic L , Reyes E , Kimball T , Larson K , Cortright K , Connor V , Hinton DE (2000) Diazinon and chlorpyrifos in urban waterways in Northern California, USA . Environ Toxicol Chem 19: 82 – 87
- Bacey J, Starner K, Spurlock F (2004) The Occurrence and Concentration of Esfenvalerate and Permethrin in Water and Sediment in the Sacramento and San Joaquin Watersheds. CDPR: EH04-01
- Barata C, Baird DJ, Nogueira AJA, Soares AMVM, Riva MC (2006) Toxicity of binary mixtures of metals and pyrethroid insecticides to *Daphnia magna* Straus. Implications for multi-substance risks assessment. Aquat Toxicol 78:1-14
- Bondarenko S, Gan J (2004) Degradation and sorption of selected organophosphate and carbamate insecticides in urban stream sediments. Environ Toxicol Chem 23:1809-1814
- Bondarenko S, Gan J (2009) Simultaneous measurement of free and total concentrations of hydrophobic compounds. Environ Sci Technol 43:3772-3777
- Bondarenko S, Putt A, Kavanaugh S, Poletika N, Gan J (2006) Time dependence of phase distribution of pyrethroid insecticides in sediment. Environ Toxicol Chem 25: 3148-3154
- Bondarenko S, Spurlock F, Gan J (2007) Analysis of pyrethroids in sediment porewater by solid-phase microextraction. Environ Toxicol Chem 26:2587-2593
- Brander SM, Werner I, White JW, Deanovic LA (2009) Toxicity of a dissolved pyrethroid mixture to *Hyalella azteca* at environmentally relevant concentrations. Environ Toxicol Chem 28:1493-1499
- Brausch KA, Anderson TA, Smith PN, Maul JD (2010) Effects of functionalized fullerenes on bifenthrin and tribufos toxicity to *Daphnia magna*: Survival, reproduction, and growth rate. Environ Toxicol Chem 29:2600-2606
- Brennan AA, Harwood AD, You J, Landrum PF, Lydy MJ (2009) Degradation of fipronil in anaerobic sediments and the effect on porewater concentrations. Chemosphere 77:22-28
- Budd R, Bondarenko S, Haver D, Kabashima J, Gan J (2007) Occurrence and bioavailability of pyrethroids in a mixed land use watershed. J Environ Qual 36:1006-1012
- Burns LA (2004) Exposure Analysis Modeling System (EXAMS): User manual and system documentation. EPA/600/R-00/081

- Burr IW (1942) Cumulative frequency functions. *Ann Math Stat* 13:215-232.
- California SWRCB (2011) State Water Resources Control Board (SWRCB) website. [Accessed January 24, 2011]. Available from:
http://www.waterboards.ca.gov/about_us/water_boards_structure/mission.shtml
- Campbell E, Palmer MJ, Shao Q, Warne M, Wilson D (2000) BurrliOZ: a computer program for calculating toxicant trigger values for the ANZECC and ARMCANZ water quality guidelines. In: National Water Quality Management Strategy, Australian and New Zealand Guidelines for Fresh and Marine Water Quality. Australian and New Zealand Environment and Conservation Council and Agricultural and Resource Management Council of Australia and New Zealand, Canberra, Australia. Available at <http://www.cmis.csiro.au/Envir/burrlioz/>
- CCME (1995) Protocol for the derivation of Canadian sediment quality guidelines for the protection of aquatic life. Canadian Council of Ministers of the Environment (CCME). 1995 CCME EPC-98E
- CDFW (2013) State and federally listed endangered and threatened animals of California. California Natural Diversity Database. California Department of Fish and Wildlife, Sacramento, CA.
<http://www.dfg.ca.gov/biogeodata/cnddb/pdfs/TEAnimals.pdf>
- CDWR (1995) Compilation of sediment & soil standards, criteria & guidelines. Quality assurance technical document 7. California Department of Water Resources (CDWR). Sacramento, CA. Available from:
http://www.water.ca.gov/pubs/waterquality/municipal_wq_investigations/mwqi_technical_documents/compilation_of_soil_and_sediment_standards_criteria_and_guidelines/compilation_of_soil_and_sediment_standards_criteria_and_guidelines_february_1995.pdf
- Chapman PM, Faribrother A, Brown D (1998) A critical evaluation of safety (uncertainty) factors for ecological risk assessment. *Environ Toxicol Chem* 17:99-108
- Clark JM, Matsumura F (1982) Two different types of inhibitory effects of pyrethroids on nerve Ca^{2+} and Ca^{2+} Mg ATPase in the squid, *Loligo pealea*. *Pest Biochem Physiol* 18:180-190
- CRWQCB-CVR (2010) 2010 California 303(d) List of Water Quality Limited Segments. Central Valley Regional Water Quality Control Board web site. [Accessed September 19, 2012]. Available from:

http://www.waterboards.ca.gov/rwqcb5/water_issues/tmdl/impaired_waters_list/2008_2010_usepa_303dlist/20082010_usepa_aprvd_303dlist.pdf

CRWQCB-CVR (2011) The Water Quality Control Plan (Basin Plan) for the California Regional Water Quality Control Board Central Valley Region, fourth edition, the Sacramento River Basin and the San Joaquin River Basin. [Accessed September 21, 2012]. Available from:

http://www.waterboards.ca.gov/rwqcb5/water_issues/basin_plans/sacsjr.pdf

CSIRO (2001) BurrliOZ v. 1.0.13. Commonwealth Scientific and Industrial Research Organization, Australia. Available at: <http://www.cmis.csiro.au/Envir/burrlioz/>

Davies NA, Edwards PA, Lawrence MAM, Taylor MG, Simkiss K. Influence of particle surfaces on the bioavailability to different species of 2,4-dichlorophenol and pentachlorophenol. *Environ Sci Technol* 33:2465-2468.

de Bruijn J, Busser F, Seinen W, Hermens J (1989) Determination of octanol/water partition coefficients for hydrophobic organic chemicals with the slow stirring method. *Environ Toxicol Chem* 8: 499-412

de Perre C, Trimble AJ, Maul JD, Lydy MJ (2014) Ecological bioavailability of permethrin and p,p'-DDT: Toxicity depends on type of organic matter resource. *Chemosphere* 96:67-73

Diaz RJ, Rosenberg R (1996) The influence of sediment quality on functional aspects of marine benthic communities. In: Munawar M, Dave G (eds). *Development and progress in sediment quality assessment: Rationale, challenges, techniques and strategies*. SPB Academic Publishing, Amsterdam, The Netherlands

Dileanis PD, Bennett KP, Domagalski JL (2002) Occurrence and transport of diazinon in the Sacramento River, California, and selected tributaries during three winter storms, January-February, 2000. United States Geological Survey, Water-Resources Investigations Report 02-4101

Dileanis PD, Brown DL, Knifong DL, Saleh D (2003) Occurrence and transport of diazinon in the Sacramento River and selected tributaries, California, during two winter storms, January-February, 2001. United States Geological Survey, Water-Resources Investigations Report 03-4111

Ding Y, Landrum PF, You J, Harwood AD, Lydy MJ (2012a) Use of solid phase microextraction to estimate toxicity: Relating fiber concentrations to toxicity – Part I. *Environ Toxicol Chem* 31:2159-2167

- Ding Y, Landrum PF, You J, Harwood AD, Lydy MJ (2012b) Use of solid phase microextraction to estimate toxicity: Relating fiber concentrations to body residues – Part II. *Environ Toxicol Chem* 31:2168-2174
- Ding Y, Landrum PF, You J, Lydy MJ (2013) Assessing bioavailability and toxicity of permethrin and DDT in sediment using matrix solid phase microextraction. *Ecotoxicol* 22:109-117
- Ding Y, Weston DP, You J, Rothert AK, Lydy MJ (2011) Toxicity of sediment-associated pesticides to *Chironomus dilutus* and *Hyalella azteca*. *Arch Environ Contam Toxicol* 61:83-92
- Di Toro DM, Hansen DJ, DeRosa LD, Berry WJ, Bell HE, Reiley MC, Zarba CS (2002) Technical basis for the derivation of equilibrium partitioning sediment quality guidelines (ESGs) for the protection of benthic organisms: Nonionic organics. Draft report. 822-R-02-041. USEPA. Office of Science and Technology and Office of Research and Development, Washington, DC
- Domagalski JL, Weston DP, Zhang M, Hladik M (2010) Pyrethroid insecticide concentrations and toxicity in streambed sediment and loads in surface waters of the San Joaquin Valley, California, USA. *Environ Toxicol Chem* 29:813-823
- Drenner RW, Hoagland KD, Smith JD, Barcellona WJ, Johnson PC, Palmieri MA, Hobson JF (1993) Effects of sediment-bound bifenthrin on gizzard shad and plankton in experimental tank mesocosms. *Environ Toxicol Chem* 12:1297-1306
- Dussault EB, Balakrishnan VK, Solomon KR, Sibley PK (2008) Chronic toxicity of the synthetic hormone 17 alpha-ethinylestradiol to *Chironomus tentans* and *Hyalella azteca*. *Environ Toxicol Chem* 27:2521-2529
- Eadie FJ, Landrum PF, Faust WR (1982) Polycyclic aromatic hydrocarbons in sediments, pore water, and the amphipod *Pontoporeia hoyi* from Lake Michigan. *Chemosphere* 11:847-858
- ECB (2003) Technical guidance document on risk assessment in support of commission directive 93/67/EEC on risk assessment for new notified substances, commission regulation (EC) no. 1488/94 on risk assessment for existing substances, directive 98/8 EC of the European Parliament and of the council concerning the placement of biocidal products on the market. Part II. Environmental Risk Assessment. European Chemicals Bureau, European Commission Joint Research Center, European Communities

- ECETOC (1993) Technical report No. 56 - aquatic toxicity data evaluation. European Centre for Ecotoxicology and Toxicology of Chemicals, Brussels. Available at <http://www.ecetoc.org/technical-reports>
- ECOTOX (2006) ECOTOX code list. Report US Environmental Protection Agency, Washington DC
- Elson PF (1967) Effects on wild young salmon of spraying DDT over New Brunswick forests. *J Fish Res Board Can* 24:731–767
- Emans HJB, vander Plassche EJ, Canton JH, Okkerman PC, Sparenburg PM (1993) Validation of some extrapolation methods used for effects assessment. *Environ Toxicol Chem* 12:2139-2154
- Ensminger M, Bergin R, Spurlock F, Goh KS (2011) Pesticide concentrations in water and sediment and associated invertebrate toxicity in Del Puerto and Orestimba Creeks, California, 2007-2008. *Environ Monit Assess* 175:573-587
- EXTOXNET (1995) Pesticide information profile, bifenthrin. The Extension Toxicology Network. Oregon State University, Corvallis, OR.
<http://extoxnet.orst.edu/pips/bifenthr.htm>
- Finney DJ (1942) The analysis of toxicity tests on mixtures of poisons. *Ann Appl Biol* 29:82–94
- Fletcher DW (1983a) 8-day dietary LC₅₀ study with FMC 54800 technical in mallard ducklings. FMC Study No: A83/966. Bio-Life Associates, Ltd. Neillsville (WI), USA: submitted to U.S. Environmental Protection Agency. USEPA MRID: 00132535
- Fletcher DW (1983b) Acute oral toxicity study with FMC 54800 technical in mallard ducks. FMC Study No: A83/964. Bio-Life Associates, Ltd. Neillsville (WI), USA: submitted to U.S. Environmental Protection Agency. USEPA MRID: 00132534
- Fojut TL, Vasquez ME, Tjeerdema RS (2011) Methodology for derivation of pesticide sediment quality criteria for the protection of aquatic life. Phase I: Review of existing methodologies. Report prepared by the University of California Davis for the Central Valley Regional Water Quality Control Board. Available at: http://www.waterboards.ca.gov/rwqcb5/water_issues/tmdl/central_valley_projects/central_valley_pesticides/sediment_quality_criteria_method_development/ucd_sed_phase1final.pdf

- Fojut TL, Palumbo AJ, Tjeerdema RS (2012) Aquatic life water quality criteria derived via the UC Davis Method: II. Pyrethroid insecticides. *Rev Environ Contamin Toxicol* 216:51-103
- Fojut TL, Vasquez ME, Poulsen AH, Tjeerdema RS (2013) Methods for deriving pesticide aquatic life criteria for sediments. *Rev Environ Contamin Toxicol* 224:97-175
- FOOTPRINT (2010) European Commission - Framework Programme for Research and Development. University of Hertfordshire. Bifenthrin general information page. <http://sitem.herts.ac.uk/aeru/iupac/Reports/78.htm>
- Gan J, Lee SJ, Liu WP, Haver DL, Kabashima JN (2005) Distribution and persistence of pyrethroids in runoff sediments. *J Environ Qual* 34:836-841
- Gerstl Z (1990) Estimation of organic chemical sorption by soils. *J Contamin Hydrol* 6:357-375
- Ghosh U, Driscoll SK, Burgess RM, Jonker MTO, Teible D, Gobas F, Choi Y, Apitz SE, Maruya KA, Gala WR, Mortimer M, Beegan C (2014) Passive sampling methods for contaminated sediments: Practical guidance for selection, calibration, and implementation. *Integr Environ Assess Manage* DOI: 10.1002/ieam.1507
- Giddings JM (2006) Overview of sediment toxicity studies with synthetic pyrethroids. Performed by Compliance Services International 61 Cross Road Rochester, MA 02770. Sponsored by Pyrethroid Working Group c/o Fred Pearson Chair PWG Coordination Committee Syngenta Crop Protection 410 Swing Road Greensboro, NC 27409. DPR study ID: 238267. EPA MRID: 46871501
- Giddings JM, Solomon KR, Maund SJ (2001) Probabilistic risk assessment of cotton pyrethroids: II. Aquatic mesocosm and field studies. *Environ Toxicol Chem* 20:660-668
- Giesy JP, Solomon KR, Coats JR, Dixon KR, Giddings JM, Kenega EK (1999) Ecological risk assessment of chlorpyrifos in North American aquatic environments. *Rev Environ Contam Toxicol* 160:1-129
- Greenberg MS, Chapman PM, Allan IJ, Anderson KA, Apitz SE, Beegan C, Bridges TS, Brown SS, Cargill IV JG, McCulloch MC, Menzie CA, Shine JP, Parkerton TF (2014) Passive sampling methods for contaminated sediment: Risk assessment and management. *Integr Environ Assess Manage* DOI: 10.1002/ieam.1511

- Harwood AD, Landrum PF, Lydy MJ (2012) Can SPME fiber and Tenax methods predict the bioavailability of biotransformed insecticides? *Environ Sci Technol* 46:2413-2419.
- Harwood AD, Landrum PF, Lydy MJ (2013a) Bioavailability-based toxicity endpoints of bifenthrin for *Hyalella azteca* and *Chironomus dilutus*. *Chemosphere* 90:1117-1122
- Harwood AD, Landrum PF, Weston DP, Lydy MJ (2013b) Using SPME fibers and Tenax to predict the bioavailability of pyrethroids and chlorpyrifos in field sediments. *Environ Pollut* 173:47-51
- Hawthorne SB, Azzolina NA, Neuhauser EF, Kreitinger JP (2007) Predicting bioavailability of sediment polycyclic aromatic hydrocarbons to *Hyalella azteca* using equilibrium partitioning, supercritical fluid extraction, and porewater concentrations. *Environ Sci Technol* 41:6297-6304
- Hawthorne SB, Grabanski CB, Miller DJ, Kreitinger JP (2005) Solid-phase microextraction measurement of parent and alkyl polycyclic aromatic hydrocarbons in milliliter sediment pore water samples and determination of K_{DOC} values. *Environ Sci Technol* 39:2795-2803
- Hayes TB, Case P, Chui S, Chung D, Haeffele C, Haston K, Lee M, Mai VP, Marjua Y, Parker J, Tsui M (2006) Pesticide mixtures, endocrine disruption, and amphibian declines: Are we underestimating the impact? *Environ Health Perspect* 114:40-50
- Hill IR (1989) Aquatic organisms and pyrethroids. *Pest Sci* 27:429-465
- Hoagland KD, Drenner RW, Smith JD, Cross DR (1993) Freshwater community responses to mixtures of agricultural pesticides: effects of atrazine and bifenthrin. *Environ Toxicol Chem* 12:627- 637
- Holmes RW, Anderson BS, Phillips BM, Hunt JW, Crane DB, Mekebri A, Connor V (2008) Statewide investigation of the role of pyrethroid pesticides in sediment toxicity in California's urban waterways. *Environ Sci Technol* 42:7003-7009
- Hose GC, Van Den Brink PJ (2004) Confirming the species-sensitivity distribution concept for endosulfan using laboratory, mesocosm, and field data. *Arch Environ Contamin Toxicol* 47: 511-520
- Host GE, Regal RR, Stephan CE (1995) Analyses of acute and chronic data for aquatic life. US Environmental Protection Agency, Washington, DC
- Hrudey SE, Chen W, Rousseaux CG (1996) Bioavailability in Environmental Risk Assessment. Lewis Publishers, CRC Press. Boca Raton, FL.

- Hunter W, Xu YP, Spurlock F, Gan J (2008) Using disposable polydimethylsiloxane fibers to assess the bioavailability of permethrin in sediment. *Environ Toxicol Chem* 27:568-575
- Ingersoll CG, MacDonald DD, Wang N, Crane JL, Field LJ, Haverland PS, Kemble NE, Lindskoog RA, Severn C, Smorong DE (2001) Predictions of sediment toxicity using consensus-based freshwater sediment quality guidelines. *Arch Environ Contam Toxicol* 41:8-21
- Kegley SE, Hill BR, Orme S, Choi AH (2008) PAN Pesticide Database. Pesticide Action Network North America. San Francisco, CA. www.pesticideinfo.org
- Kelley K, Starner K (2004) Monitoring surface waters and sediments of the Salinas and San Joaquin River Basins for organophosphate and pyrethroid pesticides. <http://www.cdpr.ca.gov/docs/sw/swmemos.htm>
- Landrum PF, Robinson SD, Gossiaux DC, You J, Lydy MJ, Mitra S, ten Hulscher TEM (2007) Predicting bioavailability of sediment-associated organic contaminants for *Diporeia* spp. and oligochaetes. *Environ Sci Technol* 41:6442-6447
- Laskowski DA (2002) Physical and chemical properties of pyrethroids. *Rev Environ Contamin Toxicol* 174:49-170
- Li H, Sun B, Chen X, Lydy MJ, You J (2013) Addition of contaminant bioavailability and species susceptibility to a sediment toxicity assessment: Application in an urban stream in China. *Environ Pollut* 178:135-141
- Liess M, Schulz R, Liess MH-D, Rother B, Kreuzig R (1999) Determination of insecticide contamination in agricultural headwater streams. *Wat Res* 33: 239–247
- Lee S, Gan J, Kim J-S, Kabashima JN, Crowley DE (2004) Microbial transformation of pyrethroid insecticides in aqueous and sediment phases. *Environ Toxicol Chem* 23:1-6
- Long JLA, House WA, Parker A, Rae JE (1998) Micro-organic compounds associated with sediments in the Humber rivers. *Sci Total Environ* 210/211:229-253
- Lydy MJ, Landrum PF, Oen AMP, Allinson M, Smedes F, Harwood AD, Li H, Maruya KA, Liu J (2014) Passive sampling methods for contaminated sediments: State of the science for organic contaminants. DOI: [10.1002/ieam.1503]
- MacDonald DD, Ingersoll CG (2002) A guidance manual to support the assessment of contaminated sediments in freshwater ecosystems. Volume III- Interpretation of the results of sediment quality investigations. Great Lakes National Program

Office: Chicago, IL. EPA-905-B02-001-C. Available at:

<http://www.cerc.usgs.gov/pubs/sedtox/volumeIII.pdf>

- Mackay D (2001) Multimedia Environmental Fate Models: The fugacity approach, Second edition. Boca Raton, FL: Lewis Publishers
- MacKay D, Arnot JA, Wania F, Bailey RE (2011) Chemical activity as an integrating concept in environmental assessment and management of contaminants. *Integr Environ Assess Manage* 7:248-255
- MacKay D, Shiu W-Y, Ma K-C (1999) Illustrated handbook of physical-chemical properties and environmental fate for organic chemicals. CRC-LLC netbase, CD-ROM version
- Maltby L, Blake N, Brock TCM, van den Brink PJ (2005) Insecticide species sensitivity distributions: importance of test species selection and relevance to aquatic ecosystems. *Environ Toxicol Chem* 24:379-388
- Maul JD, Brennan AA, Harwood AD, Lydy MJ (2008a) Effect of sediment-associated pyrethroids, fipronil, and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization, and survival. *Environ Toxicol Chem* 27:2582-2590
- Maul JD, Trimble AJ, Lydy MJ (2008b) Partitioning and matrix-specific toxicity of bifenthrin among sediments and leaf-sourced organic matter. *Environmental Toxicology and Chemistry* 27:945-952
- Maund SJ, Travis KZ, Hendley P, Giddings JM, Solomon KR (2001) Probabilistic risk assessment of cotton pyrethroids: V. Combining landscape-level exposures and ecotoxicological effects data to characterize risks. *Environ Toxicol Chem* 20:687-692
- Mayer P, Tolls J, Hermens L, Mackay D (2003) Equilibrium sampling devices. *Environ Sci Technol* 37:184A-191A
- Mayer P, Parkerton TF, Adams RG, Cargill JG, Gan J, Gouin T, Gschwend PM, Hawthorne SB, Helm P, Witt G, You J, Escher BI (2014) Passive sampling methods for contaminated sediments: Scientific rationale supporting use of freely dissolved concentrations. *Integr Environ Assess Manage* DOI: [10.1002/ieam.1508]
- McAllister WA (1988) Full life cycle toxicity of 14C-FMC 54800 to the fathead minnow (*Pimephales promelas*) in a flow-through system. FMC Study No: A86-2100. EPA MRID: 40791301

- McLaughlin MJ, Lanno R (2014) Use of “bioavailability” as a term in ecotoxicology. *Integr Environ Assess Manage* 10:138-140
- Mehler WT, Li H, Pang J, Sun B, Lydy MJ, You J (2011) Bioavailability of hydrophobic organic contaminants in sediment with different particle-size distributions. *Arch Environ Contam Toxicol* 61:74-82
- Miller TA, Salgado VL (1985) The mode of action of pyrethroids on insects. In: *The Pyrethroid Insecticides*. ED. Leahey JP. Taylor & Francis, Philadelphia, PA
- MITI (1992) Biodegradation and bioaccumulation data of existing chemicals based on the CSCL Japan. Japan Chemical Industry Ecology-Toxicology & Information Center. Ministry of International Trade and Industry, Basic Industries Bureau, Chemical Products Safety Division
- Moore DRJ, Caux P-Y (1997) Estimating low toxic effects. *Environ Toxicol Chem* 16:794-801
- Moore MT, Schulz R, Cooper CM, Smith Jr S, Rodgers Jr JH (2002) Mitigation of chlorpyrifos runoff using constructed wetlands. *Chemosphere* 46:827-835
- Mu XY, LeBlanc GA (2004) Synergistic interaction of endocrine-disrupting chemicals: model development using an ecdysone receptor antagonist and a hormone synthesis inhibitor. *Environ Toxicol Chem* 23:1085-1091
- Muir DCG, Rawn GP, Townsend BE, Lockhart WL, Greenhalgh R (1985) Bioconcentration of cypermethrin, deltamethrin, fenvalerate, and permethrin by *Chironomus tentans* larvae in sediment and water. *Environ Toxicol Chem* 9:1045-1051
- Narahashi T, Ginsburg KS, Nagata K, Song JH, Tatebayashi H (1998) Ion channels as targets for insecticides. *Neurotoxicol* 19:581-590
- Niemi GJ, Devore P, Detenbeck N, Taylor D, Lima A, Pastor J, Yount JD, Naiman RJ (1990) Overview of case-studies on recovery of aquatic systems from disturbance. *Environ Manage* 14:571-587
- Nillos MG, Lin K, Gan J (2009) Enantioselectivity in fipronil aquatic toxicity and degradation. *Environ Toxicol Chem* 28:1825-1833
- NRC (2003) Bioavailability of contaminants in soils and sediments. Processes, tools and applications. National Research Council. Washington, DC, USA: National Academy Press. 420 p

- OECD (1981) Test No. 112: dissociation constants in water. OECD publishing. Available at <http://browse.oecdbookshop.org/oecd/pdfs/browseit/9711201E.PDF>
- OECD (1995a) OECD environment monographs No. 92, OECD environmental health and safety publications, series on testing and assessment, No. 3, guidance document for aquatic effects assessment. Organisation for Economic Co-operation and Development (OECD), Paris
- OECD (1995b) Test No. 105: water solubility. OECD publishing. Available at <http://www.oecd.org/dataoecd/17/13/1948185.pdf>
- OECD (1996) Test No. 305: bioconcentration: flow-through fish test. OECD publishing. Available at <http://browse.oecdbookshop.org/oecd/pdfs/browseit/9730501E.PDF>
- OECD (2000) Test No. 106: adsorption – desorption using a batch equilibrium method. OECD publishing. Available at <http://browse.oecdbookshop.org/oecd/pdfs/browseit/9710601E.PDF>
- OECD (2001) Test No. 121: estimation of the adsorption coefficient (K_{OC}) on soil and on sewage sludge using high performance liquid chromatography (HPLC). OECD publishing. Available at <http://browse.oecdbookshop.org/oecd/pdfs/browseit/9712101E.PDF>
- OECD (2004) Test No. 111: hydrolysis as a function of pH. OECD publishing. Available at <http://browse.oecdbookshop.org/oecd/pdfs/browseit/9711101E.PDF>
- Okkerman PC, vander Plassche EJ, Emans HJB, Canton JH (1993) Validation of some extrapolation methods with toxicity data derived from multiple species experiments. *Ecotoxicol Environ Saf* 25:341-359.
- Olmstead AW, LeBlanc GA (2005) Toxicity assessment of environmentally relevant pollutant mixtures using a heuristic model. *Integr Environ Assess Manage* 1:114-122
- OPP Pesticide Ecotoxicity Database. [Accessed 24 May 2011]. Available at: <http://www.ipmcenters.org/Ecotox/DataAccess.cfm>
- Palumbo AJ, Fojut TL, Brander SM, Tjeerdema RS (2010) Water quality criteria report for bifenthrin. Phase III: Application of the pesticide water quality criteria methodology. Report prepared for the Central Valley Regional Water Quality Control Board, Rancho Cordova, CA
- Palumbo AJ, TenBrook PL, Fojut TL, Faria IR, Tjeerdema RS (2012) Aquatic life water quality criteria derived via the UC Davis method: I. Organophosphate insecticides. *Rev Environ Contam Toxicol* 216:1-49

- Pape-Lindstrom PA, Lydy MJ (1997) Synergistic toxicity of atrazine and organophosphate insecticides contravenes the response addition mixture model. *Environ Toxicol Chem* 16:2415-2420
- Parkerton TF, Maruya KA (2014) Passive Sampling in contaminated sediment assessment: Building consensus to improve decision making. *Integr Environ Assess Manage* DOI: 10.1002/ieam.1488
- Phillips BM, Anderson BS, Hunt JW, Siegler K, Voorhees JP, Tjeerdema RS, McNeill K (2012) Pyrethroid and organophosphate pesticide-associated toxicity in two coastal watersheds (California, USA). *Environ Toxicol Chem* 31:1595-1603
- Phillips BM, Anderson BS, Voorhees JP, Hunt JW, Holmes RW, Mekebri A, Connor V, Tjeerdema RS (2010) The contribution of pyrethroid pesticides to sediment toxicity in four urban creeks in California USA. *J Pestic Sci* 35:302-309
- Picard CR (2010a) 10-Day toxicity test exposing freshwater amphipods (*Hyaella azteca*) to bifenthrin applied to formulated sediment under static-renewal conditions. Performed by Springborn Smithers Laboratories, Wareham, MA, Study No. 136565.6133; submitted to Pyrethroid Working Group, Washington, DC.
- Picard CR (2010b) 10-Day toxicity test exposing midges (*Chironomus dilutus*) to bifenthrin applied to formulated sediment under static-renewal conditions following OPPTS draft guideline 850.1735. Performed by Springborn Smithers Laboratories, Wareham, MA, Study No. 136565.6143; submitted to Pyrethroid Working Group, Washington, DC.
- Pignatello (1990) Slowly reversible sorption of aliphatic halocarbons in soils. I. Formation of residual fractions. *Environ Toxicol Chem* 9:1107-1115
- Plackett RL, Hewlett PS (1952) Quantal responses to mixtures of poisons. *J R Stat Soc Series B Stat Methodology* 14:141-163
- Putt AE (2005a) Bifenthrin – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Study performed by Springborn Smithers Laboratories, Wareham, MA, project ID: 136565.6106; submitted to Pyrethroid Working Group, Washington, DC. EPA MRID 46591502. DPR record number 238254.
- Putt AE (2005b) Bifenthrin – Toxicity to estuarine amphipods (*Leptocheirus plumulosus*) during a 28-day sediment exposure. Submitted to Pyrethroid Working Group Beveridge & Diamond 1350 I Street NW Washington, DC 20005. Performed by: Springborn Smithers Laboratories 790 Main Street Wareham, MA 02571-1037. DPR study ID: 238257. EPA MRID 46591501

- Qin S, Gan J (2006) Enantiomeric differences in permethrin degradation pathways in soil and sediment. *J Agricult Food Chem* 54:9145-9151
- Reichenberg F, Mayer P (2006) Two complementary side of bioavailability: Accessibility and chemical activity of organic contaminants in sediments and soils. *Environ Toxicol Chem* 25:1239-1245
- Rider CV, LeBlanc GA (2005) An integrated addition and interaction model for assessing toxicity of chemical mixtures. *Toxicol Sci* 87:520-528
- RIVM (2001) Guidance document on deriving environmental risk limits. Traas TP (ed) RIVM report 601501 012. National Institute for Public Health and the Environment, Bilthoven, The Netherlands
- Roberts NL, Phillips C, Anderson A, MacDonald I, Dawe IS, Chanter DO (1986) The effect of dietary inclusion of FMC 54800 on reproduction in the mallard duck. FMC Study No: A84/1260. EPA MRID: 00163099
- Sabljić A, Gusten H, Verhaar H, Hermens J (1995) QSAR modeling of soil sorption. Improvements and systematics of log K_{OC} vs. log K_{OW} correlations. *Chemosphere* 31:4489-4514
- Sangster Research Laboratories (2007) LOGKOW. A databank of evaluated octanol-water partition coefficients (Log P). Available online at <http://logkow.cisti.nrc.ca/logkow/index.jsp>, Canadian National Committee for CODATA
- Schwarzenbach RP, Gschwend PM, Imboden DM (2003) *Environmental Organic Chemistry*, 2nd edition. John Wiley & Sons, Inc., Hoboken, NJ
- Semple KT, Doick KJ, Jones KC, Burauel P, Craven A, Harms H (2004) Defining bioavailability and bioaccessibility of contaminated soil and sediment is complicated. *Environ Sci Technol* 38:228A-231A.
- Sherman JW (1989) Bifenthrin pond study: Ecological effects during treatment and post treatment follow-up studies of Hagan's pond, Orrville, Alabama. FMC report no. A84-1285-02 (January 25, 1989). Unpublished report prepared by the Academy of natural Sciences of Philadelphia for FMC Corporation. EPA MRID: 40981822
- Siepmann S, Finlayson B (2000) Water quality criteria for diazinon and chlorpyrifos. California Department of Fish and Game, Administrative Report 00-3, 59 p
- Solomon KR, Giddings JM, Maund SJ (2001) Probabilistic risk assessment of cotton pyrethroids: I. Distributional analyses of laboratory aquatic toxicity data. *Environ Toxicol Chem* 20:652-659

- Sormunen AJ, Tuikka AI, Akkanen J, Leppanen MT, Kukkonen JVK (2010) Predicting the bioavailability of sediment-associated spiked compounds by using the polyoxymethylene passive sampling and Tenax ® extraction methods in sediments from three river basins in Europe. *Arch Environ Contamin Toxicol* 58:80-90
- Starner K, White J, Spurlock F, Kelley K (2008) Assessment of pyrethroid contamination of streams in high-use agricultural regions of California. In: Gan J, Spurlock F, Hendley P, Weston DP (eds) *Synthetic Pyrethroids*. ACS Symposium Series, Washington DC, p. 72-83
- Stephan CE, Rogers JW (1985) Advantages of using regression analysis to calculate results of chronic toxicity tests. In: Bahner RC, Hansen DJ (eds) *Aquatic Toxicology and Hazard Assessment*. STP 891. American Society for Testing and Materials, Philadelphia, PA, pp. 377-387
- Sun K, Krause GF, Mayer FL, Eilersieck MR, Basu AP (1995) Predicting chronic lethality of chemicals to fishes from acute toxicity test data - theory of accelerated life testing. *Environ Toxicol Chem* 14:1745-1752
- Surprenant DC (1983) Acute toxicity of FMC 54800 technical to *Daphnia magna*. Bionomics Study. FMC Study No: A83-986. EPA MRID: 00132537
- Surprenant DC (1986) Accumulation and elimination of ¹⁴C-residues by bluegill (*Lepomis macrochirus*) exposed to ¹⁴C-FMC 54800. FMC Study No: 182E54E01/85-4-176. EPA MRID: 00163094/470271-031
- Surprenant DC (1988) Bioavailability, accumulation and aquatic toxicity of ¹⁴C-FMC 54800 residues incorporated into soil. FMC Study No: A85-1576. Springborn Bionomics Study No: 282-0185-6109-000. EPA MRID: 42529902
- Tassou KT, Schulz R (2012) Combined effects of temperature and pyriproxyfen stress in a full life-cycle test with *Chironomus riparius* (Insecta). *Environ Toxicol Chem* 31:2384-2390
- TenBrook PL, Tjeerdema RS (2006) Methodology for derivation of pesticide water quality criteria for the protection of aquatic life in the Sacramento and San Joaquin Rivers basins. Phase I: Review of existing methodologies. Report prepared for the Central Valley Regional Water Quality Control Board
- TenBrook PL, Tjeerdema RS, Hann P, Karkoski J (2009) Methods for deriving pesticide aquatic life criteria. *Rev Environ Contamin Toxicol* 199:19-110

- TenBrook PL, Palumbo AJ, Fojut TL, Hann P, Karkoski J, Tjeerdema RS (2010) The University of California-Davis methodology for deriving aquatic life pesticide water quality criteria. *Rev Environ Contamin Toxicol* 209:1-155
- Tracey GA, Hansen DJ (1996) Use of biota-sediment accumulation factors to assess similarity of nonionic organic chemical exposure to benthically-coupled organisms of differing trophic mode. *Arch Environ Contamin Toxicol* 30:467-475.
- Trimble TA, You J, Lydy MJ (2008) Bioavailability of PCBs from field-collected sediments: application of Tenax extraction and matrix-SPME techniques. *Chemosphere* 71:337-344
- Trimble AJ, Belden JB, Muetting SA, Lydy MJ (2010) Determining modifications to bifenthrin toxicity and sediment binding affinity from varying potassium chloride concentrations in overlying water. *Chemosphere* 80:53-59
- UEPA (1978) Water quality criteria. *Fed Regist* 43(97):21506-21218
- USEPA (1980a) Ambient water quality criteria for aldrin/dieldrin. US Environmental Protection Agency, Washington, DC
- USEPA (1980b) Ambient water quality criteria for chlordane, EPA 440/5-80-027. US Environmental Protection Agency, Washington, DC
- USEPA (1980c) Ambient water quality criteria for endosulfan, EPA 440/5-80-046. US Environmental Protection Agency, Washington, DC
- USEPA (1980d) Ambient water quality criteria for endrin, EPA 440/5-80-047. US Environmental Protection Agency, Washington, DC
- USEPA (1985) Guidelines for deriving numerical national water quality criteria for the protection of aquatic organisms and their uses, PB-85-227049. US Environmental Protection Agency, National Technical Information Service, Springfield, VA
- USEPA (1991) Technical Support Document for Water Quality-based Toxics Control, EPA/505/2-90-001. US Environmental Protection Agency, Washington, DC
- USEPA (1996a) Product properties test guidelines. OPPTS 830.7560. Partition coefficient (n-octanol/water), generator column method, EPA 712-C-96-039. US Environmental Protection Agency Office of Prevention, Pesticides and Toxic Substances (OPPTS), Washington, DC
- USEPA (1996b) Product properties test guidelines. OPPTS 830.7550. Partition coefficient (n-octanol/water), shake flask method, EPA 712-C-96-038. US

Environmental Protection Agency Office of Prevention, Pesticides and Toxic Substances (OPPTS), Washington, DC

USEPA (1996c) Ecological Effects Test Guidelines. OPPTS 850.1735. Whole sediment acute toxicity: Invertebrates, freshwater. Public draft. EPA 712-C-96-354. April 1996. USEPA Environmental Protection Agency Office of Prevention, Pesticides and Toxic Substances (OPPTS), Washington, DC

USEPA (2000) Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Second edition. EPA 600/R-99/064. Office of Research and Development, Washington, DC

USEPA (2001) Methods for collection, storage and manipulation of sediments for chemical and toxicological analysis: Technical manual. EPA-823-B-01-002

USEPA (2003a) Water quality guidance for the Great Lakes system. Fed Regist 40

USEPA (2003b) Draft update of ambient water quality criteria for copper, EPA 822-R-03-026. US Environmental Protection Agency, Washington, DC

USEPA (2003c) Procedures for the derivation of equilibrium partitioning sediment benchmarks (ESBs) for the protection of benthic organisms: Dieldrin. EPA 600 R 02 010. US Environmental Protection Agency, Office of Research and Development, Washington DC

USEPA (2003d) Procedures for the derivation of equilibrium partitioning sediment benchmarks (ESBs) for the protection of benthic organisms: Endrin. EPA 600 R 02 009. US Environmental Protection Agency, Office of Research and Development, Washington DC

USEPA (2005a) Aquatic life ambient water quality criteria, diazinon, final, EPA-822-R-05-006. US Environmental Protection Agency, Washington, DC

USEPA (2005b) Science Advisory Board consultation document, proposed revisions to aquatic life guidelines, water-based criteria. US Environmental Protection Agency, Washington, DC

USEPA (2006a) Bifenthrin; Pesticide Tolerance. 62 CFR 62961.
<http://www.epa.gov/fedrgstr/EPA-PEST/1997/November/Day-26/p30948.htm>

USEPA (2006b) Sediment Quality Guidelines website. United States Environmental Protection Agency, Washington, DC. www.epa.gov/OST/cs/guidelines.htm

- USEPA (2006c) National Ambient Air Quality Standards website. United States Environmental Protection Agency, Washington, DC.
www.epa.gov/air/criteria.html
- USEPA (2011) Review of Methods for Characterizing Effects of Pesticides and Other Chemical Stressors to Aquatic Organisms. United States Environmental Protection Agency, Washington, DC.
- USEPA (2012) Equilibrium partitioning sediment benchmarks (ESBs) for the protection of benthic organisms: Procedures for the determination of the freely dissolved interstitial water concentrations of nonionic organics. EPA/600/R-02/012. United States Environmental Protection Agency, Office of Research and Development, Washington, DC 20460.
- USFDA (2000) Industry activities staff booklet, www.cfsan.fda.gov/~lrd/fdaact.html. United States Food and Drug Administration, Washington, DC
- van den Berg M, Birnbaum L, Bosveld ATC, Brunstrom B, Cook P, Feeley M, Giesy JP, Hanberg A, Hasegawa R, Kennedy SW, Kubiak T, Larsen JC, Van Leeuwen FXR, Liem AKD, Nolt C, Peterson RE, Poellinger L, Safe S, Schrenk D, Tillitt D, Tysklind M, Younes M, Waern F, Zacharewski T (1998) Toxic equivalency factors (TEFs) for PCBs, PCDDs, PCDFs for humans and wildlife. *Environ Health Perspect* 106:775-792
- van der Hoeven N, Nopper F, Leopold A (1997) How to measure no effect. 1. Towards a new measurement of chronic toxicology in ecotoxicology, introduction and workshop results. *Environmetrics* 8:241-248
- van Straalen NM, van Leeuwen CJ (2002) European history of species sensitivity distributions. In: Posthuma L, Suter GWI, Traas TP (eds) *Species sensitivity distributions in ecotoxicity*. Lewis Publishers, New York, NY pp. 19-34
- Verschueren K (2009) *Handbook of environmental data on organic chemicals*, 5th edition, Wiley, Hoboken, NJ
- Versteeg DJ, Belanger SE, Carr GJ (1999) Understanding single species and model ecosystem sensitivity: data-based comparison. *Environ Toxicol Chem* 18:1329-1346
- Warne MSJ, van Dam R (2008) NOEC and LOEC data should no longer be generated or used. *Australasian J Ecotoxicol* 14:1-5
- Werner I, Moran K (2008) Effects of pyrethroid insecticides on aquatic organisms. In Gan J, Spurlock F, Hendley P, Weston D (Eds). *Synthetic Pyrethroids:*

- Occurrence and Behavior in Aquatic Environments. American Chemical Society, Washington, DC
- Weston DP, Amweg EI, Mekebri A, Ogle RS, Lydy MJ (2006) Aquatic effects of aerial spraying for mosquito control over an urban area. *Environ Sci Technol* 40:5817-5822
- Weston DP, Holmes RW, You J, Lydy MJ (2005) Aquatic toxicity due to residential use of pyrethroid insecticides. *Environ Sci Technol* 39:9778-9784
- Weston DP, Holmes RW, Lydy MJ (2009a) Residential runoff as a source of pyrethroid pesticides to urban creeks. *Environ Pollut* 157:287-294
- Weston DP, Jackson CJ (2009) Use of engineered enzymes to identify organophosphate and pyrethroid-related toxicity in toxicity identification evaluations. *Environmental Science & Technology* 43:5514-5520
- Weston DP, You J, Harwood AD, Lydy MJ (2009b) Whole sediment toxicity identification evaluation tools for pyrethroid insecticides: III. Temperature manipulation. *Environmental Toxicology and Chemistry* 28:173-180.
- Weston DP, You J, Lydy MJ (2004) Distribution and toxicity of sediment-associated pesticides in agriculture-dominated water bodies of California's Central Valley. *Environ Sci Technol* 38:2752-2759
- Weston DP, Zhang MH, Lydy MJ (2008) Identifying the cause and source of sediment toxicity in an agriculture-influenced creek. *Environ Toxicol Chem* 27:953-962
- Wojtaszek BF, Buscarini TM, Chartrand DT, Stephenson GR, Thompson DG (2005) Effect of Release ® herbicide on mortality, avoidance response, and growth of amphibian larvae in two forest wetlands. *Environ Toxicol Chem* 24:2533-2544
- Wood A (2008) Compendium of Pesticide Common Names website.
<http://www.alanwood.net/pesticides/bifenthrin.html>
- Woodburn KB, Doucette WJ, Andren AW (1984) Generator column determination of octanol-water partition coefficients for selected polychlorinated biphenyl congeners. *Environ Sci Technol* 18:457-459
- Xu YP, Spurlock F, Wang ZJ, Gan J (2007) Comparison of five methods for measuring sediment toxicity of hydrophobic contaminants. *Environ Sci Technol* 41:8394-8399
- Yang WC, Gan JY, Hunter W, Spurlock F (2006a) Effect of suspended solids on bioavailability of pyrethroid insecticides. *Environ Toxicol Chem* 25:1585-1591

- Yang WC, Hunter W, Spurlock F, Gan J (2007) Bioavailability of permethrin and cyfluthrin in surface waters with low levels of dissolved organic matter. *J Environ Qual* 36:1678-1685.
- Yang Y, Hunter W, Tao S, Gan J (2009) Effects of black carbon on pyrethroid availability in sediment. *J Agricult Food Chem* 57:232-238
- Yang WC, Spurlock F, Liu WP, Gan JY (2006b) Inhibition of aquatic toxicity of pyrethroid insecticides by suspended sediment. *Environ Toxicol Chem* 25:1913-1919
- You J, Brennan A, Lydy MJ (2009) Bioavailability and biotransformation of sediment-associated pyrethroid insecticides in *Lumbriculus variegatus*. *Chemosphere* 75:1477-1482
- You J, Harwood, AD, Li H, Lydy MJ (2011) Chemical techniques for assessing bioavailability of sediment-associated contaminants: SPME versus Tenax extraction. *J Environ Monit* 13:792-800
- You J, Landrum PF, Lydy MJ (2006) Comparison of chemical approaches for assessing bioavailability of sediment-associated contaminants. *Environ Sci Technol* 40:6348-6353
- You J, Pehkonen S, Landrum PF, Lydy MJ (2007) Desorption of hydrophobic compounds from laboratory-spiked sediments measured by Tenax absorbent and matrix solid-phase microextraction. *Environ Sci Technol* 41:5672-5678
- Yount JD, Niemi GJ (1990) Recovery of lotic communities and ecosystems from disturbance – a narrative review of case studies. *Environ Manag* 14:547–569
- Zabel TF, Cole S (1999) The derivation of environmental quality standards for the protection of aquatic life in the UK. *J CIWEM* 13:436-440
- Zhang L-J, Ying G-G, Chen F, Zhao JL, Wang L, Fang YX (2012) Development and application of whole-sediment toxicity test using immobilized freshwater microalgae *Pseudokirchneriella subcapitata*. *Environ Toxicol Chem* 31:377-386
- Zischke JA, Arthur JW, Hermanutz RO, Hedtke SF, Helgen JC (1985) Effects of pentachlorophenol on invertebrates and fish in outdoor experimental channels. *Aquat Toxicol* 7:37-58

Appendix A – Guidelines for Evaluating Toxicity Tests

This document is intended to aid the user in understanding the test parameter as well as determining if a particular parameter is acceptable according to standard protocols. The acceptable test conditions in this document are based upon ASTM, USEPA and OPPTS methods. This should not be considered an absolute reference and it is recommended that the actual standard methods be reviewed. A summary table (**Table 1**) of the OPPTS 850.1735 (1996) test conditions and acceptability requirements (or guidance) is provided for *H. azteca* and *C. dilutus*. A summary table (**Table 2**) of the ASTM E 1706-05 test conditions and acceptability requirements (or guidance) for *H. azteca*, *C. dilutus*, *C. riparius*, *D. magna*, *C. dubia*, *Hexagenia spp.*, *T. tubifex* and *Diporeia spp.* for various exposure durations has been included. Sediment spiking and pore water handling procedures are based on EPA-823-B-01-002 (**Table 3**).

1. Endpoints
 - a. Standard endpoints (preferred)
 - i. Survival
 - ii. Growth –wet or dry weight (biomass), length, other measures as appropriate to the organism
 - iii. Reproduction - # young, # eggs, hatching success, fecundity, etc.
 - b. Non-Standard endpoints
 - i. Immobility (death usually shortly follows)
 - ii. Instantaneous growth rate
 - iii. AChE inhibition needs a link to recognized endpoint for that species. Generally if such study exists, the recognized endpoint is used.
 - iv. Heat shock proteins, Vitellogenin, etc. currently have no link to population effects – although these alternate endpoints could be used if there was a study linking them to population effects for the particular test species.
2. Acceptable standard (or equivalent) used
 - a. ASTM, USEPA, OPPTS, OECD, Environment Canada or equivalent
 - b. Standard methods are not currently available for all taxa, so for species not included in these methods, the reader should check for new methods and if not available, use best professional judgment to determine which category a particular test fits into.
3. Test was of appropriate duration

Exposure duration can be either acute or chronic and are defined as follows:

 - a. Acute
 1. Invertebrate tests with exposures lasting 10-14-d including survival and growth endpoints (ASTM E 1706-05 (2008), Ingersoll and MacDonald 2002)
 2. Amphibian tests with exposures lasting 10-d (ASTM E 2591-07 (2007))
 - b. Chronic - partial or full lifecycles
 1. Invertebrate tests with exposures lasting 20-60 d, preferable early life stage, including survival, growth, and possibly reproduction and emergence (ASTM E 1706-05 (2008), Ingersoll and MacDonald 2002, RIVM 2001)
 2. Any test with algae, protozoa, or plants (RIVM 2001)
4. Control
 - a. Appropriate controls must be used. Must have negative and solvent control (if solvent is used).
 - b. Acceptable control response is provided in **Table 1** for OPPTS 850.1735 (1996) and **Table 2** for ASTM 1706-05 (2008) for various organisms and exposure durations. The most likely encountered are as follows:
 - i. *H. azteca* (10 & 28 d) - $\geq 80\%$ control survival and measurable control growth
 - ii. *C. dilutus* (10 d) - $\geq 70\%$ control survival with minimum mean weight per surviving control organism of 0.6 mg dry weight or 0.48 mg ash free dry weight

5. Chemical
 - a. Purity \geq 80%
 - b. Technical grade is sufficient.
 - c. Formulations are not acceptable (e.g., pesticide W10G may indicate a 10% formulation).
6. Sediment
 - a. Sediment spike method (**Table 3**; EPA-823-B-01-002 (2001))
 - i. Shell coating technique should be used in which compound is added to sand and solvent is completely evaporated from sand before mixing with wet sediment in order to avoid affecting sediment chemistry or bioavailability.
 - ii. Jar rolling preferred over hand mixing for large batches of sediment.
 1. Prolonged rolling (i.e., > 1 week) and overfilling jars should be avoided
 - iii. Wet spiking is preferred to dry spiking.
 1. Determine % moisture on 3 replicate samples to determine dry weight of sediment.
 - b. Spike equilibration time. (**Table 3**; EPA-823-B-01-002 (2001))

Store for at least **1 month** so interstitial water and sediment can come to equilibrium.
7. Organisms.
 - a. Appropriate size/age/growth phase.
 - i. *H. azteca* (10 & 28 day exposure) – 7 to 14 day, all within 1-2 day range in age (ASTM 1706-05 (2008); OPPTS 850.1735 (1996))
 - ii. *C. dilutus* (10 day exposure)
 1. All 3rd instar (50% of organisms) or younger (OPPTS 850.1735)
 2. 2nd to 3rd instar larvae (about 10 day old; ASTM 1706-05 (2008))
 - iii. Other species.
 1. See **Table 2** for acceptable age ranges for *C. riparius*, *D. magna*, *C. dubia*, *Hexagenia spp.*, *T. tubifex* and *Diporeia spp.* for various exposure durations.
 2. Use best professional judgment if no standard method exists (i.e. fish, amphibians, etc)
 - b. No prior contaminant exposure.
 - i. If organisms are field collected, they have likely been exposed and this is not acceptable
 - ii. If it is stated that organisms were collected from an unpolluted site, this is acceptable.
 - iii. Laboratory cultures are acceptable.
 - c. Organisms randomly assigned to test containers.
 - i. The study must state this.
 - d. Adequate number per replicate/appropriate cell density.

- i. Acceptable replicate numbers and cell densities are provided in **Table 1** for OPPTS 850.1735 (1996) and **Table 2** for ASTM 1706-05 (2008) for various organisms and exposure durations. The most likely encountered are as follows:
 - 1. *H. azteca* (10 & 28 d) – 4 replicates per treatment is the absolute minimum with 8 recommended for routine analysis (ASTM 1706-05 (2008) & OPPTS 850.1735 (1996))
 - 2. *C. dilutus* (10 d) - 4 replicates per treatment is the absolute minimum with 8 recommended for routine analysis
 - 3. Other species – see **Table 2** for replicate and cell density guidance for *C. riparius*, *D. magna*, *C. dubia*, *Hexagenia spp.*, *T. tubifex* and *Diporeia spp.* under various exposure durations. ((ASTM 1706-05 (2008))
 - 4. 10 organisms per container for *H. azteca* and *C. dilutus* (ASTM 1706-05 (2008) & OPPTS 850.1735 (1996))
- e. Feeding appropriate to standard method.
 - i. Acceptable feeding regimes are provided in **Table 1** for OPPTS 850.1735 (1996) and **Table 2** for ASTM 1706-05 (2008) for various organisms and exposure durations. The most likely encountered are as follows:
 - 1. *H. azteca* (10 & 28 d) – YCT 1 to 1.5 mL (1800mg/L) daily (ASTM 1706-05 2008a & OPPTS 850.1735, respectively)
 - 2. *C. dilutus* (10 d) – Tetrafin goldfish food 1.5 mL (4 g/L) daily (ASTM 1706-05 2008a & OPPTS 850.1735)
 - 3. Other species – see **Table 2** for replicate and cell density guidance for *C. riparius*, *D. magna*, *C. dubia*, *Hexagenia spp.*, *T. tubifex* and *Diporeia spp.* under various exposure durations. (ASTM 1706-05 (2008))
- f. Organisms properly acclimated and disease free prior to testing.
 - i. If culture and testing conditions are the same, then the acclimation period is considered acceptable.
 - ii. 7 day acclimation after arrival from hatchery or if wild. (WQC)
- 8. Exposure type and renewal frequency appropriate to chemical.
 - a. Static, static-renewal or flow-through are considered exposure type descriptors.
 - i. Acceptable exposure types and renewal frequencies of overlying water are provided in **Table 1** for OPPTS 850.1735 (1996) and **Table 2** for ASTM 1706-05 (2008) for various organisms and exposure durations. The most likely encountered are as follows:
 - 1. *H. azteca* (10 & 28 d) – Static-renewal; 2 volumes per day on continuous or intermittent basis (ASTM 1706-05 (2008) & OPPTS 850.1735 (1996), respectively)
 - 2. *C. dilutus* (10 d) – Static-renewal; 2 volumes per day on continuous or intermittent basis (ASTM 1706-05 (2008) & OPPTS 850.1735 (1996), respectively)

3. Other species – see **Table 2** for exposure type guidance and overlying water renewal frequency guidance for *C. riparius*, *D. magna*, *C. dubia*, *Hexagenia spp.*, *T. tubifex* and *Diporeia spp.* under various exposure durations. (ASTM 1706-05 2008a)
 - ii. Chronic tests with fish should be flow through in any case.
 - iii. Static exposures can be acceptable but they are not recommended for compounds that may stick to glassware, evaporate or degrade quickly unless concentrations consistent throughout test. (WQC)
 1. Static exposure disadvantages include DO depletion from high COD, BOD or metabolic wastes.
 2. Possible loss of toxicant through volatilization or adsorption to test vessels from static exposures.
 3. Static exposures generally less sensitive than static-renewal or flow through tests because degradation or adsorption may reduce the apparent toxicity. (WQC)
 4. Concentrations should be confirmed, at minimum, at the start and finish of a test.
9. Dilution water source acceptable.
 - a. Culture water is acceptable.
 - b. Well water is acceptable.
 - c. Reconstituted fresh water (USEPA, ASTM hard/soft water) is acceptable.
 - i. Hardness – 90-100 mg/L
 - ii. Alkalinity – 50-70 mg/L as CaCO₃
 - iii. EC – 330-360 µS/cm
 - iv. pH – 7.8-8.2
 - d. Natural (site) waters.
 - i. Should be from an uncontaminated site and of uniform hardness, alkalinity and electrical conductivity determined by <10% monthly average change (ASTM 1706-05 (2008)). Monthly pH range of 0.4 pH units.
 - e. Surface waters
 - i. No chlorinated water.
 - ii. Check for high levels of Cu, Pb, Zn, F-, Cl-, chloroamines. (ASTM 1706-05 (2008))
 - f. Tap water is not acceptable.
 - g. Dechlorinated tap water is not ideal but acceptable.
 - h. Deionized water is not acceptable.
 - i. Deionized water with salts added is acceptable.
10. Hardness, alkalinity, conductivity, pH within organism tolerance limits and or overlying water specifications.
 - a. If standard water used but values not reported, credit given for documentation but not acceptability.
 - b. The following guidance (USEPA 2002 (taken from Marking and Dawson, 1973)) provides approximate water quality values:

	pH	Hardness mg CaCO ₃ /L	Alkalinity mg CaCO ₃ /L
Very soft	6.4-6.8	10-13	10-13
Soft	7.2-7.6	40-48	30-35
Moderately hard	7.4-7.8	80-100	57-64
Hard	7.6-8.0	160-180	110-120
Very hard	8.0-8.4	280-320	225-245

c. *H. azteca* sensitive to hardness and not found in waters with < 7 mg CaCO₃/L.

11. Dissolved oxygen ≥ 60 %.

- a. In general, DO should be > 2.5 mg/L (ASTM 1706-05 (2008) & OPPTS 850.1735 (1996)).
- b. DO concentrations may be converted from mg/L to % using **Table 4**.
 - i. Using the study temperature and water salinity, find the corresponding oxygen water solubility value (mg/L).
 - ii. % DO = (mg/L DO in study @T/ solubility of DO in water @T)*100

12. Temperature within organism tolerance (3 pts) and/or test guidance and held to ±1°C (3 pts).

- a. Daphnids, Hyalella, Mysids: 20 -25°C
- b. Warm water fish (blue gill, minnows): 20 -25°C
- c. Cold water fish (salmon, trout): 10 -14°C

13. Photoperiod and intensity.

- a. 16L:8D is standard and acceptable.
- b. 100 -1000 lux is standard and acceptable.
- c. 12L:12D is acceptable.
- d. Dark is acceptable for insect larvae or eggs.

14. Statistics.

- a. Adequate number of concentrations.
 - i. At least 5.
- b. Random or block design employed.
 - i. Adequate if stated.

- c. Adequate replication.
 - i. At least 4.
- d. Appropriate spacing between concentrations (dilution factor > 0.3).
 - i. i.e., 1, 3, 9, 21 is acceptable
 - ii. i.e., not 1, 10, 100, 1000 (dilution factor=0.1) since the range is too wide and would lead to imprecise toxicity values.
- e. Appropriate statistical methods used.
 - i. Regression (LC₅₀, EC_x)
 - 1. Linear regression
 - 2. Probit
 - 3. Trimmed Spearman-Kärber
 - ii. Hypothesis tests (chronic – NOEC/LOEC)
 - 1. Non-parametric Kruskal-Wallis
 - 2. Mann-Whitney test
 - 3. ANOVA
- f. Hypothesis tests
 - i. Statistical significance: Points should be given if the statistical test demonstrated statistical significance, i.e., the control response was significantly different from one or some of the exposures
 - ii. Significance level: Points should be given if the authors reported the significance level of the test (e.g., alpha = 0.05).
 - iii. Minimum significant difference (MSD) below recommended upper bound.¹
 - 1. The MSD is the smallest decrease in growth or reproduction from the control that could be determined as statistically significant in the test. MSD is based on the number of replicates, control performance, and power of the test. The MSD provides an indication of within-test variability and test method sensitivity.
 - 2. ¹Acceptable MSD levels are species and test method specific; see USEPA (2002) for upper bounds for several standard test species.
 - a. Minnow growth of 12-30% is acceptable.
 - b. *C. dubia* reproduction of 13-47% is acceptable.
 - 3. Percent Minimum Significant Difference (PMSD).
 - a. If the control response is 100%, the MSD and the PMSD is the same.
 - 4. The lower bound is there to provide some protection to dischargers whose toxicity tests have extremely low within treatment variability.
 - a. They detect a significant response compared to the control, but the response is not biologically significant.
 - 5. The upper bound provides some control over tests that have very high within treatment variability and lead to the

conclusion that there was no effect when the test solution was actually toxic.

- a. The variability within treatment masks the ability to detect a significant difference from the controls.
 - b. The upper bound helps weed out sloppy tests.
6. In reality, very few toxicity tests currently report MSD/PMSD information.
- iv. % of control at NOEC and/or LOEC
 1. E.g., control survival = 92%, NOEC survival=91%, LOEC survival=68%; % control at NOEC = 91%/92% = 98.9%, % control at LOEC = 68%/92% = 73.9%.
 - v. NOEC/LOEC reasonable compared to control.
 1. Reasonable is decided using professional judgment on a case by case basis, can be based on MSD upper bound and potential biological significance of response level, or by comparing the % control at NOEC and % control at LOEC to see if the levels seem reasonable.

15. Point Estimates.

- a. LC/EC values calculable.
 - i. No results of < or >.
 - ii. i.e., $LC_{50} < 100$ suggests a problem since a lower concentration was not tested.
 - iii. $LC_{50} > 100$ is informative but may indicate insensitivity.

Appendix A References

- ASTM (2007) Standard guide for conducting whole sediment toxicity tests with amphibians. American Society of Testing and Materials. ASTM designation: E 2591-07.
- ASTM (2008a) Standard tests method for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates. American Society of Testing and Materials. ASTM designation: E 1706-05 (2008).
- Ingersoll CG, MacDonald DD (2002) A Guidance Manual to Support the Assessment of Contaminated Sediments in Freshwater Ecosystems. Volume III- Interpretation of the results of sediment quality investigations. Great Lakes National Program Office: Chicago, IL. EPA-905-B02-001-C.

- OPPTS 850. 1735 (1996) Whole sediment acute toxicity: Invertebrates, freshwater.
USEPA Office of Prevention, Pesticides and Toxic Substances, EPA 712-C-96-354.
- RIVM (2001) Guidance document on deriving environmental risk limits. Traas TP (ed) RIVM report 601501 012. National Institute of Public Health and the Environment, Bilthoven, The Netherlands.
- USEPA (2000) Understanding and accounting for method variability in whole effluent toxicity applications under the national pollutant discharge elimination system program. Office of Wastewater Management, U.S. Environmental Protection Agency, Washington, D.C. 20460. EPA/833/R-00/003.
<http://www.toxicity.com/pdf/epa2000june.pdf>
- USEPA (2000a) Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Second edition. EPA 600/R-99/064. Office of Research and Development. Washington D. C.
- USEPA (2001) Methods for collection, storage and manipulation of sediments for chemical and toxicological analysis: technical manual. EPA 823-B-01-002. U.S. Environmental Protection Agency, Office of Water, Washington, DC.
- USEPA (2002) Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th edition, EPA-821-R-02-013. United States Environmental Protection Agency, Washington, DC.
<http://www.epa.gov/waterscience/methods/wet/disk3/ctf.pdf>
- USEPA. 2002b. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th edition, EPA-821-R-02-012. United States Environmental Protection Agency, Washington, D. C.
<http://www.epa.gov/waterscience/methods/wet/disk2/index.html>

Appendix B – Toxicity Data

Summaries for Bifenthrin

Interim Bioavailable Sediment Quality Criteria Report for Bifenthrin

Appendix B

Toxicity data summary sheets

Appendix B1: Acceptable data rated RR

Appendix B2: Supplemental data rated RL, LR, or LL

Within each section, studies are listed in alphabetical order by species name, when there are multiple summaries for one species, they are listed in alphabetical order by author. Unused lines were deleted from tables.

In the notes column, it is noted in bold type if points were taken off for documentation or acceptability based on the reliability rating tables. The number of points taken off for each parameter are detailed in the notes section at the bottom of each summary.

Appendix B1

Studies rated RR

Toxicity Data Summary

Chironomus dilutus (formerly *C. tentans*)

Maul JD, Brennan AA, Harwood AD, Lydy MJ (2008a) Effect of sediment-associated pyrethroids, fipronil and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization and survival. Environ Toxicol Chem 27:2582-2590.

Relevance

Score: 100 (Survival); 85 (Growth)
Rating: R (Survival); L (Growth)

Reliability

Score: 82 – survival, 80.5 - growth
Rating: R

* Relevance points taken off for: Growth - Control response not acceptable (15).

<i>C. dilutus</i>	Maul et al. 2008a	
Parameter	Value	Comment
Test method cited	USEPA 2000	600/R-99-064
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	Formerly <i>C. tentans</i>
Family in North America?	Yes	
Age/size at start of test/growth phase	Mid to late 3 rd instar larvae	
Source of organisms	Lab culture	Southern Illinois U.
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Yes	
Test vessels randomized?	Yes	
Test duration	10 d	
Effect 1	Survival	
Control response 1	84%	Combined solvent & negative control results because not sig. different (p<0.05)
Effect 2	Immobilization	Defined as inability to perform typical S-shape response to probing
Control response 2	Not reported	

<i>C. dilutus</i>	Maul et al. 2008a	
Parameter	Value	Comment
Effect 3	Growth - Body mass by ash free dry mass (AFDM) - Daily instantaneous growth rate (IGR)	
Control response 3	AFDM: 0.3 mg IGR: 1.003	Estimated from Fig. 1A AFDM response not acceptable (>0.48 mg) Accept. Points
Effect 4	Body condition index (BCI)	Calculated by regressing AFDM of controls against head size score of exposed organisms
Control response 4	Not reported	
Temperature	23°C	
Test type	Static renewal	Daily renewal 75%
Photoperiod/light intensity	16 h light:8 h dark	
Overlying water source	EPA reconstituted moderately hard water	
pH	6.61-6.74	
Hardness mg/L as CaCO ₃	Not reported	Doc./Accept. Points
Alkalinity mg/L as CaCO ₃	Not reported	Doc./Accept. points
Conductivity	275-396 uS/cm	
Dissolved Oxygen	6.12-6.78 mg/L	
Sediment source	Soil collected 15 km south of Carbondale, IL	
Organic carbon content	0.97%	
Particle size distribution (sand, silt, clay)	Not reported	Sieved to <500 µm
Sediment spike procedure	150 uL acetone solution added dropwise to sediment slurry while mixing 1 h	Accept. points
Sediment spike equilibration time	14 d in dark 4°C	Accept. points
Sediment to Solution ratio	50 g DW: 700 mL	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	

<i>C. dilutus</i>	Maul et al. 2008a	
Parameter	Value	Comment
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Interstitial water DOC	Not applicable	
Feeding	1 mL of 6 g/L of tetrafin solution daily	
Purity of test substance	>98%	Chem service
Measured is what % of nominal?	80-120%	
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	See spike procedure	
Concentration 1 Meas ($\mu\text{g/g OC}$)	2.2	5 reps/conc and 10 midges/rep Nominal conc. NR Doc. points
Concentration 2 Meas ($\mu\text{g/g OC}$)	2.9	5 reps/conc and 10 midges/rep
Concentration 3 Meas ($\mu\text{g/g OC}$)	3.7	5 reps/conc and 10 midges/rep
Concentration 4 Meas ($\mu\text{g/g OC}$)	4.7	5 reps/conc and 10 midges/rep
Concentration 5 Meas ($\mu\text{g/g OC}$)	5.3	5 reps/conc and 10 midges/rep
Concentration 6 Meas ($\mu\text{g/g OC}$)	6.8	5 reps/conc and 10 midges/rep
Control	Solvent and negative	5 reps/conc and 10 midges/rep
LC ₅₀ (95% confidence interval) $\mu\text{g/g OC}$	6.2 (5.1-8.7)	Method: Log probit analysis
EC ₅₀ (95% confidence interval) $\mu\text{g/g OC}$	Immobilization: 2.2 (1.9-2.4) Growth AFDM: 2.4 (1.6-2.8) Growth IGR: 1.5 (1.2-1.6)	Method: Maximum likelihood analysis
EC ₂₀ $\mu\text{g/g OC}$	Growth AFDM: 1.0 (0.7-1.3) Growth IGR: 0.6 (0.5-0.7)	
NOEC $\mu\text{g/g OC}$	AFDM: < 2.2 IGR: < 2.2	
LOEC $\mu\text{g/g OC}$	AFDM: 2.2 IGR: 2.2	Linear interpolation method MSD: not reported Doc./Accept. points

<i>C. dilutus</i>	Maul et al. 2008a	
Parameter	Value	Comment
MATC (GeoMean NOEC,LOEC) (µg/kg)	Not calculable	
% of control at NOEC	Not calculable	
% of control at LOEC	AFDM: 0.15/0.3*100=50% IGR: 1.001/1.004*100=99.7%	From Fig. 1A

Notes:

Article refers to NOEC values but not specifically stated.

Lethal to sublethal ratios were also reported:

LC50/EC50_immobilization: 2.9

LC50/EC20_AFDM: 6.5

LC50/EC20_IGR: 10.7

Reliability points taken off for:

Survival – point estimates: Mean (86, 78) = 82

Documentation: Nominal concentrations (2), Overlying water hardness (1), Overlying water alkalinity (1), Particle size distribution (2), Hypothesis Testing (8). Total: 100-14=86

Acceptability: Sediment spike method (4), Spike equilibration time (6), Carrier solvent evaporation (4), Overlying water hardness (1), Overlying water alkalinity (1), Temperature variation NR (3), Hypothesis tests (3). Total: 100-22=78

Growth-point estimates and LOEC: Mean (92, 69) = 80.5

Documentation: Nominal concentrations (2), Overlying water hardness (1), Overlying water alkalinity (1), Particle size distribution (2), Minimum significant difference (2). Total: 100-8=92

Acceptability: Control response within guideline (10), Sediment spike method (4), Spike equilibration time (6), Carrier solvent evaporation (4), Overlying water hardness (1), Overlying water alkalinity (1), Temperature variation NR (3), Minimum significant difference (1), NOEC compared to control (1). Total: 100-31=69

Toxicity Data Summary

Chironomus dilutus

Picard CR (2010b) 10-Day toxicity test exposing midges (*Chironomus dilutus*) to bifenthrin applied to formulated sediment under static-renewal conditions following OPPTS draft guideline 850.1735. Performed by Springborn Smithers Laboratories, Wareham, MA, Study No. 136565.6143; submitted to Pyrethroid Working Group, Washington, DC.

Relevance
Score: 100
Rating: R

Reliability
Score: 88.5
Rating: R

<i>C. dilutus</i>	Picard 2010b	
Parameter	Value	Comment
Test method cited	EPA 2000	
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	11 day old, 3 rd instar larvae	
Source of organisms	Lab culture	Environmental Consulting & Testing, Superior, WI
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Yes	
Test vessels randomized?	Not reported	Accept. points
Test duration	10 day	
Data for multiple times?	No	
Effect 1	Mortality	
Control response 1	Negative control: 94% Solvent control: 85%	Pooled control: 89%
Effect 2	Growth (total dry wt. per organism)	
Control response 2	Pooled control: 0.92 mg	
Temperature	21- 25 °C	Accept. points
Test type	Static renewal	
Photoperiod/light intensity	16 h/8 h dark; 530-960 lux	

<i>C. dilutus</i>	Picard 2010b	
Parameter	Value	Comment
Overlying water	Well water	
pH	6.7-7.2	
Hardness	64-76 mg/L as CaCO ₃	
Alkalinity	20-26 mg/L as CaCO ₃	
Conductivity	390-400 µmhos/cm	
Dissolved Oxygen	3.7 – 8.2 mg/L	Accept. points
Ammonia-N	<0.1 – 2.2 mg/L	
Sediment formulated?	Yes	Method: OECD 218
Organic carbon content	2.3%	
Particle size distribution (sand, silt, clay)	79%, 6%, 15%	
pH	7.1	
Percent moisture	36.22%	
Sediment spike method	Jar rolling technique	Initially rolled 4 h at 15 rpm at room temp.
Sediment spike equilibration time	14 d at 4°C	Mixed 2x/week for 2 h at room temp. Accept. points
Sediment to Solution ratio	100:175 mL	100 mL sediment = 143 g wet wt. or 91.3 g dry wt.
Sediment extraction/analysis method	Ext/cleanup and GC/MS	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Centrifugation	1200 g 15-30 min
Interstitial water chemical extraction	SPME, conditions not reported	
Interstitial water chemical analysis	Not reported	
DOC	81-120 mg C/L	
Feeding	Flake fish food	
Purity of test substance	95.7%	
Measured is what % of nominal?	73-88% in sediment spikes	Accept. points
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	0%	10 mL of acetone evaporated from 0.05 kg sand
Concentration 1 Nom; Meas (µg/kg)	16; 13	8 Reps, 10 per rep

<i>C. dilutus</i>	Picard 2010b	
Parameter	Value	Comment
Concentration 2 Nom; Meas (µg/kg)	31; 23	8 Reps,10 per rep
Concentration 3 Nom; Meas (µg/kg)	63; 48	8 Reps,10 per rep
Concentration 4 Nom; Meas (µg/kg)	130; 110	8 Reps,10 per rep
Concentration 5 Nom; Meas (µg/kg)	250; 200	8 Reps,10 per rep
Concentration 6 Nom; Meas (µg/kg)	500; 400	8 Reps,10 per rep
Control	Solvent and negative controls	8 Reps,10 per rep
LC ₅₀ (95% confidence interval)	Dry weight basis 350 (310-400) µg/kg DW OC-normal basis 15.2 (13.4-17.4) µg/g OC	Method: Probit analysis (TOXSTAT)
EC ₅₀ (95% confidence interval)	Dry weight basis 160 (140-180) µg/kg DW OC-normal basis 6.96 (6.09-7.83) µg/g OC	Method: Linear interpolation (TOXSTAT)
NOEC	Dry weight basis Survival: 110 µg/kg DW Growth: 110 µg/kg DW OC-normal basis Survival: 4.78 µg/g OC Growth: 4.78 µg/g OC	Method: Bonferroni's t-test p: 0.05 MSD: Not reported Doc./Accept. points
LOEC	Dry weight basis Survival: 200 µg/kg DW Growth: >110 µg/kg DW OC-normal basis Survival: 8.70 µg/g OC Growth: >4.78 µg/g OC	Same as above
MATC (GeoMean NOEC,LOEC)	Dry weight basis Survival: 148 µg/kg DW OC-normal basis Survival: 6.45 µg/g OC	
% of control at NOEC	Survival: 84%/89%=94% Growth: 0.72mg/0.92mg=78%	Pooled controls
% of control at LOEC	Survival: 68%/89%=76%	Pooled controls

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Measured sediment concentrations are the mean of measurements at day 0 and day 10.

Although the study states pore water results are in a supplemental report, the data was never made available due to analytical and sample holding time issues.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: $100-2=98$

Acceptability: Measured concentration within 20% nominal (4), Spike equilibration time (6), Dissolved oxygen < 60% saturation (5), Temperature variation (3), Random design (2), Minimum significant difference (1). Total: $100-21=79$

Reliability Score: Mean (98, 79) = 88.5

Toxicity Data Summary

Chironomus dilutus (formerly *C. tentans*)

Putt AE (2005a) Bifenthrin – Toxicity to midge (*Chironomus tentans*) during a 10-day sediment exposure. Study performed by Springborn Smithers Laboratories, Wareham, MA, project ID: 136565.6106; submitted to Pyrethroid Working Group, Washington, DC. EPA MRID 46591502. DPR record number 238254.

Relevance
Score: 100
Rating: R

Reliability
Score: 96
Rating: R

<i>C. dilutus</i>	Putt 2005a	
Parameter	Value	Comment
Test method cited	EPA 2000	
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	Formerly <i>C. tentans</i>
Family in North America?	Yes	
Age/size at start of test/growth phase	10 d old, 2 nd – 3 rd instar larvae	Head capsule 0.25-0.45 mm confirms life stage
Source of organisms	Springborn Smithers lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Yes	
Test vessels randomized?	Not reported	Accept. Points
Test duration	10 day	
Effect 1	Survival	
Control response 1	91% negative control; 83% solvent control	
Effect 2	Growth	Ash free dry weight
Control response 2	1.99 mg pooled control	
Temperature	21-24°C	Accept. Points
Test type	Static renewal	Renew 50 mL water 7x/day
Photoperiod/light intensity	16 h light:8 h dark; 660-1000 lux	
Overlying water	Well water	

<i>C. dilutus</i>	Putt 2005a	
Parameter	Value	Comment
pH	6.4-6.8	
Hardness	40-52 mg/L as CaCO ₃	
Alkalinity	26-46 mg/L as CaCO ₃	
Conductivity	240-270 µmhos/cm	
Dissolved Oxygen	1.8 – 8.3 mg/L	
TOC/DOC	Not stated	
Ammonia-N	0.26 – 1.2 mg/L @ day 0	<0.10 mg/L @ day 10
Chemical analysis	Yes via LSC	
Sediment source	Natural freshwater; Glen Charlie Pond, Wareham, MA	
Organic carbon content	5.5%	
Particle size distribution (sand, silt, clay)	83%, 12%, 5.5%	Sieved to remove particles > 2 mm
pH	4.9	
Percent solids	38.78%	
Sediment spike procedure	Spiked 0.05 kg silica with 9 mL acetone solution, evaporated solvent, then added to 2 kg wet sediment (0.7756 kg dry wt.)	Rolled for 4 hr after initial spike
Sediment spike equilibration time	Jar rolling technique 31 days at 4°C	Roll once/week for 2 h during equilibration
Sediment to Solution ratio	100ml(4cm):175 mL	122 g wet t or 47 g dry wt.
Interstitial water monitored?	Yes	
Interstitial water isolation method	Centrifugation	30 min at 10,000 g
Interstitial water chemical analysis method	LSC (Liquid scintillation counting)	2 mL interstitial water
DOC	t ₀ : 9.2-13.4 mg/L day 10: 6.8-12.6 mg/L	
Feeding	Flakes fish food suspension once daily	1.5 mL of 4.0 mg/mL per vessel
Purity of test substance	96.4% ¹⁴ C-bifenthrin; specific activity 24.4 mCi/mmol using HPLC-radiochemical detection (RAM)	Technical (93%) used for range finding
Concentrations measured?	Yes: sediment, interstitial water, and overlying water	
Measured is what % of nominal?	Sediment: 88%-95%	
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in	0	

<i>C. dilutus</i>	Putt 2005a	
Parameter	Value	Comment
test solutions		
Concentration 1 Nom; Meas (mean of t_0 and day 10)	Sediment dry wt.: 90; 83 $\mu\text{g}/\text{kg}$ OC-normal*: 1.5 $\mu\text{g}/\text{g}$ OC Interstitial water (meas): 0.24 $\mu\text{g}/\text{L}$	8 reps, 10 midges/rep
Concentration 2 Nom; Meas (mean of t_0 and day 10)	Dry Weight: 180; 170 $\mu\text{g}/\text{kg}$ OC-normal*: 3 $\mu\text{g}/\text{g}$ OC Interstitial water (meas): 0.56 $\mu\text{g}/\text{L}$	8 reps, 10 midges/rep
Concentration 3 Nom; Meas (mean of t_0 and day 10)	Dry Weight: 350; 330 $\mu\text{g}/\text{kg}$ OC-normal*: 6 $\mu\text{g}/\text{g}$ OC Interstitial water (meas): 0.51 $\mu\text{g}/\text{L}$	8 reps, 10 midges/rep
Concentration 4 Nom; Meas (mean of t_0 and day 10)	Dry Weight: 700; 610 $\mu\text{g}/\text{kg}$ OC-normal*: 11 $\mu\text{g}/\text{g}$ OC Interstitial water (meas): 1.7 $\mu\text{g}/\text{L}$	8 reps, 10 midges/rep
Concentration 5 Nom; Meas (mean of t_0 and day 10)	Dry Weight: 1400; 1200 $\mu\text{g}/\text{kg}$ OC-normal*: 22 $\mu\text{g}/\text{g}$ OC Interstitial water (meas): 2.9 $\mu\text{g}/\text{L}$	8 reps, 10 midges/rep
Concentration 6 Nom; Meas (mean of t_0 and day 10)	Dry Weight: 2800; 2500 $\mu\text{g}/\text{kg}$ OC-normal*: 45 $\mu\text{g}/\text{g}$ OC Interstitial water (meas): 5.3 $\mu\text{g}/\text{L}$	8 reps, 10 midges/rep
Control	Solvent and negative	8 reps, 10 midges/rep
LC ₅₀	> 2500 $\mu\text{g}/\text{kg}$ dry weight (DW)	Method: Inhibition concentration (TOXSTAT 3.5)
EC ₅₀ (95% confidence interval)	Growth: 780 (600 – 910) $\mu\text{g}/\text{kg}$ DW OC-normal: 14.2 (10.9-16.5) $\mu\text{g}/\text{g}$ OC	Method: Inhibition concentration (TOXSTAT 3.5)
NOEC	Survival: 1200 $\mu\text{g}/\text{kg}$ DW; 22 $\mu\text{g}/\text{g}$ OC; 2.9 $\mu\text{g}/\text{L}$ interstitial water Growth: 83 $\mu\text{g}/\text{kg}$ DW; 1.5 $\mu\text{g}/\text{g}$ OC; 0.24 $\mu\text{g}/\text{L}$ interstitial water	Method: nonparametric tests (TOXSTAT 3.5) Survival: Steel's Many-One Rank Test Growth: Bonferroni's Test

<i>C. dilutus</i>	Putt 2005a	
Parameter	Value	Comment
		p: 0.05 MSD: not reported Doc./Accept. points
LOEC	Survival: 2500 µg/kg DW; 45 µg/g OC; 5.3 µg/L interstitial water Growth: 170 µg/kg DW; 3 µg/g OC; 0.56 µg/L interstitial water	Same as above
MATC (GeoMean NOEC,LOEC) (µg/kg)	Survival: 1732 µg/kg DW; 31 µg/g OC; 3.9 µg/L interstitial water Growth: 119 µg/kg DW; 2 µg/g OC; 0.37 µg/L interstitial water	
% of control at NOEC	Survival: 88/83*100=106% Growth: 1.93/1.99*100=97%	
% of control at LOEC	Survival: 56/83*100=67% Growth: 1.53/1.99*100=77%	

Notes:

Protocol meets requirements USEPA Test method 100.2 “Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates” (USEPA, 2000) and 40 CFR, Part 158.

Radiolabeled bifenthrin used in toxicity testing.

*The OC-normalized sediment concentrations were calculated based on an OC content of 5.5%.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: 100-2=98

Acceptability: Temperature held to ±1°C (3), Random design (2), Minimum significant difference (1). Total: 100-6=94

Reliability score: Mean (98, 94) = 96

Toxicity Data Summary

Chironomus dilutus (Formerly *C. tentans*)

Xu Y, Spurlock F, Wang Z, Gan J (2007) Comparison of five methods for measuring sediment toxicity of hydrophobic contaminants. *Environ Sci Technol* 41: 8394-8399.

Relevance
Score: 100
Rating: R

Reliability
Score: 77.5
Rating: R

<i>C. dilutus</i>	Xu et al. 2007	
Parameter	Value	Comment
Test method cited	USEPA 2000	EPA-600/R-99/064
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	Formerly <i>C. tentans</i>
Family in North America?	Yes	
Age/size at start of test/growth phase	2 nd -3 rd instar larvae	
Source of organisms	Lab culture	Stock purchased from Aquatic Biosystems
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	93% (mean)	
Temperature	23±1°C	
Test type	Static renewal	80% water change daily
Photoperiod/light intensity	16 h light:8 h dark	
Overlying water	Reconstituted hard water	80% change daily; mass loss negligible
pH	Measured, not reported	Doc./Accept. points
Hardness	Measured, not reported	Doc./Accept. points
Alkalinity	Measured, not reported	Doc./Accept. points
Conductivity	Measured, not reported	Doc./Accept. points
Dissolved Oxygen	> 2.5 mg/L	
Sediment formulated?	No, field collected	
Organic carbon content	SDC: 1.44%	

<i>C. dilutus</i>	Xu et al. 2007	
Parameter	Value	Comment
	SR: 1.88% BM: 5.03%	
Particle size distribution (sand, silt, clay)	Not reported	< 2 mm (wet sieved) Doc. points
pH	Not reported	Doc. points
Percent moisture	Not reported	Doc. points
Sediment spike procedure	1 mL acetone added to 10 g sand, solvent evaporated, then added to 400 g sediment, tumbled overnight	Jar rolling method
Sediment spike equilibration time	30 d in dark, 4°C; 1 sediment batch equilibrated 90 d	Jars rolled 1x/wk for 2 h
Sediment to Solution ratio	30 g: 200 mL RHW	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Centrifugation	10,000 g, 30 min
Interstitial water chemical extraction method	SPME and LLE	
Interstitial water chemical analysis method	GC-ECD	
DOC	SDC: 37.8 mg/L SR: 29.7 mg/L BM: 66.8 mg/L SDC-90d: 22.8 mg/L	
Feeding	Daily 6 mg tetrafin	
Purity of test substance	98.0%	
Concentrations measured?	Yes – sediment & interstitial water	
Measured is what % of nominal?	Sediment: 74.7-94.8% Interstitial water: 87.6-114.9%	
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Chemical method documented?	Yes, GC-ECD	
Concentration of carrier (if any) in test solutions	None	
Concentration 1 (µg/kg)	Not reported, 6 concentrations	4 Reps, 10 per rep -Nominal & Meas. Conc. NR Doc./Accept. points
Concentration 2 (µg/kg)	Not reported,	4 Reps, 10 per rep

<i>C. dilutus</i>	Xu et al. 2007	
Parameter	Value	Comment
	6 concentrations	
Concentration 3 (µg/kg)	Not reported, 6 concentrations	4 Reps, 10 per rep
Concentration 4 (µg/kg)	Not reported, 6 concentrations	4 Reps, 10 per rep
Concentration 5 (µg/kg)	Not reported, 6 concentrations	4 Reps, 10 per rep
Concentration 6 (µg/kg)	Not reported, 6 concentrations	4 Reps, 10 per rep
Control	Solvent	4 Reps, 10 per rep
Sediment dry weight basis LC ₅₀ (95% confidence interval) µg/kg	SDC: 418 (291-588) SR: 560 (367-955) BM: 924 (611-1448) SDC-90d: 692 (420-1718)	Method: Not reported Doc./Accept. points
OC-normalized sediment basis LC ₅₀ (95% confidence interval) mg/kg OC	SDC: 29.0 (20.1-40.8) SR: 29.8 (19.5-50.8) BM: 18.3 (12.1-28.7) SDC-90d: 49.4 (30.0-122.7)	Same as above
Whole interstitial water basis LC ₅₀ (95% confidence interval) µg/L	SDC: 0.314 (0.239-0.424) SR: 0.258 (0.161-0.469) BM: 0.608 (0.446-0.867) SDC-90d: 0.402 (0.255-0.915)	Same as above
DOC-normalized interstitial water basis LC ₅₀ (95% confidence interval) mg/kg DOC	SDC: 8.31 (6.32-11.21) SR: 8.68 (5.42-15.79) BM: 9.10 (6.68-12.98) SDC-90d: 17.63 (11.18-40.13)	Same as above
Freely dissolved interstitial water basis (via SPME) LC ₅₀ (95% confidence interval) µg/L	SDC: 0.048 (0.041-0.056) SR: 0.053 (0.034-0.051) BM: 0.048 (0.041-0.058) SDC-90d: 0.051	Same as above

<i>C. dilutus</i>	Xu et al. 2007	
Parameter	Value	Comment
	(0.039-0.072)	

Notes:

Reliability points taken off for:

Documentation: Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water conductivity (1), Overlying water pH (1), Sediment particle size distribution (2), Statistical method (5), Hypothesis tests (8). Total: 100-31=69

Acceptability: Organisms randomly assigned (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water conductivity (1), Overlying water pH (1), Random design (2), Appropriate spacing between concentrations (2), Statistical method (2), Hypothesis tests (3). Total: 100-14=86

Reliability score: Mean (69, 86) = 77.5

Toxicity Data Summary

Hyalella azteca

Maul JD, Trimble AJ, Lydy MJ (2008b) Partitioning and matrix-specific toxicity of bifenthrin among sediments and leaf-sourced organic matter. *Environmental Toxicology and Chemistry* 27:945-952.

Relevance
Score: 100
Rating: R

Reliability
Score: 81
Rating: R

<i>H. azteca</i>	Maul et al. 2008b	
Parameter	Value	Notes
Results published or in signed, dated format?	Yes	
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family relevant for North America?	Yes	
Age/size at start of test/growth phase	14-21 days	
Source of organisms	Lab culture	
Have organisms been pre-exposed to contaminants?	No	
Were animals acclimated and disease-free?	Yes	
Were animals randomized?	Yes	
Were test vessels randomized?	Yes	
Test duration	10 d	

<i>H. azteca</i>	Maul et al. 2008b	
Parameter	Value	Notes
Data for multiple durations?	No	
Effect 1	Survival	
Control response 1	Sed: 90% Leaf: 93% Mix: 100%	
Temperature	18.9-20.8°C	Accept. points
Exposure type	Static renewal	50% water changed daily
Photoperiod/light intensity	16:8-h light:dark	
Overlying water source	EPA moderately hard water	
pH	7.3-7.77	
Hardness	Not reported	Doc./Accept. points
Alkalinity	Not reported	Doc./Accept. points
Dissolved oxygen	7.25-9.15 mg/L	Aerated continuously
Conductivity	366-395 µS/cm	
DOC	Not reported	
Sediment source	Natural soils/sediments	Soil collected from 15 km south Carbondale, IL
Organic carbon content	Sed: 0.69 ± 0.10% Coarse particulate organic matter: 42.6% Fine particulate organic matter: 40.0% Very fine particulate organic matter: 39.2%	All particulate matter from maple leaves
Particle size distribution (sand, silt, clay)	14%, 70%, 16%	Sieved to < 500 µm

<i>H. azteca</i>	Maul et al. 2008b	
Parameter	Value	Notes
Sediment spike method	Spiked with 50 uL of dosing stock solution (acetone), mixed by hand for 2 min.	Solvent not evaporated Accept. points
Sediment spike equilibration time	14 d at 4°C in darkness	Accept. points
Sediment-to-solution ratio	3 types of treatments Sed: 36.02 ± 0.01 g (dry wt.):500 mL water Leaf: 539.7 ± 0.1 mg (dry wt.): 500 mL water Mix: 18.01 ± 0.01 g sed (dry wt.), 269.8 ± 0.1 mg leaf (dry wt.):500 mL water	Each treatment had approximately 250 mg OC
Sediment extraction and chemical analysis method	Not reported	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Interstitial water DOC	Not applicable	
Feeding	1 mL of 1800 mg/L yeast-cerophyll-trout chow daily	
Purity of test chemical	¹⁴ C-labeled: 98.7% Unlabeled: 98.0%	
% Measured compared to nominal	87.5% for sediment and leaf matrices based on matrix spike recovery	
Were toxicity values calculated based on nominal or measured concentrations?	Nominal adjusted based on matrix spike recovery	

<i>H. azteca</i>	Maul et al. 2008b	
Parameter	Value	Notes
Concentration of carrier (if any) in test solutions	Acetone solvent not evaporated, 50 µL/ treatment	Accept. points
Concentration 1 Nom (µg/g OC)	0.07	3 reps, 10 per rep Doc. points – meas. concentrations not reported
Concentration 2 Nom (µg/g OC)	0.18	3 reps, 10 per rep
Concentration 3 Nom (µg/g OC)	0.69	3 reps, 10 per rep
Concentration 4 Nom (µg/g OC)	2.15	3 reps, 10 per rep
Concentration 5 Nom (µg/g OC)	8.33	3 reps, 10 per rep
Control Type	Solvent	3 reps, 10 per rep
LC ₅₀ (95% confidence interval) µg/g OC	Sed: 0.105 (0.078-0.130) Leaf: 0.065 (0.044-0.082) Mix: 0.152 (0.089-0.199)	Method: maximum likelihood probit & Abbott's correction
NOEC (µg/g OC)	Not reported Sed: not calculable Leaf: not calculable Mix: 0.065	Method: log-probit
LOEC (µg/g OC)	Sed: 0.065 Leaf: 0.065 Mix: 0.184	Method: Dunnett's test p: 0.05 MSD: not reported Doc./Accept. points
MATC (geometric mean NOEC, LOEC) µg/g OC	Mix: 0.109	
% control at NOEC	Not calculable	Doc./Accept. points
% control at LOEC	Not calculable	

ASTM = American Society for Testing and Materials, DOC = dissolved organic carbon, EC₅₀ = exposure concentration that causes effect in 50% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, MATC = Maximum acceptable toxicant concentration, meas = measured, MSD = minimum significant difference, NOEC = no observed effect concentration, nom = nominal, reps = replicates, p = p-value for statistical significance, TOC = total organic carbon, USEPA = United States Environmental Protection Agency.

Notes:

Reliability points taken off for:

Documentation (Table 9): Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Minimum significant difference (2), % control at NOEC/LOEC (2). Total: 100-16=84

Acceptability (Table 10): Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Overlying water hardness (1), Overlying water alkalinity (1), Temperature variation (3), Hypothesis tests (3). Total: 100-22=78

Reliability scores: Mean (84, 78)=81

Toxicity Data Summary

Hyaella azteca

Picard CR (2010a) 10-Day toxicity test exposing freshwater amphipods (*Hyaella azteca*) to bifenthrin applied to formulated sediment under static-renewal conditions. Performed by Springborn Smithers Laboratories, Wareham, MA, Study No. 136565.6133; submitted to Pyrethroid Working Group, Washington, DC.

Relevance
Score: 100
Rating: R

Reliability
Score: 94
Rating: R

<i>H. azteca</i>	Picard 2010a	
Parameter	Value	Comment
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyaellidae	
Genus	<i>Hyaella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	7 day old	
Source of organisms	Springborn Smithers lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Yes	
Test vessels randomized?	Not reported	Accept. points
Test duration	10 day	
Data for multiple times?	No	10 day only
Effect 1	Survival	
Control response 1	Negative control: 98% Solvent control: 93%	Pooled control: 95%
Effect 2	Growth (total dry wt. per organism)	
Control response 2	Pooled control: 0.11 mg	
Temperature	21- 25 °C	Accept. points
Test type	Static renewal	
Photoperiod/light intensity	16 h/8 h dark; 500-910 lux	
Overlying water	Well water	
pH	6.4-7.1	
Hardness	66-70 mg/L	

<i>H. azteca</i>	Picard 2010a	
Parameter	Value	Comment
Alkalinity	22 mg/L	
Conductivity	420-430 µmhos/cm	
Dissolved Oxygen	3.4 – 8.4 mg/L	
TOC	0.54 mg/L	
Ammonia-N	<0.01 – 0.30 mg/L	
Sediment source	Formulated	Method: OECD 218
Organic carbon content	2.1%	
Particle size distribution (sand, silt, clay)	71%, 7%, 22%	
Sediment spike procedure	Jar rolling technique. 10 mL of acetone evaporated from 0.05 kg sand	Initially rolled 4 h at 15 rpm at room temp. Mixed 2x/week for 2 h at room temp.
Sediment spike equilibration time	14 d at 4°C,	Accept. points
Sediment to Solution ratio	100:175 mL	100 mL sediment = 141 g wet wt or 89.6 g dry wt
Sediment extraction/analysis method	Ext/cleanup and instrument analysis	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Centrifugation	1200 g 15-30 min
Interstitial water chemical extraction	SPME	
Interstitial water chemical analysis	Not reported	
Interstitial water DOC	98-140 mg C/L	
Feeding	1 mL of YCT daily	Per replicate vessel
Purity of test substance	95.7%	
Measured is what % of nominal?	93-110% in sediment spikes at day 0	97-130% in stock solutions
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	0%	10 mL of acetone evaporated from 0.05 kg sand
Concentration 1 Nom; Meas (µg/kg)	0.25; 0.25	8 Reps, 10 per rep
Concentration 2 Nom; Meas (µg/kg)	0.5; 0.45	8 Reps, 10 per rep
Concentration 3 Nom; Meas (µg/kg)	1.0; 0.92	8 Reps, 10 per rep

<i>H. azteca</i>	Picard 2010a	
Parameter	Value	Comment
Concentration 4 Nom; Meas (µg/kg)	2.0; 1.9	8 Reps,10 per rep
Concentration 5 Nom; Meas (µg/kg)	4.0; 3.6	8 Reps,10 per rep
Concentration 6 Nom; Meas (µg/kg)	8.0; 7.7	8 Reps,10 per rep
Control	Solvent and negative controls	8 Reps,10 per rep
LC ₅₀ (95% confidence interval)	Dry weight basis 3.7 (3.3-4.1) µg/kg DW OC-normal basis 0.18 (0.16-0.20) µg/g OC	Method: Spontaneous Logit analysis (TOXSTAT)
EC ₅₀ (95% confidence interval)	Dry weight basis > 7.7 µg/kg DW OC-normal basis > 0.37 µg/g OC	Method: Linear interpolation (TOXSTAT); empirically estimated
NOEC	Dry weight basis Survival: 1.9 µg/kg DW Growth: 0.45 µg/kg DW OC-normal basis Survival: 0.09 µg/g OC Growth: 0.02 µg/g OC	Method: Bonferroni's t-test p: 0.05 MSD: Not reported Doc./Accept. points
LOEC	Dry weight basis Survival: 3.6 µg/kg DW Growth: 0.92 µg/kg DW OC-normal basis Survival: 0.17 µg/g OC Growth: 0.04 µg/g OC	Same as above
MATC (GeoMean NOEC,LOEC)	Dry weight basis Survival: 2.6 µg/kg DW Growth: 0.64 µg/kg DW OC-normal basis Survival: 0.12 µg/g OC Growth: 0.03 µg/g OC	
% of control at NOEC	Survival: 88%/95%=93% Growth: 0.1 mg/0.11 mg=91%	Pooled controls
% of control at LOEC	Survival: 50%/95%=53% Growth: 0.09mg/0.11mg=82%	Pooled controls

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Measured sediment concentrations are the mean of measurements at day 0 and day 10.

Although the study states pore water results are in a supplemental report, the data was never made available due to analytical and sample holding time issues.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total: $100-2=98$

Acceptability: Spike equilibration time (4), Temperature variation (3), Random design (2), Minimum significant difference (1). Total: $100-10=90$

Reliability Score: Mean (98, 90) = 94

Toxicity Data Summary

Hyalella azteca

Weston DP, Jackson CJ (2009) Use of engineered enzymes to identify organophosphate and pyrethroid-related toxicity in toxicity identification evaluations. Environmental Science & Technology 43:5514-5520.

Relevance
Score: 100
Rating: R

Reliability
Score: 74.5
Rating: R

<i>H. azteca</i>	Weston & Jackson 2009	
Parameter	Value	Notes
Results published or in signed, dated format?	Yes	
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family relevant for North America?	Yes	
Age/size at start of test/growth phase	7-10 days	
Source of organisms	Lab culture	
Have organisms been pre-exposed to contaminants?	No	
Were animals acclimated and disease-free?	Yes	
Were animals randomized?	Not reported	Accept. points

<i>H. azteca</i>	Weston & Jackson 2009	
Parameter	Value	Notes
Were test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple durations?	No	
Effect 1	Survival	
Control response 1	≥95%	
Temperature	23 ± 0.1°C	
Exposure type	Static renewal	100 mL water changed 3x/day
Photoperiod/light intensity	Not reported	Doc. points
Overlying water source	EPA moderately hard water	
pH	6.76 ± 0.13	
Hardness	Not reported	Doc./Accept. points
Alkalinity	Not reported	Doc./Accept. points
Dissolved oxygen	7.34 ± 0.32 mg/L	
Conductivity	367 ± 25 µS/cm	
Sediment source	3 natural soils/sediments: TON, BAY, LPH	
Organic carbon content	TON: 0.56% BAY: 1.77% LPH: 4.43%	
Particle size distribution (sand, silt, clay)	TON: 14%, 62%, 24% BAY: 52%, 38%, 10% LPH: 46%, 47%, 7%	
Sediment spike procedure	Sediment spiked in	Solvent not

<i>H. azteca</i>	Weston & Jackson 2009	
Parameter	Value	Notes
	bulk in 1L jars with ≤200 µL acetone stock solutions; homogenized by hand mixing & rolling 1 h	evaporated Accept. points
Sediment spike equilibration time	14 d at 4°C in darkness	Accept. points
Sediment-to-solution ratio	75 mL sediment:250 mL water	
Sediment extraction/analysis method	Extracted via Tenax for 6 h or 24 h, Tenax was solvent extracted & analyzed via LSC	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Not applicable	
Interstitial water chemical analysis method	Extracted via SPME fibers equilibrated for 28 d on shaker table. Fibers extracted with hexane for 36 h; analyzed via LSC	
Interstitial water TOC; DOC	Not reported	
Feeding	1 mL of yeast- cerophyll-trout chow	
Purity of test chemical	¹⁴ C-labeled: ≥ 98% Unlabeled: technical	
% Measured compared to nominal	Not reported	Accept. points
Were toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in	Acetone solvent not	Accept. points

<i>H. azteca</i>	Weston & Jackson 2009	
Parameter	Value	Notes
test solutions	evaporated, conc. not reported	
Concentration 1 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep Doc. points - nom & meas concentrations not reported Accept. points – conc. spacing not reported
Concentration 2 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 3 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 4 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 5 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 6 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Control Type	Negative and solvent	4 reps, 10 per rep
LC ₅₀ (95% confidence interval) µg/g OC	Test 1: 0.76 (0.69-0.85) Test 2: 0.73 (0.62-0.84) Test 3: 0.76 (0.69-0.85) Test 4: 0.89 (0.74-1.04) Test 5: 0.97 (0.82-1.10) Test 6: 0.73 (0.62-0.84)	Method: probit

ASTM = American Society for Testing and Materials, DOC = dissolved organic carbon, EC₅₀ = exposure concentration that causes effect in 50% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, MATC = Maximum acceptable toxicant concentration, meas = measured, MSD = minimum significant difference, NOEC = no observed effect concentration, nom = nominal, reps = replicates, p = p-value for statistical significance, TOC = total organic carbon, USEPA = United States Environmental Protection Agency.

Notes:

Different letters after LC₅₀ or EC₅₀ values within a single concentration type indicates that they are significantly different ($p \leq 0.05$).

Reliability points taken off for:

Documentation (Table 9): Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Photoperiod (3), Hypothesis tests (8). Total: 100-25=75

Acceptability (Table 10): Measured concentrations within 20% nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Overlying water hardness (1), Overlying water alkalinity (1), Random design (2), Hypothesis tests (3). Total: 100-26=74

Reliability scores: Mean (75, 74)=74.5

Appendix B2

Supplemental data rated RL, LR, or LL

Toxicity Data Summary

Ampelisca abdita

Anderson BD, Lowe S, Phillips BM, Hunt JW, Vorhees J, Clark S, Tjeerdema RS (2008) Relative sensitivities of toxicity test protocols with the amphipods *Eohaustorius estuarius* and *Ampelisca abdita*. *Ecotoxicology and Environmental Safety* 69:24-31.

Relevance

Score: 70

Rating: L

Reliability

Score: 63.5

Rating: L

*Relevance points taken off for: saltwater species (15); control results not reported (7.5)

<i>A. abdita</i>	Anderson et al. 2008	
Parameter	Value	Comment
Test method cited	USEPA 1994	Estuarine and marine amphipods
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Ampeliscidae	
Genus	<i>Ampelisca</i>	
Species	<i>abdita</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Not reported	Doc./Accept. points
Source of organisms	Lab culture	Brezina & Assoc.
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Not reported	Accept. points
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	Not reported	Doc./Accept. points
Temperature	20°C	
Test type	Static	
Photoperiod/light intensity	Not reported	Doc. points
Overlying water	Filtered seawater diluted with distilled water to 28 ‰ salinity	
pH	Not reported	Accept. points

<i>A. abdita</i>	Anderson et al. 2008	
Parameter	Value	Comment
Hardness mg/L as CaCO ₃	Not reported	Doc./Accept. points
Alkalinity mg/L as CaCO ₃	Not reported	Doc./Accept. points
Conductivity	Not reported, 28 ‰ salinity	Doc./Accept. points
Dissolved Oxygen	Not measured. Slow aeration of test vessels	Doc./Accept. points
TOC/DOC	Not reported	
Chemical analysis?/Method	No	
Sediment formulated?	Yes	Equal parts Salinas River sediment from a reference site & clean sand, amended with 0.75% peat
Organic carbon	0.78%	
Particle size distribution (sand, silt, clay)	13.57% med sand; 48.17% fine sand; 38.27% silt+clay (% fines)	
pH	Not reported	Doc. points
Percent solids	350 mL water: 107.5 g sed	
Sediment spike procedure	50 mL acetone sol'n added to empty jar & allowed to evaporate; sediment & water added to jar & rolled for 1 st 24 h of eq. time	
Sediment spike equilibration time	7 d	Accept. points
Sediment to Solution ratio	50mL:200mL	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
pH	Not applicable	
TOC; DOC	Not applicable	
Feeding	Not reported	Doc./Accept. points
Purity of test substance	Not reported	Doc./Accept. points
Concentrations measured?	Yes	
Measured is what % of nominal?	47.0-84.4%	Accept. points

<i>A. abdita</i>	Anderson et al. 2008	
Parameter	Value	Comment
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Chemical method documented?	Yes	EPA 1660
Concentration of carrier (if any) in test solutions	0 (evaporated)	
Concentration 1 Nom (mg/kg DW)	Test 1: 0.18 Test 2: 0.18	5 reps, 5 org/rep
Concentration 2 Nom; Meas (mg/kg DW)	Test 1: 0.32; 0.242 Test 2: 0.32; 0.270	5 reps, 5 org/rep
Concentration 3 Nom; Meas (mg/kg DW)	Test 1: 1.00; 0.470 Test 2: 1.0; 0.705	5 reps, 5 org/rep
Concentration 4 Nom; Meas (mg/kg DW)	Test 1: 3.2; 2.790 Test 2: 3.2; 2.65	5 reps, 5 org/rep
Concentration 5 Nom (mg/kg DW)	Test 1: 10 Test 2: 10	5 reps, 5 org/rep
Control	Solvent and negative	5 reps, 5 org/rep
LC ₅₀ (mg/kg DW)	Test 1: 1.373 Test 2: 0.522 Mean: 0.948 (SD=0.432)	Method: ToxCalc software

Notes:

Reliability points taken off for:

Documentation: Organism age (5), Chemical purity (5), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Sediment pH (1), Photoperiod (3), Hypothesis tests (8). Total: 100-27=73

Acceptability: Control response (9), Chemical purity (10), Measured concentrations within 20% nominal (4), Sediment spike equilibration time (2), Organisms age (3), Organisms randomly assigned (1), Feeding (3), Organisms acclimated (1), Overlying water hardness (2), Overlying water alkalinity (2), Overlying water dissolved oxygen (6), Overlying water conductivity (1), Overlying water pH (2), Temperature variation (3), Random design (2), Hypothesis tests (3). Total: 100-54=46

Reliability score: Mean (73, 46)=63.5

Toxicity Data Summary

Chironomus dilutus

Harwood AD, Landrum PF, Lydy MJ (2013a) Bioavailability-based toxicity endpoints of bifenthrin for *Hyalella azteca* and *Chironomus dilutus*. Chemosphere 90:1117-1122.

Relevance

Score: 90

Rating: R

Reliability

Score: 71

Rating: L

*Relevance points taken off for: No standard method cited (10)

C. dilutus	Harwood et al. 2013a	
Parameter	Value	Notes
Results published or in signed, dated format?	Yes	
Test method cited	Not reported	Accept. points
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family relevant for North America?	Yes	
Age/size at start of test/growth phase	3 rd instar	
Source of organisms	Lab culture	
Have organisms been pre-exposed to contaminants?	No	
Were animals acclimated and disease-free?	Yes	

C. dilutus	Harwood et al. 2013a	
Parameter	Value	Notes
Were animals randomized?	Not reported	Accept. points
Were test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple durations?	No	
Effect 1	Survival	
Control response 1	≥80%	
Effect 2	Immobilization	Inability to perform typical S-shaped swimming motion when gently prodded
Control response 2	≤ 15% *	
Temperature	23 ± 0.1°C	
Exposure type	Static renewal	100 mL water changed 3x/day
Photoperiod/light intensity	Not reported	Doc. points
Overlying water source	EPA moderately hard water	
pH	6.76 ± 0.13	
Hardness	Not reported	Doc., Accept. points
Alkalinity	Not reported	Doc., Accept. points
Dissolved oxygen	7.34 ± 0.32 mg/L	
Conductivity	367 ± 25 µS/cm	
TOC; DOC	Not reported	
Chemical analysis method	Not analyzed	

C. dilutus	Harwood et al. 2013a	
Parameter	Value	Notes
Sediment source	3 natural soils/sediments: TON, BAY, LPH	
Organic carbon content	TON: 0.56% BAY: 1.77% LPH: 4.43%	
Particle size distribution (sand, silt, clay)	TON: 14%, 62%, 24% BAY: 52%, 38%, 10% LPH: 46%, 47%, 7%	
Sediment spike method	Sediment spiked in bulk in 1L jars with ≤ 200 μ L acetone stock solutions; homogenized by hand mixing & rolling 1 h	Solvent not evaporated Accept. points
Sediment spike equilibration time	14 d at 4°C in darkness	Accept. points
Sediment-to-solution ratio	50 g sediment (dry wt.) and < 250 mL of water	
Sediment extraction and chemical analysis method	Extracted via Tenax for 6 h or 24 h, Tenax was solvent extracted & analyzed via LSC	
Interstitial water monitored?	Yes	
Interstitial water extraction method	Not applicable	
Interstitial water chemical analysis method	Extracted via SPME fibers equilibrated for 28 d on shaker table. Fibers extracted with hexane for 36 h; analyzed via LSC	
Interstitial water DOC	Not reported	
Feeding	1 mL of 6 g/L Tetramin daily	
Purity of test chemical	¹⁴ C-labeled: $\geq 98\%$ Unlabeled: technical	

C. dilutus	Harwood et al. 2013a	
Parameter	Value	Notes
% Measured compared to nominal	Not reported	Accept. points
Were toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	Acetone solvent not evaporated, conc. not reported	Accept. points
Concentration 1 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep Doc. points - nom & meas concentrations not reported Accept. points – conc. spacing not reported
Concentration 2 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 3 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 4 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 5 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 6 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Control Type	Negative and solvent	4 reps, 10 per rep
LC ₅₀ (95% confidence interval) Sediment dry weight basis	TON: 0.087 (0.071-0.105) ^a BAY: 0.108 (0.086-0.125) ^a LPH: 0.189 (0.144-0.229) ^b	Method: log-probit

C. dilutus	Harwood et al. 2013a	
Parameter	Value	Notes
µg/kg DW		
LC ₅₀ (95% confidence interval) OC-normalized sediment basis µg/g OC	TON: 9.04 (7.43-10.9) ^a BAY: 6.08 (4.86-7.06) ^b LPH: 4.26 (3.26-5.17) ^b	Method: log-probit
LC ₅₀ (95% confidence interval) SPME basis (normalized to PDMS conc.) µg/mL PDMS	TON: 2.44 (1.95-2.98) ^a BAY: 1.75 (1.35-2.010) ^a LPH: 0.905 (0.634-1.18) ^b	Method: log-probit
LC ₅₀ (95% confidence interval) Calculated interstitial water concentration basis ng/L	TON: 8.84 (7.08-10.8) ^a BAY: 6.35 (4.92-7.30) ^a LPH: 3.28 (2.30-4.27) ^b	Method: log-probit
LC ₅₀ (95% confidence interval) 6 h Tenax extractable concentration basis µg/g OC	TON: 2.74 (2.09-3.51) ^a BAY: 1.76 (1.39-2.09) ^a LPH: 0.576 (0.422-0.691) ^b	Method: log-probit
LC ₅₀ (95% confidence interval) 24 h Tenax extractable concentration basis µg/g OC	TON: 4.57 (3.73-5.62) ^a BAY: 2.27 (1.91-2.66) ^b LPH: 1.49 (1.12-1.92) ^c	Method: log-probit
EC ₅₀ (95% confidence interval) Sediment dry weight basis µg/kg DW	TON: 0.052 (0.044-0.058) ^a BAY: 0.071 (0.059-0.077) ^b LPH: 0.083 (0.057-0.103) ^b	Method: log-probit
EC ₅₀ (95% confidence interval) OC-normalized sediment basis µg/g OC	TON: 5.46 (4.60-6.06) ^a BAY: 4.02 (3.38-4.37) ^b LPH: 1.87 (1.29-2.32) ^c	Method: log-probit

C. dilutus	Harwood et al. 2013a	
Parameter	Value	Notes
EC ₅₀ (95% confidence interval) SPME basis (normalized to PDMS conc.) µg/mL PDMS	TON: 1.37 (1.09-1.56) ^a BAY: 1.15 (1.01-1.26) ^a LPH: 0.411 (0.281-0.505) ^b	Method: log-probit
EC ₅₀ (95% confidence interval) Calculated interstitial water concentration basis ng/L	TON: 4.97 (3.98-5.66) ^a BAY: 4.17 (3.67-4.56) ^a LPH: 1.49 (1.02-1.84) ^b	Method: log-probit
EC ₅₀ (95% confidence interval) 6 h Tenax extractable concentration basis µg/g OC	TON: 1.39 (1.00-1.65) ^a BAY: 1.10 (0.952-1.20) ^a LPH: 0.225 (0.150-0.287) ^b	Method: log-probit
EC ₅₀ (95% confidence interval) 24 h Tenax extractable concentration basis µg/g OC	TON: 2.75 (2.34-3.03) ^a BAY: 1.60 (1.44-1.71) ^b LPH: 0.626 (0.424-0.758) ^c	Method: log-probit

ASTM = American Society for Testing and Materials, DOC = dissolved organic carbon, EC₅₀ = exposure concentration that causes effect in 50% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, MATC = Maximum acceptable toxicant concentration, meas = measured, MSD = minimum significant difference, NOEC = no observed effect concentration, nom = nominal, reps = replicates, p = p-value for statistical significance, TOC = total organic carbon, USEPA = United States Environmental Protection Agency.

Notes:

* Control response for immobility endpoint was acquired via personal communication with the author, Amanda Harwood (amandaharwood@gmail.com).

Different letters after LC₅₀ or EC₅₀ values within a single concentration type indicates that they are significantly different ($p \leq 0.05$).

Reliability points taken off for:

Documentation (Table 9): Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Photoperiod (3), Hypothesis tests (8). Total: 100-25=75

Acceptability (Table 10): Standard method (5), Measured concentrations within 20% nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Overlying water hardness (1), Overlying water alkalinity (1), Random design (2), Dilution factor (2), Hypothesis tests (3). Total: 100-33=67

Reliability scores: Mean (75, 67) = 71

Toxicity Data Summary

Chironomus dilutus

Trimble AJ, Belden JB, Mueting SA, Lydy MJ (2010) Determining modifications to bifenthrin toxicity and sediment binding affinity from varying potassium chloride concentrations in overlying water. *Chemosphere* 80:53-59.

Relevance

Score: 85

Rating: L

Reliability

Score: 77

Rating: R

*Relevance points taken off for: Acceptable bioavailable concentrations not used (nominal instead of measured; 15).

<i>C. dilutus</i>	Trimble et al. 2010	
Parameter	Value	Comment
Test method cited	EPA 2000	EPA/600/R-99-064
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	2 rd instar larvae	
Source of organisms	Lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Yes	
Test duration	10 d	
Data for multiple times?	Not reported	
Effect 1	Survival	
Control response 1	>88%	
Temperature	23 ± 1.8 °C	Accept. points
Test type	Static renewal	50% water change daily
Photoperiod/light intensity	16 h: 8 h dark	
Overlying water	Moderately hard reconstituted water	
pH	Not reported but measured, & within EPA guidelines	Doc. points
Hardness mg/L as CaCO ₃	Not reported	Doc./Accept. points

<i>C. dilutus</i>	Trimble et al. 2010	
Parameter	Value	Comment
Alkalinity mg/L as CaCO ₃	Not reported	Doc./Accept. points
Conductivity	Not reported but measured, & within EPA guidelines	Doc. points
Dissolved Oxygen	Not reported but measured, & within EPA guidelines	Doc. points
Ammonia-N	< 0.1 mg/L	
Sediment formulated?	No	Touch of Nature field station reference site, Carbondale, IL
Organic carbon	0.983 ± 0.10%	
Particle size distribution (sand, silt, clay)	14%, 72%, 14%	
Sediment spike method	Solvent & volume not reported; sediment-water slurries were spiked in bulk and mixed for 10 min	Doc./Accept. points
Sediment spike equilibration time	14 d at 4°C	Accept. points
Sediment to Solution ratio	40 g wt.: 250 mL	
Sediment extraction/analysis method	Solvent extraction and GC/ECD	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
DOC	Not applicable	
Feeding	Every other day 1mL of 6 g/L TetraFin fish food	
Purity of test substance	99.7% technical Radiolabeled: 97.7%, 41.97 µCi/µmol	
Measured is what % of nominal?	80-120%	
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Chemical method documented?	Yes	
Concentration of carrier (if any) in test solutions	Not reported	Accept. points
Concentration 1 Nom (ng/g)	9.16	Meas. conc. not

<i>C. dilutus</i>	Trimble et al. 2010	
Parameter	Value	Comment
		reported Doc. Points 10 per rep, # of reps not reported Accept. points
Concentration 2 Nom (ng/g)	18.3	
Concentration 3 Nom (ng/g)	36.6	
Concentration 4 Nom (ng/g)	73.3	
Concentration 5 Nom (ng/g)	146	
Concentration 6 Nom (ng/g)	293	
Concentration 6 Nom (ng/g)	586	
Control	Solvent and negative	
LC ₅₀	Dry weight basis 0.0793 mg/kg DW OC-normal basis 8.1 mg/kg OC	Method: probit 95% fiducial intervals shown in Fig. 1

Notes:

Reliability points taken off for:

Documentation: Measured concentrations (3), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Sediment spike method (4), Hypothesis tests (8). Total: 100-21=79

Acceptability: Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Temperature variation (3), Overlying water hardness (1), Overlying water alkalinity (1), Adequate replication (2), Hypothesis tests (3). Total: 100-25=75

Reliability score: Mean (79, 75) = 77

Toxicity Data Summary

Eohaustorius estuarius

Anderson BS, Lowe S, Phillips BM, Hunt JW, Vorhees J, Clark S, Tjeerdema RS (2008) Relative sensitivities of toxicity test protocols with the amphipods *Eohaustorius estuarius* and *Ampelisca abdita*. *Ecotoxicology and Environmental Safety* 69:24-31.

Relevance

Score: 70

Rating: L

Reliability

Score: 63.5

Rating: L

*Relevance points taken off for: saltwater species (15); control results not reported (7.5)

<i>E. estuarius</i>	Anderson et al. 2008	
Parameter	Value	Comment
Test method cited	USEPA 1994	Estuarine and marine amphipods
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Haustoriidae	
Genus	<i>Eohaustorius</i>	
Species	<i>estuarius</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Not reported	Doc./Accept. points
Source of organisms	Lab culture	Northwestern Aquatic Sciences
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Not reported	Accept. points
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	Not reported	Doc./Accept. points
Temperature	15°C	
Test type	Static	
Photoperiod/light intensity	Not reported	Doc. points
Overlying water	Filtered seawater diluted with distilled water to 20 ‰ salinity	
pH	Not reported	Accept. points

<i>E. estuarius</i>	Anderson et al. 2008	
Parameter	Value	Comment
Hardness mg/L as CaCO ₃	Not reported	Doc./Accept. points
Alkalinity mg/L as CaCO ₃	Not reported	Doc./Accept. points
Conductivity	Not reported, 20 ‰ salinity	Doc./Accept. points
Dissolved Oxygen	Not measured. Slow aeration of test vessels	Doc./Accept. points
TOC/DOC	Not reported	
Chemical analysis?/Method	No	
Sediment formulated?	Yes	Equal parts Salinas River sediment from a reference site & clean sand, amended with 0.75% peat
Organic carbon	0.78%	
Particle size distribution (sand, silt, clay)	13.57% med sand; 48.17% fine sand; 38.27% silt+clay (% fines)	
pH	Not reported	Doc. points
Percent solids	350 mL water: 107.5 g sed	
Sediment spike procedure	50 mL acetone sol'n added to empty jar & allowed to evaporate; sediment & water added to jar & rolled for 1 st 24 h of eq. time	
Sediment spike equilibration time	7 d	Accept. points
Sediment to Solution ratio	50mL:200mL	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
pH	Not applicable	
TOC; DOC	Not applicable	
Feeding	Not reported	Doc./Accept. points
Purity of test substance	Not reported	Doc./Accept. points
Concentrations measured?	Yes	
Measured is what % of nominal?	47.0-84.4%	Accept. points

<i>E. estuarius</i>	Anderson et al. 2008	
Parameter	Value	Comment
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Chemical method documented?	Yes	EPA 1660
Concentration of carrier (if any) in test solutions	0 (evaporated)	
Concentration 1 Nom (mg/kg DW)	Test 1: 0.0018 Test 2: 0.0018	5 reps, 5 org/rep
Concentration 2 Nom; Meas (mg/kg DW)	Test 1: 0.0032; 0.0023 Test 2: 0.0032; 0.0027	5 reps, 5 org/rep
Concentration 3 Nom; Meas (mg/kg DW)	Test 1: 0.01; 0.008 Test 2: 0.01; 0.008	5 reps, 5 org/rep
Concentration 4 Nom; Meas (mg/kg DW)	Test 1: 0.32; 0.020 Test 2: 0.32; 0.018	5 reps, 5 org/rep
Concentration 5 Nom (mg/kg DW)	Test 1: 0.1 Test 2: 0.1	5 reps, 5 org/rep
Control	Solvent and negative	5 reps, 5 org/rep
LC ₅₀ (mg/kg DW)	Test 1: 0.009 Test 2: 0.006 Mean: 0.008 (SD=0.008)	Method: ToxCalc software

Notes:

Reliability points taken off for:

Documentation: Organism age (5), Chemical purity (5), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Sediment pH (1), Photoperiod (3), Hypothesis tests (8). Total: 100-27=73

Acceptability: Control response (9), Chemical purity (10), Measured concentrations within 20% nominal (4), Sediment spike equilibration time (2), Organisms age (3), Organisms randomly assigned (1), Feeding (3), Organisms acclimated (1), Overlying water hardness (2), Overlying water alkalinity (2), Overlying water dissolved oxygen (6), Overlying water conductivity (1), Overlying water pH (2), Temperature variation (3), Random design (2), Hypothesis tests (3). Total: 100-54=46

Reliability score: Mean (73, 46)=63.5

Toxicity Data Summary

Hyalella azteca

Amweg EL, Weston DP (2007a) Whole sediment toxicity identification evaluation tools for pyrethroid insecticides: I. Piperonyl butoxide addition. Environ Toxicol Chem 26:2389-2396.

Relevance
Score: 100
Rating: R

Reliability
Score: 64
Rating: L

<i>H. azteca</i>	Amweg & Weston 2007a	
Parameter	Value	Comment
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	7-10 d	
Source of organisms	Not reported	Doc. points
Have organisms been exposed to contaminants?	Not reported	Accept. points
Animals acclimated and disease-free?	Not reported	Accept. points
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	90-100%	96% average
Temperature	23°C	
Test type	Static-renewal	Daily renewal
Photoperiod/light intensity	16 h light:8 h dark	
Overlying water	Moderately hard water	Reconstituted from MQ water
pH	Not reported	Doc./ Accept. points
Hardness mg/L as CaCO ₃	Measured, Not reported	Doc./ Accept. points
Alkalinity mg/L as CaCO ₃	Measured, Not reported	Doc./ Accept. points
Conductivity	Measured, Not reported	Doc./ Accept. points
Dissolved Oxygen	Measured, Not reported	Doc./ Accept. points
Sediment formulated?	20:80 blend of Lake Anza and San Pablo Dam reservoir sediments	

<i>H. azteca</i>	Amweg & Weston 2007a	
Parameter	Value	Comment
Organic carbon content	1.7-2.1%	
Particle size distribution (sand, silt, clay)	Not reported	Sieved to < 1 mm Doc. points
Sediment spike procedure	200 µL acetone/kg, mixed with electric drill	Accept. points
Sediment spike equilibration time	12-26 day at 4°C	Accept. points
Sediment to Solution ratio	75 mL:300 mL water	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
DOC	Not applicable	
Feeding	Yeast, cerophyll, trout chow mix	Daily; no amounts
Purity of test substance	>98%	Chem service
Concentrations measured?	Yes	
Measured is what % of nominal?	83% in sediment	
Toxicity values calculated based on nominal or measured concentrations?	Measured sediment	
Concentration of carrier (if any) in test solutions	200 uL acetone/kg wet sediment	All pesticides and PAH
Concentration 1	Not reported	10 per rep, 3-4 reps -Nominal & Meas. Conc. NR Doc./ Accept. points
Concentration 2	Not reported	
Concentration 3	Not reported	
Concentration 4	Not reported	
Concentration 5	Not reported	
Concentration 6	Not reported	
Control	Solvent and negative	10 per rep, 3-4 reps
LC ₅₀ (95% confidence interval) µg/g OC	0.26 (0.24-0.28)	Method: Spearman-Kärber method

Notes:

Protocol follows EPA 2000 Methods for measuring the toxicity and bioaccumulation. 2nd. EPA/600/R-99/064.

Study also determined LC₅₀ of PBO alone and a mixture of bifenthrin, chlorpyrifos and PBO.

Reliability points taken off for:

Documentation: Organism source (4), Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Sediment particle size distribution (1), Hypothesis tests (8). Total: 100-31=69

Acceptability: Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organism not contaminated prior (3), Organisms randomly assigned (1), Organisms acclimated/disease-free (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (5), Overlying water conductivity (1), Overlying water pH (1), Temperature variation (3), Number of concentrations (3), Random design (2), Appropriate spacing of concentrations (2), Hypothesis tests (3). Total: 100-41=59

Reliability score: Mean (69, 59) = 64

Toxicity Data Summary

Hyalella azteca

Amweg EL, Weston DP (2007b) Whole sediment toxicity identification evaluation tools for pyrethroid insecticides: II. Esterase addition. Environ Toxicol Chem 26: 2397-2404.

Relevance
Score: 100
Rating: R

Reliability
Score: 66
Rating: L

<i>H. azteca</i>	Amweg & Weston 2007b	
Parameter	Value	Comment
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	7-14 d	
Source of organisms	Not reported	Doc. points
Have organisms been exposed to contaminants?	Not reported	Accept. points
Animals acclimated and disease-free?	Not reported	Accept. points
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Not reported	Accept. points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	>90%	96% average
Temperature	23°C	
Test type	Static-renewal	Daily renewal
Photoperiod/light intensity	16 h light:8 h dark	
Overlying water	Moderately hard water	Reconstituted from MQ water
pH	Measured, Not reported	Doc./ Accept. points
Hardness mg/L as CaCO ₃	Measured, Not reported	Doc./ Accept. points
Alkalinity mg/L as CaCO ₃	Measured, Not reported	Doc./ Accept. points
Conductivity	Measured, Not reported	Doc./ Accept. points
Dissolved Oxygen	Measured, Not reported	Doc./ Accept. points
Sediment formulated?	20:80 blend of Lake Anza and San Pablo Dam reservoir sediments	
Organic carbon content	2%	

<i>H. azteca</i>	Amweg & Weston 2007b	
Parameter	Value	Comment
Particle size distribution (sand, silt, clay)	Not reported	Sieved to < 1 mm Doc. points
Sediment spike procedure	200 µL acetone/kg, mixed with electric drill	Accept. points
Sediment spike equilibration time	7-14 days at 4°C	Accept. points
Sediment to Solution ratio	50-75 mL sediment:275 mL water	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Feeding	Yeast, cerophyll, trout chow mix	Daily; no amounts
Purity of test substance	Technical	Chem service
Concentrations measured?	Yes	
Measured is what % of nominal?	61%	Accept. points
Toxicity values calculated based on nominal or measured concentrations?	Not reported, likely nominal	
Chemical method documented?	Yes	Solvent extraction, cleanup, GC/ECD
Concentration of carrier (if any) in test solutions	<200 uL acetone/kg wet sediment	
Concentration 1	5 concentrations with a 0.6 dilution factor	10 per rep, 3 reps -Nominal & Meas. Conc. NR Doc./ Accept. points
Concentration 2	Not reported	
Concentration 3	Not reported	
Concentration 4	Not reported	
Concentration 5	Not reported	
Control type	Negative	10 per rep, 3 reps
LC ₅₀ (95% confidence interval) µg/g OC	0.22	Method: Spearman-Kärber method

Notes:

Reliability points taken off for:

Documentation: Organism source (4), Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Sediment particle size distribution (1), Hypothesis tests (8). Total: 100-31=69

Acceptability: Measured concentration within 20% of nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organism not contaminated prior (3), Organisms randomly assigned (1), Organisms acclimated/disease-free (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Temperature variation (3), Random design (2), Hypothesis tests (3).

Total: 100-37=63

Reliability score: Mean (69, 63) = 66

Toxicity Data Summary

Hyalella azteca

Amweg EL, Weston DP, Ureda NM (2005) Use and toxicity of pyrethroid pesticides in the Central Valley, California, UAS. Environ Toxicol Chem 24: 966-972.

Relevance

Score: 85

Rating: L

Reliability

Score: 70

Rating: L

*Relevance points taken off for: Toxicity values were not based on acceptable bioavailable concentrations (15). They were based on nominal (not measured) concentrations.

<i>H. azteca</i>	Amweg et al. 2005	
Parameter	Value	Comment
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	6-10 d	< 350 µm, < 500 µm
Source of organisms	Not reported	Doc. points
Have organisms been exposed to contaminants?	Not reported	Accept. points
Animals acclimated and disease-free?	Not reported	Accept. Points
Animals randomized?	Not reported	Accept. Points
Test vessels randomized?	Not reported	Accept. Points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	94%	
Effect 2	Growth	
Control response 2	Negative: 75-85 µg Solvent: 78-90 µg	From Fig. 2A
Temperature	23°C	Accept. Points
Test type	Static-renewal	80% renewal every other day
Photoperiod/light intensity	16:8 h/ not stated	
Dilution water (overlying water)	Moderately hard water	Reconstituted from MQ water
pH	Measured, Not reported	Doc. points
Hardness mg/L as CaCO ₃	Measured, Not reported	Doc. points
Alkalinity mg/L as CaCO ₃	Measured, Not reported	Doc. points

<i>H. azteca</i>	Amweg et al. 2005	
Parameter	Value	Comment
Conductivity	Measured, Not reported	Doc. points
Dissolved Oxygen	Measured, Not reported	Doc. points
Sediment source	3 Natural sediments: American River (AR) Del Puerto Creek (DPC) Pacheco Creek (PC)	
Organic carbon content	AR: 1.4% DPC: 1.1% PC: 6.5%	
Particle size distribution (sand, silt, clay)	% silts & clays AR: 43.1% DPC: 31.7% PC: 21.3%	
Sediment spike procedure	<200 µL acetone /kg , mixed with electric drill	Accept. points
Sediment spike equilibration time	11-12 day at 4°C	Accept. points
Sediment to Solution ratio	50-75 mL:300 mL water	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
DOC	Not applicable	
Feeding	Yeast, cerophyll, trout chow mix	Daily; no amounts
Purity of test substance	> 98%	Chem service
Concentrations measured?	Yes	
Measured is what % of nominal?	70% average	Accept. points
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Concentration of carrier (if any) in test solutions	<200 µL acetone/kg wet sediment	Accept. Points
Concentration 1 Nom (µg/g OC)	0.057	10 per rep; 3 reps -Meas. conc. NR Doc./Accept. points
Concentration 2 Nom (µg/g OC)	0.09	10 per rep; 3 reps
Concentration 3 Nom (µg/g OC)	0.1	10 per rep; 3 reps
Concentration 4 Nom (µg/g OC)	0.2	10 per rep; 3 reps
Concentration 5 Nom (µg/g OC)	0.3	10 per rep; 3 reps
Concentration 6 Nom (µg/g OC)	0.6	10 per rep; 3 reps

<i>H. azteca</i>	Amweg et al. 2005	
Parameter	Value	Comment
Concentration 7 Nom ($\mu\text{g/g OC}$)	0.9	10 per rep; 3 reps
Concentration 8 Nom ($\mu\text{g/g OC}$)	1.6	10 per rep; 3 reps
Control	Solvent and negative	10 per rep; 3 reps
OC-normalized LC ₅₀ (95% confidence interval) $\mu\text{g/g OC}$	AR: 0.63 (0.57-0.69) DPC: 0.57 (0.51-0.69) PC: 0.37 (0.29-0.43)	Method: trimmed Spearman-Karber
Dry weight based LC ₅₀ (95% confidence interval) ng/g DW	AR: 8.58 (7.7-9.7) DPC: 6.6 (5.7-7.7) PC: 23.5 (18.9-28)	Method: trimmed Spearman-Karber
NOEC ($\mu\text{g/g OC}$)	Growth (from Fig. 2A) AR: 0.11 DPC: 0.34 PC: 0.11	Method: one-tailed Bonferroni's t-test p: 0.05 MSD: not reported Doc./Accept. points
LOEC ($\mu\text{g/g OC}$)	Growth AR: 0.23 DPC: 0.60 PC: 0.23	Same as above
MATC (GeoMean NOEC,LOEC) ($\mu\text{g/kg}$)	Growth AR: 0.06 DPC: 0.16 PC: 0.05	Calculated
% of control at NOEC	Growth AR: $75/80*100=94\%$ DPC: $72/78*100=92\%$ PC: $75/92*100=82\%$	Estimated from Fig. 2A with solvent control results
% of control at LOEC	Growth AR: $62/80*100=78\%$ DPC: $40/78*100=51\%$ PC: $50/92*100=54\%$	Same as above

Notes:

Protocol follows EPA 2000 "Methods for measuring the toxicity and bioaccumulation. 2nd. EPA/600/R-99/064.

The above LC₅₀ values and test concentrations have been corrected as directed in the erratum to the original article. Bifenthrin correction factor was 2.86.

Reliability points taken off for:

Documentation : Organism source (4), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Minimum significant difference (2). Total: $100-22=78$

Acceptability: Measured concentration within 20% (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organism not contaminated prior (3), Organisms randomly assigned (1), Organisms properly acclimated (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (5), Overlying water conductivity (1), Overlying water pH (1), Temperature variation (3), Random design (2), Minimum significant difference (1). Total: 100-38=62

Reliability score: Mean (78, 62) = 70

Toxicity Data Summary

Hyaella azteca

Harwood AD, Landrum PF, Lydy MJ (2013a) Bioavailability-based toxicity endpoints of bifenthrin for *Hyaella azteca* and *Chironomus dilutus*. Chemosphere 90:1117-1122.

Relevance

Score: 90

Rating: R

Reliability

Score: 71

Rating: L

*Relevance points taken off for: No standard method cited (10)

<i>H. azteca</i>	Harwood et al. 2013a	
Parameter	Value	Notes
Results published or in signed, dated format?	Yes	
Test method cited	Not reported	Accept. points
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyaellidae	
Genus	<i>Hyaella</i>	
Species	<i>azteca</i>	
Family relevant for North America?	Yes	
Age/size at start of test/growth phase	7-10 days	
Source of organisms	Lab culture	
Have organisms been pre-exposed to contaminants?	No	
Were animals acclimated and disease-free?	Yes	
Were animals randomized?	Not reported	Accept. points
Were test vessels randomized?	Not reported	Accept. points

<i>H. azteca</i>	Harwood et al. 2013a	
Parameter	Value	Notes
Test duration	10 d	
Data for multiple durations?	No	
Effect 1	Survival	
Control response 1	≥ 95%	
Effect 2	Immobilization	Failure to move with normal swimming motions
Control response 2	≤ 10%	
Temperature	23 ± 0.1°C	
Exposure type	Static renewal	100 mL water changed 3x/day
Photoperiod/light intensity	Not reported	Doc. points
Overlying water source	EPA moderately hard water	
pH	6.76 ± 0.13	
Hardness	Not reported	Doc., Accept. points
Alkalinity	Not reported	Doc., Accept. points
Dissolved oxygen	7.34 ± 0.32 mg/L	
Conductivity	367 ± 25 µS/cm	
DOC	Not reported	
Sediment source	3 natural soils/sediments: TON, BAY, LPH	
Organic carbon content	TON: 0.56% BAY: 1.77% LPH: 4.43%	
Particle size distribution (sand, silt, clay)	TON: 14%, 62%, 24% BAY: 52%, 38%, 10% LPH: 46%, 47%, 7%	

<i>H. azteca</i>	Harwood et al. 2013a	
Parameter	Value	Notes
Sediment spike procedure	Sediment spiked in bulk in 1L jars with ≤ 200 μ L acetone stock solutions; homogenized by hand mixing & rolling 1 h	Solvent not evaporated Accept. points
Sediment spike equilibration time	14 d at 4°C in darkness	Accept. points
Sediment-to-solution ratio	50 g sediment (dry wt.) and < 250 mL of water	
Sediment extraction and chemical analysis method	Extracted via Tenax for 6 h or 24 h, Tenax was solvent extracted & analyzed via LSC	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Not applicable	
Interstitial water chemical analysis method	Extracted via SPME fibers equilibrated for 28 d on shaker table. Fibers extracted with hexane for 36 h; analyzed via LSC	
Interstitial water DOC	Not reported	
Feeding	1 mL of yeast-cerophyll-trout chow	
Purity of test chemical	¹⁴ C-labeled: $\geq 98\%$ Unlabeled: technical	
% Measured compared to nominal	Not reported	Accept. points
Were toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	Acetone solvent not evaporated, conc. not reported	Accept. points

<i>H. azteca</i>	Harwood et al. 2013a	
Parameter	Value	Notes
Concentration 1 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep Doc. points - nom & meas concentrations not reported Accept. points – conc. spacing not reported
Concentration 2 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 3 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 4 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 5 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Concentration 6 (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep
Control Type	Negative and solvent	4 reps, 10 per rep
LC ₅₀ (95% confidence interval) Sediment dry weight basis µg/kg DW	TON: 0.0046 (0.0024-0.0060) ^a BAY: 0.0139 (0.0125-0.0155) ^b LPH: 0.0262 (0.0200-0.0402) ^c	Method: log-probit
LC ₅₀ (95% confidence interval) OC-normalized sediment basis µg/g OC	TON: 0.829 (0.434-1.07) ^a BAY: 0.784 (0.704-0.877) ^a LPH: 0.592 (0.454-0.909) ^a	Method: log-probit
LC ₅₀ (95% confidence interval) SPME basis (normalized to PDMS conc.) µg/mL PDMS	TON: 0.155 (0.089-0.202) ^a BAY: 0.149 (0.132-0.164) ^a LPH: 0.163 (0.121-0.233) ^a	Method: log-probit

<i>H. azteca</i>	Harwood et al. 2013a	
Parameter	Value	Notes
LC ₅₀ (95% confidence interval) Calculated interstitial water concentration basis ng/L	TON: 0.563 (0.326-0.731) ^a BAY: 0.540 (0.480-0.596) ^a LPH: 0.594 (0.440-0.847) ^a	Method: log-probit
LC ₅₀ (95% confidence interval) 6 h Tenax extractable concentration basis µg/g OC	TON: 0.175 (0.093- 0.226) ^{ab} BAY: 0.206 (0.183-0.233) ^a LPH: 0.096 (0.072-0.156) ^b	Method: log-probit
LC ₅₀ (95% confidence interval) 24 h Tenax extractable concentration basis µg/g OC	TON: 0.246 (0.147-0.317) ^a BAY: 0.368 (0.327-0.418) ^b LPH: 0.204 (0.159-0.298) ^a	Method: log-probit
EC ₅₀ (95% confidence interval) Sediment dry weight basis µg/kg DW	TON: 0.0035 (0.00138- 0.00512) ^a BAY: 0.0111 (0.0100- 0.0120) ^b LPH: 0.0173 (0.0142- 0.0206) ^c	Method: log-probit
EC ₅₀ (95% confidence interval) OC-normalized sediment basis µg/g OC	TON: 0.631 (0.246-0.915) ^a BAY: 0.625 (0.567-0.678) ^a LPH: 0.391 (0.320-0.465) ^a	Method: log-probit
EC ₅₀ (95% confidence interval) SPME basis (normalized to PDMS conc.) µg/mL PDMS	TON: 0.110 (0.0659- 0.150) ^a BAY: 0.126 (0.109-0.136) ^a LPH: 0.124 (0.099-0.149) ^a	Method: log-probit
EC ₅₀ (95% confidence interval) Calculated interstitial water concentration basis ng/L	TON: 0.400 (0.239-0.543) ^a BAY: 0.458 (0.395-0.500) ^a LPH: 0.448 (0.359-0.540) ^a	Method: log-probit
EC ₅₀ (95% confidence interval) 6 h Tenax extractable concentration basis µg/g OC	TON: 0.134 (0.044- 0.198) ^{ab} BAY: 0.161 (0.145-0.176) ^a LPH: 0.063 (0.049-0.075) ^b	Method: log-probit
EC ₅₀ (95% confidence interval) 24 h Tenax extractable concentration basis	TON: 0.183 (0.092- 0.257) ^{ab} BAY: 0.286 (0.251-0.313) ^a	Method: log-probit

<i>H. azteca</i>	Harwood et al. 2013a	
Parameter	Value	Notes
µg/g OC	LPH: 0.139 (0.114-0.164) ^b	

ASTM = American Society for Testing and Materials, DOC = dissolved organic carbon, EC₅₀ = exposure concentration that causes effect in 50% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, MATC = Maximum acceptable toxicant concentration, meas = measured, MSD = minimum significant difference, NOEC = no observed effect concentration, nom = nominal, reps = replicates, p = p-value for statistical significance, TOC = total organic carbon, USEPA = United States Environmental Protection Agency.

Notes:

Different letters after LC₅₀ or EC₅₀ values within a single concentration type indicates that they are significantly different ($p \leq 0.05$).

Reliability points taken off for:

Documentation (Table 9): Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Photoperiod (3), Hypothesis tests (8). Total: 100-25=75

Acceptability (Table 10): Standard method (5), Measured concentrations within 20% nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Overlying water hardness (1), Overlying water alkalinity (1), Random design (2), Dilution factor (2), Hypothesis tests (3). Total: 100-33=67

Reliability scores: Mean (75, 67)=71

Toxicity Data Summary

Hyaella azteca

Trimble AJ, Belden JB, Mueting SA, Lydy MJ (2010) Determining modifications to bifenthrin toxicity and sediment binding affinity from varying potassium chloride concentrations in overlying water. Chemosphere 80:53-59.

Relevance

Score: 85

Rating: L

Reliability

Score: 77

Rating: R

*Relevance points taken off for: Acceptable bioavailable concentrations not used (nominal instead of measured; 15).

<i>H. azteca</i>	Trimble et al. 2010	
Parameter	Value	Comment
Test method cited	EPA 2000	EPA/600/R-99-064
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyaellidae	
Genus	<i>Hyaella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	7-14 days	
Source of organisms	Lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Not reported	Accept. points
Test vessels randomized?	Yes	
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	>90%	
Temperature	23 ± 1.8 °C	Accept. points
Test type	Static renewal	50% water renewed daily
Photoperiod/light intensity	16 h: 8 h dark	
Overlying water source	Moderately hard reconstituted water	
pH	Measured, not reported	
Hardness mg/L as CaCO ₃	Not reported	Doc./Accept. points
Alkalinity mg/L as CaCO ₃	Not reported	Doc./Accept.

<i>H. azteca</i>	Trimble et al. 2010	
Parameter	Value	Comment
		points
Conductivity	Measured, not reported	Doc. points
Dissolved Oxygen	Measured, not reported	Doc. points
Sediment source	natural	Touch of Nature reference site, Carbondale, IL
Organic carbon	0.983%	±0.10%
Particle size distribution (sand, silt, clay)	14, 72, 14%	
Sediment spike procedure	Solvent & volume not reported; sediment-water slurries were spiked in bulk and mixed for 10 min	Doc./Accept. points
Sediment spike equilibration time	14 d at 4°C	Accept. points
Sediment to Solution ratio	40 g wt.: 250 mL	
Sediment extraction/analysis methods	Solvent extraction and GC/ECD	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Interstitial water DOC	Not applicable	
Feeding	Every other day 1 mL of 6 g/L TetraFin fish food	
Purity of test substance	99.7% technical Radiolabeled: 97.7%, 41.97 $\mu\text{Ci}/\mu\text{mol}$	
Measured is what % of nominal?	80-120%	
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Concentration of carrier (if any) in test solutions	Not reported	Accept. points
Concentration 1 Nom (ng/g)	9.16	Meas. conc. not reported Doc. Points 10 per rep, # reps not reported Accept. points
Concentration 2 Nom (ng/g)	18.3	

<i>H. azteca</i>	Trimble et al. 2010	
Parameter	Value	Comment
Concentration 3 Nom (ng/g)	36.6	
Concentration 4 Nom (ng/g)	73.3	
Concentration 5 Nom (ng/g)	146	
Concentration 6 Nom (ng/g)	293	
Concentration 6 Nom (ng/g)	586	
Control	Solvent and negative	
LC ₅₀	Dry weight basis 0.00194 mg/kg DW OC-normal basis 0.197 mg/kg OC	Method: probit 95% fiducial intervals shown in Fig. 1

Notes:

Reliability points taken off for:

Documentation: Measured concentrations (3), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Sediment spike method (4), Hypothesis tests (8). Total: 100-21=79

Acceptability: Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Temperature variation (3), Overlying water hardness (1), Overlying water alkalinity (1), Adequate replication (2), Hypothesis tests (3). Total: 100-25=75

Reliability score: Mean (79, 75) = 77

Toxicity data summary sheet

Hyalella azteca

Weston DP, You J, Harwood AD, Lydy MJ (2009b) Whole sediment toxicity identification evaluation tools for pyrethroid insecticides: III. Temperature manipulation. Environmental Toxicology and Chemistry 28:173-180.

Relevance
Score: 100
Rating: R

Reliability
Score: 72.5
Rating: L

<i>H. azteca</i>	Weston et al. 2009b	
Parameter	Value	Notes
Results published or in signed, dated format?	Yes	
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family relevant for North America?	Yes	
Age/size at start of test/growth phase	7-10 days	
Source of organisms	Lab culture	
Have organisms been pre-exposed to contaminants?	No	
Were animals acclimated and disease-free?	Yes	
Were animals randomized?	Not reported	Accept. points
Were test vessels randomized?	Not reported	Accept. points
Test duration	10 d	

<i>H. azteca</i>	Weston et al. 2009b	
Parameter	Value	Notes
Data for multiple durations?	No	
Effect 1	Survival	
Control response 1	Negative controls 13°C: 97% 18°C: 95% 23°C: 95% 28°C: 86% Solvent controls Mean across temps: 97%	Mean control responses
Temperature	13, 18, 23, and 28°C	Variation not reported Accept. points
Exposure type	Static renewal	250 mL water changed 2x/day
Photoperiod/light intensity	16 h light: 8 h dark	
Overlying water source	EPA moderately hard water	
pH	Measured, not reported	Within limits of std. method Doc. points
Hardness	Measured, not reported	Within limits of std. method Doc. points
Alkalinity	Measured, not reported	Within limits of std. method Doc. points
Dissolved oxygen	Measured, not reported	Within limits of std. method Doc. points
Conductivity	Not reported	Doc./Accept. points
Sediment source	Natural sediment	American River at Folsom Lake, CA

<i>H. azteca</i>	Weston et al. 2009b	
Parameter	Value	Notes
Organic carbon content	1.87%	
Particle size distribution (sand, silt, clay)	Not reported	Doc. points
Sediment spike procedure	Sediment spiked with acetone stock solutions; homogenized with electric drill mixer	Solvent not evaporated Accept. points
Sediment spike equilibration time	16-22 d at 4°C in darkness	Accept. points
Sediment-to-solution ratio	50-75 mL sediment:250 mL water	
Sediment extraction/analysis method	Solvent sonication extraction & cleanup, analyzed via GC/ECD	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Interstitial water DOC	Not applicable	
Feeding	1 mL of yeast-cerophyll-trout chow	Per beaker, daily.
Purity of test chemical	Technical grade	
% Measured compared to nominal	Sediments at 13-23°C: 61-87% Sediment at 28°C: 46-58%	Accept. points
Were toxicity values calculated based on nominal or measured concentrations?	Nominal	
Concentration of carrier (if any) in test solutions	Acetone solvent not evaporated, 0.2-0.8 uL	Accept. points

<i>H. azteca</i>	Weston et al. 2009b	
Parameter	Value	Notes
	acetone/g wet sediment	
Concentration 1 Nom; Meas (mg/kg)	5-7 concentrations. Dilution factor: 0.6 Actual concentrations not reported	3-4 reps, 10 per rep Doc. points - nom & meas conc. not reported
Concentration 2 Nom; Meas (mg/kg)	5-7 concentrations Dilution factor: 0.6 Actual concentrations not reported	3-4 reps, 10 per rep
Concentration 3 Nom; Meas (mg/kg)	5-7 concentrations Dilution factor: 0.6 Actual concentrations not reported	3-4 reps, 10 per rep
Concentration 4 Nom; Meas (mg/kg)	5-7 concentrations Dilution factor: 0.6 Actual concentrations not reported	3-4 reps, 10 per rep
Concentration 5 Nom; Meas (mg/kg)	5-7 concentrations Dilution factor: 0.6 Actual concentrations not reported	3-4 reps, 10 per rep
Concentration 6 Nom; Meas (mg/kg)	5-7 concentrations Dilution factor: 0.6 Actual concentrations not reported	3-4 reps, 10 per rep
Control Type	Negative and solvent	3-4 reps, 10 per rep
LC ₅₀ (95% confidence interval) µg/g OC	13°C: ~0.25 (estimated from Fig. 1A) 18°C: 0.45 (0.39-0.51) 23°C: 0.99 (0.97-1.02) 28°C: ~1 (estimated from Fig. 1A)	Method: Trimmed Spearman-Kärber (ToxCalc)

ASTM = American Society for Testing and Materials, DOC = dissolved organic carbon, EC₅₀ = exposure concentration that causes effect in 50% of a test population, LC₅₀ = exposure concentration that is lethal to 50% of a test population, LOEC = lowest observed effect concentration, MATC = Maximum acceptable toxicant concentration, meas = measured, MSD = minimum significant difference, NOEC = no observed effect concentration, nom = nominal, reps = replicates, p = p-value for statistical significance, TOC = total organic carbon, USEPA = United States Environmental Protection Agency.

Notes:

Different letters after LC₅₀ or EC₅₀ values within a single concentration type indicates that they are significantly different ($p \leq 0.05$).

Reliability points taken off for:

Documentation (Table 9): Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water conductivity (1), Overlying water pH (1), Dissolved oxygen (2), Particle size distribution (1), Hypothesis tests (8). Total: 100-27=73

Acceptability (Table 10): Measured concentrations within 20% nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Overlying water conductivity (1), Temperature variation (3), Random design (2), Hypothesis tests (3). Total: 100-28=72

Reliability scores: Mean (73, 72)= 72.5

Toxicity Data Summary

Leptocheirus plumulosus

Putt AE (2005b) Bifenthrin – Toxicity to estuarine amphipods (*Leptocheirus plumulosus*) during a 28-day sediment exposure. Study performed by Springborn Smithers Laboratories, Wareham, MA, project ID: 13656.6107; submitted to Pyrethroid Working Group, Washington, DC. EPA MRID 46591502. DPR record number 238257.

Relevance

Score: 85

Rating: L

*Relevance points off for: saltwater species

Reliability

Score: 94.5

Rating: R

<i>L. plumulosus</i>	Putt 2005b	
Parameter	Value	Comment
Test method cited	EPA 2001	
Phylum	Arthropoda	
Class	Malacostraca	
Order	Amphipoda	
Family	Aoridae	
Genus	<i>Leptocheirus</i>	
Species	<i>plumulosus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Neonate/>0.25mm,<0.6mm	
Source of organisms	Springborn Smithers lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	Observed 48 h at 20% salinity and 20°C
Animals randomized?	Yes	
Test vessels randomized?	Not reported	
Test duration	28 day	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	94% negative control; 95% solvent control survival	Pooled control survival 95%
Effect 2	Growth	
Control response 2	2.61 mg in negative control; 2.64 mg solvent control	Dry weight averages
Temperature	24-27°C	
Test type	Static renewal	Renew 400 mL water 3x/week
Photoperiod/light intensity	16 h;8 h dark; 620-950 lux	

<i>L. plumulosus</i>	Putt 2005b	
Parameter	Value	Comment
Overlying water	Filtered seawater	
pH	7.3 to 8.1	
Hardness	Not reported	
Alkalinity	Not reported	
Conductivity	19-21 ‰ salinity	
Dissolved Oxygen	5.0 – 7.2 mg/L	
TOC; DOC	<2 mg/L; Not reported	
Ammonia-N	day 0: 2.8 – 3.2 mg/L day 28: <0.10 – 0.17 mg/L	
Chemical analysis?/Method	Yes	LSC
Sediment formulated?	No	Natural marine; Little harbor beach, Wareham, MA
Organic carbon	4.1%	
Particle size distribution (sand, silt, clay)	68%, 19%, 13%	
pH	6.9	
Percent solids	39.15%	
Sediment spike procedure	Jar rolling technique	9 mL acetone added to 0.05 kg sand, solvent evaporated, added to 2 kg wet sediment (0.7830 kg dry wt)
Sediment spike equilibration time	29 days at 4°C	Rolled once/week for 2 h at room temp
Sediment to Solution ratio	175ml(2cm):725 mL	217 g wet wt. or 84 g dry wt
Interstitial water monitored?	Yes	
Interstitial water extraction method	Centrifuge 30 min at 10,000g	Entire sample
Interstitial water chemical extraction method	None	
Interstitial water chemical analysis method	LSC (Liquid scintillation counting)	2 mL interstitial water + 15 mL cocktail
pH	6.6-7.1	Over 28 days
DOC	day 0: 11.7-25.8 day 28: 11.5-60.7	
Ammonia-N	day 0: 15.2 -17.1 mg/L day 28: 1.6 – 3.4 mg/L	No adverse effects
Feeding	Flakes fish food suspension 3x/week following water	0-13days 2mL of 10mg/mL; 14-

<i>L. plumulosus</i>	Putt 2005b	
Parameter	Value	Comment
	renewal	27days 4mL of 10mg/mL
Purity of test substance	96.4% ¹⁴ C-bifenthrin; specific activity 24.4 mCi/mmol using HPLC-radiochemical detection (RAM)	Technical (93%) used for range finding
Concentrations measured?	Yes	
Measured is what % of nominal?	Sediment: 89%-120%	94%-130% in stock solutions
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Chemical method documented?	Yes	LSC
Concentration of carrier (if any) in test solutions	9 mL acetone added to 0.05 kg sand, evaporate, add to 2 kg wet sediment (0.7830 kg dry wt)	
Concentration 1	Sediment (Nom; Meas): 5.6; 5.4 (µg/kg DW Interstitial water (Meas): <0.20 µg/L Overlying water:<0.076 µg/L	5 reps, 20 per rep
Concentration 2	Sediment (Nom; Meas): 17; 20 µg/kg DW Interstitial water (Meas): 0.31 µg/L Overlying water:<0.077 µg/L	5 reps, 20 per rep
Concentration 3	Sediment (Nom; Meas): 50; 50 µg/kg DW Interstitial water (Meas): 0.50 µg/L Overlying water:<0.077 µg/L	5 reps, 20 per rep
Concentration 4	Sediment (Nom; Meas): 150; 130 µg/kg DW Interstitial water (Meas): 1.4 µg/L Overlying water: 0.093 µg/L	5 reps, 20 per rep
Concentration 5	Sediment (Nom; Meas): 450; 440 µg/kg DW Interstitial water (Meas): 4.8 µg/L Overlying water: 0.235 µg/L	5 reps, 20 per rep
Concentration 6	Sediment (Nom; Meas):	5 reps, 20 per rep

<i>L. plumulosus</i>	Putt 2005b	
Parameter	Value	Comment
	1350; 1500 µg/kg DW Interstitial water (Meas): 14 µg/L Overlying water: 0.655 µg/L	
Control	Solvent and negative	5 reps, 20 per rep
LC ₅₀ (95% confidence interval)	Dry weight basis 240 (220 – 260) µg/kg DW	Method: Inhibition concentration method (TOXSTAT 3.5)
EC ₅₀ (95% confidence interval)	Dry weight basis 150 (130 – 180) µg/kg DW	Method: Inhibition concentration method (TOXSTAT 3.5)
NOEC	Dry weight basis Survival: 50 µg/kg DW Growth: 50 µg/kg DW	Method: Bonferroni's Test (growth), Wilcoxon's Rank-Sum Test (survival) p: 0.05 MSD: Not reported
LOEC	Dry weight basis Survival: 130 µg/kg DW Growth: 130 µg/kg DW	Same as above
MATC (GeoMean NOEC,LOEC) (µg/kg)	Dry weight basis Survival: 81 µg/kg DW Growth: 81 µg/kg DW	
% of control at NOEC	Survival: 96%/95%=101% Growth: 2.35mg/2.63mg=89%	Pooled controls
% of control at LOEC	Survival: 75%/95%=79% Growth: 1.41mg/2.63mg=54%	

Notes:

Radiolabeled bifenthrin used in toxicity testing.

Reliability points taken off for:

Documentation: Overlying water hardness (1), Overlying water alkalinity (1), Overlying water conductivity, Minimum significant difference (2). Total: 100-5=95

Acceptability: Temperature variation (3), Random design (2), Minimum significant difference (1). Total: 100-6=94

Reliability score: Mean (95, 94) = 94.5