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Evaluation of Numerical Sediment Quality Targets for the St. Louis River Area of Concern

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Abstract. Numerical sediment quality targets (SQTs) for the protection of sediment-dwelling organisms have been established for the St. Louis River Area of Concern (AOC), 1 of 42 current AOCs in the Great Lakes basin. The two types of SQTs were established primarily from consensus-based sediment quality guidelines. Level I SQTs are intended to identify contaminant concentrations below which harmful effects on sediment-dwelling organisms are unlikely to be observed. Level II SQTs are intended to identify contaminant concentrations above which harmful effects on sediment-dwelling organisms are likely to be observed. The predictive ability of the numerical SQTs was evaluated using the matching sediment chemistry and toxicity data set for the St. Louis River AOC. This evaluation involved determination of the incidence of toxicity to amphipods (*Hyalella azteca*) and midges (*Chironomus tentans*) within five ranges of Level II SQT quotients (*i.e.*, mean probable effect concentration quotients [PEC-Qs]). The incidence of toxicity was determined based on the results of 10-day toxicity tests with amphipods (endpoints: survival and growth) and 10-day toxicity tests with midges (endpoints: survival and growth). For both toxicity tests, the incidence of toxicity increased as the mean PEC-Q ranges increased. The incidence of toxicity observed in these tests was also compared to that for other geographic areas in the Great Lakes region and in North America for 10- to 14-day amphipod (*H. azteca*) and 10- to 14-day midge (*C. tentans* or *C. riparius*) toxicity tests. In general, the predictive ability of the mean PEC-Qs was similar across geographic areas. The results of these predictive ability evaluations indicate that collectively the mean PEC-Qs provide a reliable basis for classifying sediments as toxic or not toxic in the St. Louis River AOC, in the larger geographic areas of the Great Lakes, and elsewhere in North America.

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The St. Louis River constitutes the second largest tributary to Lake Superior. The headwaters begin in northeastern Minnesota, and the lower estuary, which covers an area of approximately 12,000 acres, bisects the border between Duluth, MN, and Superior, WI (MPCA and WDNR 1992). The lower estuary culminates in the Duluth–Superior Harbor, which is one of the largest inland seaports in the world and the most heavily used port in the Great Lakes basin.

The middle and lower portions of the estuary support a variety of industrial, commercial, residential, and recreational activities. In addition, these areas provide essential habitats for aquatic organisms and aquatic-dependent wildlife species. However, aquatic habitats in some of these areas have been adversely affected by economic development of the St. Louis River over the past 130 years. Accordingly, the International Joint Commission (IJC) designated the lower St. Louis River as one of 43 Areas of Concern (AOCs) in the Great Lakes basin in 1987 (IJC 1989); one site has since been delisted as an AOC.

The IJC, through a formal protocol agreement between Canada and the United States, is charged with reviewing the remedial action plans (RAPs) for each AOC. The RAPs are being prepared in a staged approach to evaluate impaired uses, develop and implement a plan for restoring beneficial uses, and evaluate the success of any remedial measures that are conducted. The RAP process must embody a comprehensive ecosystem approach and include substantial citizen participation (MPCA and WDNR 1992). The ecosystem approach is a geographically comprehensive approach to environmental planning and management that recognizes the interrelated nature of environmental media and that humans are key components of ecological systems (CCME 1996). This approach places equal emphasis on concerns related to the environment, the economy, and the community.

Management of contaminated sediments has been identified as a high priority in the RAP process because they are known to contribute to several use impairments in the St. Louis River AOC, including the issuance of fish advisories, restrictions on dredging, and habitat impairments to bottom-feeding organisms. An ecosystem-based management approach has been used to develop a framework for assessing and managing sediment quality conditions in the St. Louis River AOC (Crane *et al.* 2000). One of the 16 ecosystem objectives that has been established for the St. Louis River AOC is to maintain a healthy and well-balanced aquatic ecosystem where native species can live and reproduce naturally and are not restricted from thriving due to substrate degradation (MPCA and WDNR 1992). This objective implies that sediment quality conditions should be maintained such that the benthic community, including epibenthic and infaunal species, is protected and, where necessary, restored (Crane *et al.* 2000).

Evaluation of the extent to which this objective is being met for the benthic community necessitates the identification of ecosystem health indicators and associated metrics. Accordingly, Crane *et al.* (2000) identified a suite of indicators to support assessments of contaminated sediments, including sediment chemistry, sediment toxicity, benthic invertebrate community structure, sediment quality triad, physical characteristics, tissue chemistry, biomarkers in fish, water chemistry, porewater toxicity, and water column and elutriate toxicity. A set of candidate metrics that identifies key measurable characteristics of the associated ecosystem health indicators has also been recommended for the St. Louis River AOC (Crane *et al.* 2000). Incorporation of these indicators and metrics in sediment assessment initiatives will result in the development of a consistent database of information that can be used to assess sediment quality conditions, identify sediment management priorities, and determine if sediment-related use impairments have been alleviated in the St. Louis River AOC.

Sediment chemistry is an important indicator of sediment quality conditions in the St. Louis River AOC, for which the concentrations of chemicals of potential concern (COPCs) represent the associated metric. The COPCs in the St. Louis River AOC include: polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides (*e.g.*, DDT metabolites, toxaphene), dioxins and furans, and trace metals (*e.g.*, copper, lead, mercury, nickel, zinc; Crane *et al.* 2000). Although measurements of chemical concentrations provide information that can be used to directly assess trends in sediment quality conditions, numerical targets are also needed to provide more direct linkages to the ecosystem objectives. Such sediment quality targets (SQTs) define the ranges of chemical concentrations in sediments necessary to protect the benthic community.

The Minnesota Pollution Control Agency (MPCA) has not previously developed or adopted SQTs for the state of Minnesota. Instead, the MPCA has identified contaminated areas in the St. Louis River AOC by comparing the sediment chemistry data with informal (*i.e.*, nonenforceable) sediment quality guidelines (SQGs) that have been promulgated by other jurisdictions (Persaud *et al.* 1993; Smith *et al.* 1996). To further the progress on the St. Louis River RAP (MPCA and WDNR 1992), the MPCA established biological effects–based SQTs for COPCs in the St. Louis River AOC (Crane *et al.* 2000). However, information of the predictive ability of the SQTs was needed to support their implementation in the RAP process.

This paper was prepared to report the results of an evaluation of predictive ability of the SQTs using the matching sediment chemistry and toxicity data set available for the St. Louis River AOC. More specifically, we evaluated the extent to which sediments containing a mixture of contaminants can be correctly classified as toxic or nontoxic using sediment chemistry data and the SQTs (*i.e.*, through the use of mean probable effect concentration quotients [PEC-Qs]; MacDonald *et al.* 2000; Ingersoll *et al.* 2001). In addition, the results of a comparison of the incidences of toxicity to amphipods and midges in St. Louis River AOC sediment with that in sediments from other sites in the Great Lakes region and elsewhere in North America is presented.

Materials and Methods

Identification of Narrative Objectives

Participants in the development of the St. Louis River RAP identified five water uses that need to be protected and/or restored in the St. Louis River AOC. These designated water uses include fish and aquatic life, wildlife, human health, recreation and aesthetics, and shipping and navigation. It is generally agreed that these water uses should be protected, but it may not be possible to provide a uniform level of use protection throughout the St. Louis River AOC. In particular, physical disturbances resulting from seiches, wind-induced waves, ice scour, navigational dredging, prop wash, and other factors can reduce the potential for maintaining productive benthic communities in certain locations. In addition, ongoing nonpoint discharges of COPCs (*e.g.*, upland industrial and commercial activities, spills) have the potential to reduce the long-term effectiveness of any sediment clean-up activities.

In recognition of the challenges that are associated with sediment management in the St. Louis River AOC (Crane *et al.* 2000), two types of narrative SQTs were established by the MPCA. The Level I SQTs are intended to identify contaminant concentrations below which harmful effects on sediment-dwelling organisms are unlikely to be observed. In contrast, the Level II SQTs are intended to identify contaminant concentrations above which harmful effects on sediment-dwelling organisms are likely to be observed. The narrative objectives for both levels of SQTs do not address the potential for bioaccumulation, nor the associated effects on those species that consume aquatic organisms (*i.e.*, wildlife and humans).

These narrative objectives recognize that water-based economic activities (such as shipping, operation of small craft marinas, waterfront development, and tourism-related activities) are essential to the vitality of the communities that border the St. Louis River AOC, including Duluth, MN, and Superior, WI. Though these activities contribute significantly to the local economy, they also have the potential to degrade water quality conditions (*i.e.*, through spills or stormwater runoff) and/or reduce the stability of sediments (*i.e.*, through navigational dredging or prop wash). Therefore, the potential for maintaining an unaltered benthic invertebrate community is likely reduced in certain areas, even in the absence of chemical contamination in the sediments.

Strategy for Establishing Numerical Sediment Quality Targets

Both theoretical and empirical approaches have been used to derive numerical SQGs for freshwater ecosystems. Seven distinct approaches were evaluated to support the selection of procedures for deriving numerical SQTs for the St. Louis River AOC, including the screening level concentration, effects range and effects level, apparent effects threshold, equilibrium partitioning, logistic regression modeling, consensus-based, and tissue residue approaches (Crane *et al.* 2000). The logistic regression modeling approach has been used successfully with data sets of matching sediment chemistry and toxicity data from marine ecosystems (Field *et al.* 1999, 2002). However, the results of preliminary analyses conducted using the entire North America–wide database of matching sediment chemistry and toxicity data (*i.e.*, the SEDTOX database; Ingersoll *et al.* 2001) for freshwater ecosystems revealed that insufficient data were available to generate reliable logistic regression models for any of the toxicity test endpoints that were represented in the database (*e.g.*, *Hyalella azteca* growth or survival in 10- to 14-day toxicity tests). As such, it was apparent that it would not be possible to develop logistic regression models using a portion of the database only (*i.e.*, the data from the St. Louis River AOC).

After a review of the other approaches, the following strategy was used to recommend numerical SQTs for the protection of sediment-dwelling organisms in the St. Louis River AOC (Crane *et al.* 2000). First, the consensus-based SQGs that were derived by MacDonald *et al.* (2000) were adopted for all of the substances for which they were available. The consensus-based SQGs consisted of threshold effect concentrations (TECs), which were consistent with the narrative intent of the Level I SQTs, and probable effect concentrations (PECs), which were consistent with the narrative intent of the Level II SQTs. Second, the most reliable of the other effects-based freshwater SQGs that have been published (CCME 1999; NYSDEC 1999) were adopted for those chemicals for which consensus-based SQGs were not available. In this context, the term *reliable* is defined as the ability of the SQGs to correctly classify sediments as toxic or nontoxic based on the data used to derive the guidelines (Long and MacDonald 1998).

This strategy was developed to provide a consistent basis for establishing reliable and regionally applicable SQTs for PCOCs in the St. Louis River AOC. The consensus-based SQGs, that were derived by MacDonald *et al.* (2000) and evaluated by Ingersoll *et al.* (2001), appeared to be the most relevant for establishing numerical SQTs for the St. Louis River AOC for several reasons. According to Swartz (1999) and MacDonald *et al.* (2000), consensus-based SQGs provide a unifying synthesis of the existing SQGs, reflect causal rather than correlative effects, and account for the effects of contaminant mixtures. Therefore, the consensus-based SQGs (adopted as SQTs) are likely to provide useful tools for assessing sediment quality conditions in the St. Louis River AOC.

The Level II SQTs that corresponded to the reliable PECs (MacDonald *et al.* 2000; Table 1), were then evaluated to determine their relevance to the St. Louis River AOC. This evaluation involved two principal steps, including development of a database of matching sediment chemistry and toxicity data and assessment of the incidence of toxicity within five ranges of mean PEC-Qs. The evaluation was based on dry weight concentrations because previous studies have demonstrated that normalization of SQGs for PAHs or PCBs to TOC (Barrick *et al.* 1988; Long *et al.* 1995; US EPA 1996) or normalization of simultaneously extractable metal (SEM) concentrations to acid volatile sulfide (AVS) concentrations (Long *et al.* 1998) did not improve the predictions of toxicity in field collected sediments.

Development of Project Database

An extensive search of the scientific literature, as well as contact with stakeholders and various experts in the sediment quality assessment field, was conducted to acquire matching sediment chemistry and toxicity data for the St. Louis River AOC. All of the data sets and associated documents that were retrieved during the course of this study were critically evaluated to determine their scientific and technical validity. To support this critical evaluation, a comprehensive set of screening criteria were developed in cooperation with the Science Advisory Group on Sediment Quality Assessment (Crane *et al.* 2000). These screening criteria provided a means of consistently evaluating the methods that were used in each study, including the procedures that were used to collect, handle, and transport sediment samples, the protocols that were applied to conduct sediment toxicity tests, the methods that were used to determine the concentrations of contaminants in sediments, and the statistical tests that were applied to the study results. In many cases, additional contact with investigators and professional judgment was needed to determine if the screening criteria had been satisfied. The data sets that met the screening criteria were incorporated into spreadsheets in Microsoft Excel™ format, printed, and verified against the original data sources. Overall, application of these quality assurance procedures was intended to ensure that only high-quality and fully verified data were incorporated into the project database.

All of the verified, matching sediment chemistry and toxicity data assembled from the St. Louis River AOC were incorporated into a Microsoft Access™ database. These data were captured on a per sample basis. Each record in the resulting database included the reference citation, a brief description of the study area (*i.e.*, by water body and reach), a description of the sampling locations (including georeferenced data, if available), information on the toxicity tests that were conducted (including species tested, metric measured, and test duration), type of material tested (*i.e.*, whole sediment, porewater, or elutriate), and the chemical concentrations (expressed on a dry weight basis). Other supporting data, such as total organic carbon (TOC) concentrations, SEM concentrations, AVS concentrations, particle size distribution, and water temperature were also included in the individual data records, as available. The St. Louis River database was subsequently incorporated into a larger North America–wide database of matching sediment chemistry and toxicity data (i.e., the SEDTOX database), to which the same screening criteria had been applied.

Data on the concentrations of both total and SEM metals were captured in the project database and used in the predictive ability evaluation. As a conservative estimate, SEM concentrations were assumed to be equivalent to total metal concentrations in the database for the sediment samples for which only SEM concentrations were available for cadmium, copper, nickel, lead, and zinc. This assumption was consistent with procedures used in the larger SEDTOX database, and it was based on an evaluation of corresponding SEM and total metal data sets.

Evaluation of Mixtures of Sediment-Associated Contaminants

Sediments in the St. Louis River AOC are known to contain complex mixtures of contaminants (Crane *et al.* 2000). Thus the predictive ability of the Level II SQTs (through the use of mean PEC-Qs) is likely to increase when the SQTs for the various COPCs are used together to classify sediments from the St. Louis River AOC. More specifically, an evaluation was conducted to determine the incidence of toxicity within the following ranges of mean PEC-Qs: ≤ 0.1, > 0.1 to ≤ 0.5, > 0.5 to ≤ 1.0, > 1.0 to ≤ 5.0, and > 5.0. These ranges were analogous to the mean PEC-Q ranges used by Ingersoll *et al.* (2001) to evaluate the predictive ability of freshwater SQGs. In this evaluation, mean PEC-Qs were calculated using the methods that were recommended by Ingersoll *et al.* (2001) and outlined in Crane *et al.* (2000). In brief, the mean PEC-Qs were calculated as follows:

$$\text{PEC-Q} = \frac{\text{chemical concentration (in dry wt.)}}{\text{corresponding PEC value}}$$

$$\text{mean PEC-Q} = \frac{(\text{mean PEC-Q}_{\text{metals}} + \text{PEC-Q}_{\text{Total PAHs}} + \text{PEC-Q}_{\text{Total PCBs}})}{n}$$

Table 1. Level I and Level II sediment quality targets for the protection of sediment-dwelling organisms in the St. Louis River AOC

Chemical	Aquatic Life Level I SQT	Aquatic Life Level II SQT	Source[a]
Metals (in mg/kg DW)			
Arsenic[c]	9.8	33	MacDonald *et al.* (2000)
Cadmium[bc]	0.99	5.0	MacDonald *et al.* (2000)
Chromium[c]	43	110	MacDonald *et al.* (2000)
Copper[bc]	32	150	MacDonald *et al.* (2000)
Lead[bc]	36	130	MacDonald *et al.* (2000)
Mercury	0.18	1.1	MacDonald *et al.* (2000)
Nickel[c]	23	49	MacDonald *et al.* (2000)
Zinc[bc]	120	460	MacDonald *et al.* (2000)
PAHs (in μg/kg DW)			
2-Methylnaphthalene	20	200	CCME (1999)
Acenaphthene	6.7	89	CCME (1999)
Acenaphthylene	5.9	130	CCME (1999)
Anthracene[b]	57	850	MacDonald *et al.* (2000)
Fluorene	77	540	MacDonald *et al.* (2000)
Naphthalene[bc]	180	560	MacDonald *et al.* (2000)
Phenanthrene[bc]	200	1,200	MacDonald *et al.* (2000)
Benz(a)anthracene[bc]	110	1,100	MacDonald *et al.* (2000)
Benzo(a)pyrene[bc]	150	1,500	MacDonald *et al.* (2000)
Chrysene[bc]	170	1,300	MacDonald *et al.* (2000)
Dibenz(a,h)anthracene	33	140	MacDonald *et al.* (2000); CCME (1999)
Fluoranthene[b]	420	2,200	MacDonald *et al.* (2000)
Pyrene[bc]	200	1,500	MacDonald *et al.* (2000)
Total PAHs[bc]	1,600	23,000	MacDonald *et al.* (2000)
PCBs (in μg/kg DW)			
Total PCBs[bc]	60	680	MacDonald *et al.* (2000)
Pesticides (in μg/kg DW)			
Chlordane[b]	3.2	18	MacDonald *et al.* (2000)
Dieldrin[b]	1.9	62	MacDonald *et al.* (2000)
Sum DDD[b]	4.9	28	MacDonald *et al.* (2000)
Sum DDE[bc]	3.2	31	MacDonald *et al.* (2000)
Sum DDT[b]	4.2	63	MacDonald *et al.* (2000)
Total DDT[b]	5.3	570	MacDonald *et al.* (2000)
Endrin	2.2	210	MacDonald *et al.* (2000)
Heptachlor epoxide[b]	2.5	16	MacDonald *et al.* (2000)
Lindane (gamma-BHC)	2.4	5	MacDonald *et al.* (2000)
Toxaphene	0.1	32	NYSDEC (1999)[d]
Mean PEC-Q	0.1	0.6	Ingersoll *et al.* (2001)

DW = dry weight; SQT = sediment quality target; PEC-Q = probable effect concentration quotient.
[a] Some SQT values were rounded to two significant figures from the original source.
[b] Reliable consensus-based TEC values that were adopted as Level I SQTs (*i.e.*, predictive ability ≥ 75% and ≥ 20 samples below the TEC [MacDonald *et al.* 2000]).
[c] Reliable consensus-based PEC values that were adopted as Level II SQTs (*i.e.*, predictive ability ≥ 75% and ≥ 20 samples predicted to be toxic [MacDonald *et al.* 2000]).
[d] Originally based on μg/g OC; assumed TOC = 1%.

where n = number of classes of chemicals for which sediment chemistry data were available (*i.e.*, 1 to 3).

Only the metals for which reliable SQTs were available were used to calculate mean PEC-Qs.

Results and Discussion

Identification of Level I and Level II SQTs

The strategy for identifying effects-based SQTs for the protection of sediment-dwelling organisms yielded Level I and Level II SQTs for 8 trace metals, 13 individual PAHs, total PAHs, PCBs, and 10 organochlorine pesticides (Table 1). Because most of the Level I and Level II SQTs were adopted from the consensus-based TECs and PECs, respectively, the published information on the reliability of the TECs and PECs was used in the evaluation of individual SQTs (as further discussed in Crane *et al.* 2000). Based on reliability analyses that were carried out on a nationwide database (which did not include the St. Louis River data set), the individual consensus-based TECs were considered to provide a reliable basis for assessing the quality of freshwater sediments if more than 75% of the sediment samples were correctly predicted to be nontoxic (Mac-

Donald *et al.* 2000). In addition, the individual consensus-based PECs were considered to be reliable if more than 75% of the sediment samples were correctly predicted to be toxic (MacDonald *et al.* 2000). Consequently, the target levels of both false positives (*i.e.*, samples incorrectly classified as toxic) and false negatives (*i.e.*, samples incorrectly classified as not toxic) were 25% using the TEC and PEC values (MacDonald *et al.* 2000). The consensus-based SQGs were considered to be reliable only if a minimum of 20 samples were included in the predictive ability evaluation (CCME 1995).

Population of the Matching Sediment Chemistry and Toxicity Database

The verified studies contained in the St. Louis River AOC sediment toxicity database provided eight data sets (Table 2) with which to evaluate the predictive ability of the Level II SQTs (through the use of mean PEC-Qs; Crane *et al.* 2000). These studies provided sediment toxicity results for 168 sediment samples, with the greatest amount of data available for the 10-day amphipod (*H. azteca*) and midge (*Chironomus tentans*) toxicity tests. Data on the concentrations of metals and PAHs were available for most of these samples. The SEM entries in the database were primarily from a Regional Environmental Monitoring and Assessment Program (R-EMAP) study conducted in the St. Louis River AOC (Breneman *et al.* 2000). There was little matching sediment chemistry and toxicity data available for total PCBs, and no such data for pesticides. The database included broad geographic coverage of the St. Louis River AOC (Figure 1).

Sediment samples in the St. Louis River AOC database were designated as toxic for the following individual metrics if the response observed in test sediments was significantly different ($\alpha = 0.05$) from the response observed in the reference or control sediments: amphipod (*H. azteca*) survival or growth (10-day exposure), midge (*C. tentans*) survival or growth (10-day exposure), oligochaete (*Lumbriculus variegatus*) survival (10-day exposure); and daphnid (*Ceriodaphnia dubia* or *Daphnia magna*) survival (48-h exposure). The results of control samples were not included in the database. Overall toxicity was assigned to a sediment sample if toxicity was observed for one or more of the above metrics. Microtox® was not included as an indicator of toxicity in this assessment because it failed to improve our ability to discriminate among sediment samples in the St. Louis River AOC relative to the other toxicity metrics (Crane *et al.* 2000).

Predictive Ability of Mean PEC-Q Ranges for the St. Louis River AOC

The predictive ability of the mean PEC-Qs was evaluated using the incidence of toxicity information contained in the matching sediment chemistry and toxicity database for the St. Louis River AOC (Table 3). For each type of toxicity test category given in Table 3, the greatest number of matching sediment chemistry and toxicity samples were available for the two lowest mean PEC-Q ranges (*i.e.*, ≤ 0.1 and > 0.1 to ≤ 0.5). For the next three higher mean PEC-Q ranges, the minimum data requirements (*i.e.*, 20 samples per category) were not met (Table 3). As such, comparisons of the predictive ability of different toxicity tests at higher mean PEC-Q ranges should be made with caution. Nevertheless, overall trends in the incidence of sediment toxicity can be distinguished with the available data set (Table 3).

The results of the predictive ability evaluation indicate that the incidence of acute toxicity to amphipods (*H. azteca*) and midges (*C. tentans*), resulting from 10-day toxicity tests, tends to be low (*i.e.*, 6.8% and 6.5%, respectively) when the concentrations of sediment-associated contaminants are low (*i.e.*, as indicated by mean PEC quotients of ≤ 0.1; Table 3). A low incidence of toxicity (*i.e.*, 10%) was also observed for all tests combined (excluding Microtox) for sediments with mean PEC-Q ranges ≤ 0.1 (Table 3). Importantly, the incidence of sediment toxicity in the St. Louis River AOC sediments generally increased with increasing contaminant concentrations. In particular, a high incidence of toxicity (*i.e.*, $\geq 75\%$) was observed at mean PEC-Qs > 5.0 for 10-day amphipod and midge toxicity tests (Table 3). Although these results are useful for assessing the applicability of the Level II SQTs in the St. Louis River AOC, this evaluation was limited by the absence of data at higher contaminant concentrations and from longer-term toxicity tests. This represents an important data gap because acute toxicity data do not provide an adequate basis for evaluating toxicity of sediment-associated contaminants in longer-term exposures (Ingersoll *et al.* 2001).

The lack of toxicity at some sites as the level of contamination increased may also be due to nonbioavailable contaminants. Assuming that AVS binds a molar equivalent of SEM metals (Di Toro *et al.* 1990), SEM would not be available for uptake by benthic biota at sites with a higher concentration of AVS. Furthermore, most benthic organisms, including those used in toxicity tests, survive in sediments that have a thin oxidized surface layer and then an anoxic layer. The anoxic layer can have higher AVS concentrations, which would reduce the metal activity to which these organisms are exposed (Di Toro *et al.* 1992). When SEM exceeds AVS by a factor of five (on a molar basis), a higher incidence of toxicity (80% to 90%) has been observed in freshwater and saltwater sediment amphipod tests (US EPA 1997). Thus, $[SEM] - [AVS] \geq 5$ is a better predictor of sediment toxicity to amphipods.

Given the historical usage of coal-derived products in the Duluth–Superior area, PAHs are a widespread contaminant of concern in the lower St. Louis River estuary. PAHs that are associated with soot-type particles are less bioavailable than PAHs associated with sand, silt, and clay particles. Studies on Milwaukee Harbor, WI, sediments revealed that PAHs associated with coal-derived particles, aged over several decades in the field, appeared to be far from reaching an equilibrium sorption state due to the extremely slow diffusivities through the polymer-like coal matrix (Ghosh *et al.* 2001). Humic substances can also render PAHs less bioavailable to aquatic biota, particularly in the porewater and water column. Perminova *et al.* (2001) found that the aromatics enriched humic materials are the most efficient detoxifying agents in relation to PAHs.

Geographic Comparisons of Mean PEC-Q Ranges for Amphipods and Midges

Ingersoll *et al.* (2001) assembled matching sediment chemistry and toxicity data from a variety of geographic locations in

Table 2. Summary of verified sediment toxicity data for the St. Louis River AOC

Reference	Species	Medium	Duration	Endpoint
Ankley *et al.* (1994)	*C. tentans*	Bulk sediment	10 days	Growth (weight mg)
Ankley *et al.* (1994)	*C. tentans*	Bulk sediment	10 days	Percent survival
Ankley *et al.* (1994)	*C. tentans*	Bulk sediment	10 days	Percent survival/normal and UV
Ankley *et al.* (1994)	*C. tentans*	Bulk sediment	10 days	Percent weight using UV light
Ankley *et al.* (1994)	*H. azteca*	Bulk sediment	10 days	Growth (weight mg)
Ankley *et al.* (1994)	*H. azteca*	Bulk sediment	10 days	Percent survival
Ankley *et al.* (1994)	*H. azteca*	Bulk sediment	10 days	Percent survival/normal and UV
Ankley *et al.* (1994)	*H. azteca*	Bulk sediment	10 days	Percent weight using UV light
Ankley *et al.* (1994)	*L. variegatus*	Bulk sediment	10 days	Growth (weight mg)
Ankley *et al.* (1994)	*L. variegatus*	Bulk sediment	10 days	Percent survival
Ankley *et al.* (1994)	*L. variegatus*	Bulk sediment	10 days	Percent survival/normal and UV
Ankley *et al.* (1994)	*L. variegatus*	Bulk sediment	10 days	Percent weight using UV light
Crane *et al.* (1997)	*C. tentans*	Bulk sediment	10 days	Percent survival
Crane *et al.* (1997)	*H. azteca*	Bulk sediment	10 days	Percent survival
IT Corp. (1997)	*C. tentans*	Bulk sediment	10 days	Growth (weight mg)
IT Corp. (1997)	*C. tentans*	Bulk sediment	10 days	Percent survival
IT Corp. (1997)	*H. azteca*	Bulk sediment	10 days	Growth (weight mg)
IT Corp. (1997)	*H. azteca*	Bulk sediment	10 days	Percent survival
MPCA (1996)	*C. tentans*	Bulk sediment	10 days	Percent survival
MPCA (1997a)	*C. tentans*	Bulk sediment	10 days	Percent survival
MPCA (1997a)	*H. azteca*	Bulk sediment	10 days	Percent survival
MPCA (1997a)	*P. phosphoreum*[a]	Bulk sediment	30 min	Bioluminescence (EC_{50} expressed as % DW sediment)
MPCA (1997a)	*P. phosphoreum*	Porewater	30 min	% Reduction in bioluminescence (relative to control)
MPCA (1997b)	*C. tentans*	Bulk sediment	10 days	Growth (weight mg)
MPCA (1997b)	*C. tentans*	Bulk sediment	10 days	Percent survival
MPCA (1997b)	*H. azteca*	Bulk sediment	10 days	Percent survival
MPCA (1997b)	*P. phosphoreum*	Bulk sediment	15 min	Bioluminescence (EC_{50} expressed as % DW sediment)
MPCA (1997b)	*P. phosphoreum*	Porewater	15 min	% Reduction in bioluminescence (relative to control)
Smith *et al.* (1992)	*C. tentans*	Bulk sediment	10 days	Percent survival
Smith *et al.* (1992)	*D. magna*	Bulk sediment	48 h	Percent survival
Wenck Associates (1995)	*C. dubia*	Bulk sediment	48 h	Percent survival
Wenck Associates (1995)	*C. tentans*	Bulk sediment	10 days	Growth (weight mg)
Wenck Associates (1995)	*C. tentans*	Bulk sediment	10 days	Percent survival
Wenck Associates (1995)	*H. azteca*	Bulk sediment	10 days	Percent survival

UV = ultraviolet; DW = dry weight.
[a] *Photobacterium phosphoreum*.

North America to evaluate the predictive ability of the consensus-based SQGs (*i.e.*, using the SEDTOX database). These data were used to compare the incidence of toxicity in the St. Louis River AOC data set to that in the Great Lakes and North American data sets for 10- to 14-day amphipod (*H. azteca*) and midge (*C. tentans* or *C. riparius*) toxicity tests. This comparison was done to determine if the predictive ability of the Level II SQTs (*i.e.*, through the mean PEC-Qs) differed among geographic areas.

The amphipod and midge data set for the St. Louis River AOC comprised a substantial portion of the total Great Lakes data set (49% and 32%, respectively) and approximately 24% of the total North American data set for both organisms (Crane *et al.* 2000). The contribution of the St. Louis River AOC data set was even more pronounced at mean PEC-Qs ≤ 0.1, accounting for 88% of the Great Lakes amphipod data set and 47% of the Great Lakes midge data set (Crane *et al.* 2000). Thus, in making comparisons of the incidence of toxicity between the St. Louis River AOC, Great Lakes, and North American data sets, it is also important to exclude the St. Louis River AOC data from the Great Lakes and North American data sets to examine independent data sets.

The Great Lakes amphipod and midge data comprised 47% and 75%, respectively, of the North American data set (Crane *et al.* 2000). This preponderance of Great Lakes sediment toxicity data in the North American data set may be attributed to the following factors: the IJC's listing of 43 AOCs around the Great Lakes area in 1987 (IJC 1989) and subsequent assessments of contaminated sediments in these AOCs, the successful implementation of sediment assessment recommendations from the U.S. Environmental Protection Agency's (EPA) Assessment and Remediation of Contaminated Sediments program (US EPA 1994), and an increase in funding of sediment-related projects in the Great Lakes area by the U.S. EPA's Great Lakes National Program Office during the 1990s.

The predictive ability of the mean PEC-Qs for 10- to 14-day amphipod and midge tests are given in Tables 4 and 5, respectively. The incidence of toxicity for both tests was calculated

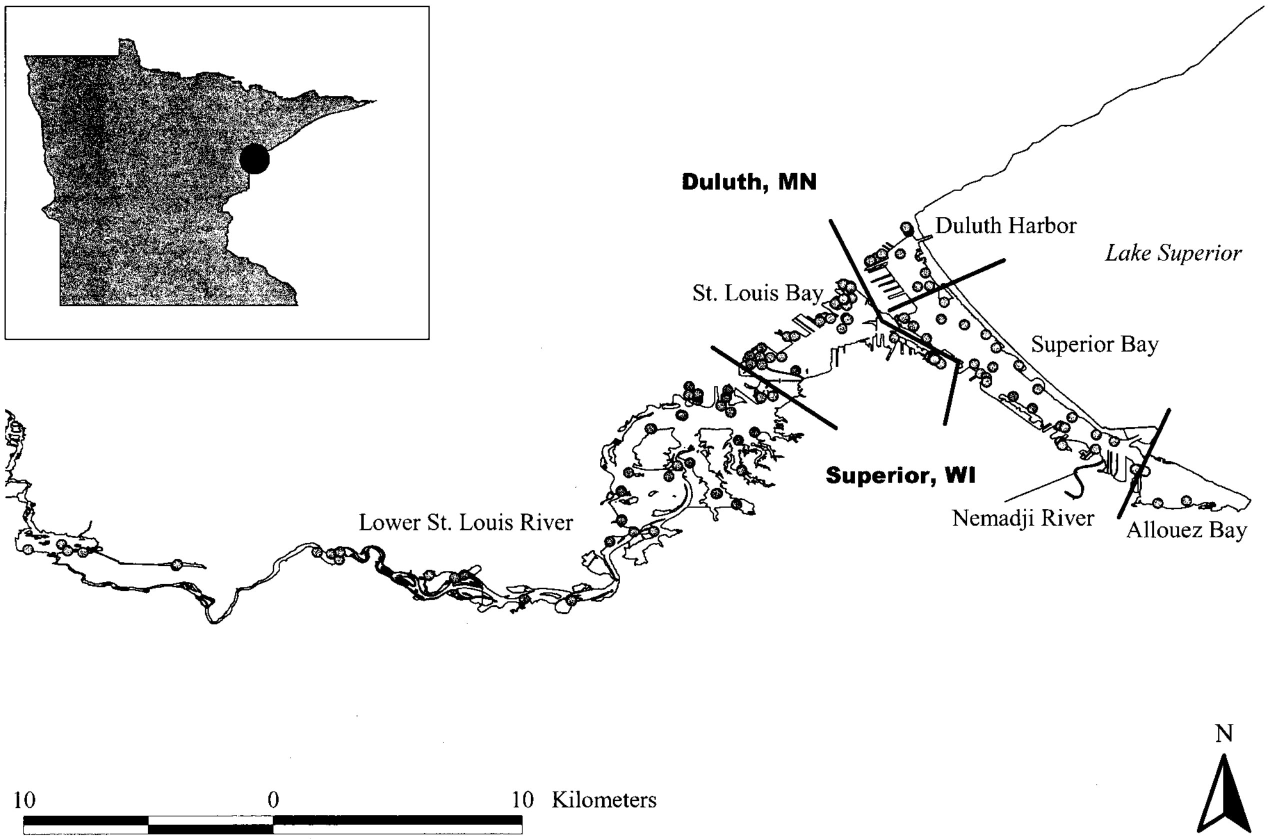

Fig. 1. Location of sediment sampling sites included in the matching sediment chemistry and toxicity database for the St. Louis River AOC. Major water body boundaries are designated on the map

Table 3. Incidence of toxicity for mean PEC-Q ranges as determined using matching sediment chemistry and toxicity data from the St. Louis River AOC (number of samples given in parentheses)

	Incidence of Toxicity		
Mean PEC-Q Range	10-Day *H. azteca* Growth or Survival (%)	10-Day *C. tentans* Growth or Survival (%)	All Tests Combined (Excluding Microtox) (%)
≤ 0.1	6.8 (44)	6.5 (46)	10 (49)
> 0.1 to ≤ 0.5	11 (80)	12 (74)	16 (87)
> 0.5 to ≤ 1.0	30 (10)	20 (10)	27 (11)
> 1.0 to ≤ 5.0	27 (11)	36 (11)	36 (14)
> 5.0	75 (4)	100 (5)	100 (5)
Overall	14 (149)	16 (146)	19 (166)

PEC-Q = probable effect concentration quotient.
Sites 102-TR and 044-TR, from the R-EMAP study (Breneman *et al.* 2000), were removed from the incidence of toxicity calculations due to incomplete sediment chemistry data (*i.e.*, PAHs, PCBs) for these known contaminated areas.

for the following freshwater geographic areas: St. Louis River AOC, other Great Lakes sites (excluding the St. Louis River AOC data), other North American sites (excluding the St. Louis River AOC data), non–Great Lakes sites in North America, all Great Lakes sites (including the St. Louis River AOC data), and all North American sites (including the St. Louis River AOC data). The above categories enabled comparisons of the St. Louis River AOC data set to be made with both independent data sets and with data sets inclusive of the St. Louis River AOC data. These comparisons were most appropriate for the two lowest mean PEC-Q ranges (≤ 0.1 and > 0.1 to ≤ 0.5), because the minimum data requirements (20 samples) were met for most geographic areas. Any comparisons of the three higher mean PEC-Q ranges should be made with caution due to the small number of sediment samples from the St. Louis River AOC (i.e., ≤ 11 samples; Tables 4 and 5).

Based on the results of 10- to 14-day toxicity tests with the amphipod *H. azteca*, the incidence of toxicity tended to in-

Table 4. Incidence of toxicity in freshwater sediments within ranges of mean PEC-Qs, based on the results of 10- to 14-day amphipod tests (number of samples given in parentheses)

	Incidence of Toxicity (%): 10- to 14-Day Amphipod (*H. azteca*) Tests[a]					
Mean PEC-Q Range	St. Louis River AOC	Other Great Lakes Sites[b]	Other North American Sites[b]	Non–Great Lakes Sites	All Great Lakes Sites	All North American Sites
≤ 0.1	6.8 (44)	50 (6)	24 (96)	22 (90)	12 (50)	18 (140)
> 0.1 to ≤ 0.5	11 (80)	25 (56)	19 (198)	16 (142)	17 (136)	16 (278)
> 0.5 to ≤ 1.0	30 (10)	52 (27)	37 (62)	26 (35)	46 (37)	36 (72)
> 1.0 to ≤ 5.0	27 (11)	68 (37)	42 (79)	19 (42)	58 (48)	40 (90)
> 5.0	75 (4)	77 (30)	70 (63)	64 (33)	76 (34)	70 (67)

PEC-Q = probable effect concentration quotient.
[a] Excludes sites 102-TR and 044-TR, from the R-EMAP study (Breneman *et al.* 2000), due to incomplete sediment chemistry data (*i.e.*, PAHs, PCBs) for these known contaminated areas.
[b] Excludes the St. Louis River AOC data.

Table 5. Incidence of toxicity in freshwater sediments within ranges of mean PEC-Qs, based on the results of 10- to 14-day midge tests (number of samples given in parentheses)

	Incidence of Toxicity (%): 10- to 14-Day Midge (*C. tentans* or *C. riparius*) Tests[a]					
Mean PEC-Q Range	St. Louis River AOC	Other Great Lakes Sites[b]	Other North American Sites[b]	Non–Great Lakes Sites	All Great Lakes Sites	All North American Sites
≤ 0.1	6.5 (46)	19 (52)	28 (72)	50 (20)	13 (98)	19 (118)
> 0.1 to ≤ 0.5	12 (74)	23 (135)	18 (222)	12 (87)	19 (209)	17 (296)
> 0.5 to ≤ 1.0	20 (10)	59 (34)	46 (52)	22 (18)	50 (44)	42 (62)
> 1.0 to ≤ 5.0	36 (11)	47 (57)	43 (75)	28 (18)	46 (68)	42 (86)
> 5.0	100 (5)	63 (30)	61 (36)	50 (6)	68 (35)	66 (41)

PEC-Q = probable effect concentration quotient.
[a] Excludes sites 102-TR and 044-TR, from the R-EMAP study (Breneman *et al.* 2000), due to incomplete sediment chemistry data (*i.e.*, PAHs, PCBs) for these known contaminated areas.
[b] Excludes the St. Louis River AOC data.

crease with greater mean PEC-Q ranges for the St. Louis River AOC, all Great Lakes, and all North American data sets (Table 4). This pattern was also observed for the other Great Lakes sites, except at mean PEC-Qs ≤ 0.1 where the incidence of toxicity was 50% (n = 6). All of the toxic samples came from the Sheboygan River, WI (Ingersoll *et al.* 2001). The Sheboygan River samples had high control survival (97%), resulting in samples with survival as high as 88% being designated as toxic. As more records are entered into the SEDTOX database, it may be possible to develop other ways of designating samples as toxic or nontoxic (*i.e.*, in addition to significance only). The incidence of toxicity at the other North American sites and non–Great Lakes sites was slightly lower at a mean PEC-Q range of > 0.1 to ≤ 0.5 than at mean PEC-Qs of ≤ 0.1 (Table 4).

The incidence of toxicity in the St. Louis River AOC for short-term amphipod tests can also be compared to the incidence of toxicity in North America for long-term amphipod toxicity tests (*i.e.*, 28- to 42-day survival, growth, or reproduction tests with amphipods [*H. azteca*]). Ingersoll *et al.* (2001) reported that mean PEC-Qs of ≤ 0.1 and > 0.1 to ≤ 0.5 were associated with a low incidence of sediment toxicity (*i.e.*, 10% and 17%, respectively), based on the results of 28- to 42-day amphipod tests. By comparison, the incidence of toxicity from these long-term amphipod tests was similar to the results from short-term amphipod tests for the St. Louis River AOC (*i.e.*, 6.8–11%; Table 4) and all Great Lakes sites (*i.e.*, 12–17%; Table 4). Survival was the principal metric for the short-term amphipod tests. Although few growth data were available for the 10-day amphipod tests conducted on St. Louis River AOC sediments, Ingersoll *et al.* (2001) noted that the relationship between toxicity and mean PEC-Q was similar when either survival alone or survival and growth together were used to classify a sample as toxic for 10-day amphipod tests conducted on sediments from throughout North America.

More matching sediment chemistry and toxicity data are needed to assess fully the predictive ability of the SQTs at mean PEC-Qs of > 0.5 in the St. Louis River AOC. However, those data that are available suggest that the incidence of acute toxicity is moderate (*i.e.*, 27–30%) at mean PEC-Qs of > 0.5 to ≤ 5.0. By comparison, long-term toxicity tests (*i.e.*, 28 to 42 days) in North America showed a relatively high incidence of toxicity (*i.e.*, 56%; 15 of 27 samples) to amphipods at mean PEC-Qs of > 0.5 to ≤ 1.0. Sediments with mean PEC-Qs of > 1.0 to ≤ 5.0 and > 5.0 were usually toxic to amphipods (*i.e.*, 96% and 100%, respectively; Ingersoll *et al.* 2001). These results emphasize the importance of using longer-term toxicity tests in assessments of sediment quality conditions. The 28- to 42-day amphipod test is more sensitive than either the 10-day amphipod or midge tests (Ingersoll *et al.* 2001), and its use would reduce the potential for false negatives at low mean PEC-Qs. In addition, use of these chronic amphipod tests increases the potential for detecting toxicity at moderate mean PEC-Qs (Ingersoll *et al.* 2001).

The predictive ability of the mean PEC-Q ranges for 10- to 14-day midge tests is given in Table 5. For the St. Louis River AOC, the incidence of toxicity in the midge tests increased from 6.5% at mean PEC-Qs $\leq$ 0.1 to 100% at mean PEC-Qs $>$ 5.0 (Table 5). At mean PEC-Qs $\leq$ 0.1, the incidence of toxicity to midges was less in the St. Louis River AOC (6.5%) than the other geographic areas, particularly for the non–Great Lakes sites (50%; n = 20). Most of the toxicity at the non–Great Lakes sites was attributed to a study of the Tennessee portion of the lower Mississippi River (Ingersoll *et al.* 2001); this data set was not further examined to determine possible factors contributing to sediment toxicity. At mean PEC-Qs $>$ 0.1 to $\leq$ 0.5, the incidence of toxicity was the same for the St. Louis River AOC and non–Great Lakes sites and somewhat greater for the other geographic areas (Table 5). In addition, the incidence of toxicity for midges and amphipods was virtually the same in the St. Louis River AOC for the lowest mean PEC-Q ranges (Tables 4 and 5). The minimum data requirements (20 samples per category) were not met for the three higher mean PEC-Q ranges for midges in the St. Louis River AOC (Table 5). Thus, only limited comparisons can be made to other geographic areas.

The results of these predictive ability evaluations indicate that, collectively, the mean PEC-Qs provide a reliable basis for classifying sediments as toxic or not toxic. At the two lowest mean PEC-Q ranges, the results for the St. Louis River AOC were generally similar to those results generated for different geographic areas for 10- to 14-day amphipod tests (Table 4) and for 10- to 14-day midge tests (Table 5). Therefore, the Level I and Level II SQTs for chemicals of potential concern (especially trace metals and PAHs) are likely to provide a reliable basis for assessing sediment quality conditions in the St. Louis River AOC.

Sediments in the St. Louis River AOC generally contain complex mixtures of contaminants (Crane *et al.* 2000). For this reason, assessments of sediment quality conditions relative to the protection of sediment-dwelling organisms should be conducted using the SQTs together (*i.e.*, through the calculation of mean PEC-Qs). Sediments with mean PEC-Qs of $\leq$ 0.1 should be considered to provide the highest level of protection for sediment-dwelling organisms (*i.e.*, Level I); the probability of observing chronic sediment toxicity is $<$ 10% in sediments with these chemical characteristics (Ingersoll *et al.* 2001). Sediments with mean PEC-Qs of $>$ 0.1 to $\leq$ 0.6 should be considered to provide a moderate level of protection for sediment-dwelling organisms (*i.e.*, Level II); the probability of observing chronic sediment toxicity is $<$ 50% in sediments with these chemical characteristics (Ingersoll *et al.* 2001). At mean PEC-Qs of $>$ 0.6, the probability of observing chronic sediment toxicity is higher (*i.e.*, $>$ 50%), indicating that sediment-dwelling organisms would be afforded a lower level of protection (Ingersoll *et al.* 2001).

The results of this study provide useful guidance for designing detailed site investigations at sites that are known to contain contaminated sediments (*i.e.*, based on sediment chemistry data). Because longer-term toxicity tests provide the most effective mean of discriminating among moderately contaminated sediment samples and because *in situ* benthic macroinvertebrates are exposed to contaminated sediments for an extended time period, it is prudent to evaluate sediment toxicity using the 28- to 42-day *H. azteca* test (endpoints: survival and growth) in sediments with mean PEC-Qs $<$ 5.0. However, it is likely to be more cost-effective to utilize acute toxicity tests to characterize the toxicity of more highly contaminated sediments (*i.e.*, mean PEC-Qs $\geq$ 5.0).

Limitations of Level I and Level II SQTs

The Level I and Level II SQTs presented herein should not be used for predicting effects in wildlife or humans through bioaccumulation pathways. Other bioaccumulation-based SQTs, adopted from the New York State Department of Environmental Conservation (NYSDEC 1999), are presented in Crane *et al.* (2000) for that purpose. The Level I and Level II SQTs are most applicable for use at sites containing soft sediments that include fine-grained particles. The SQT values should not be used for assessments of upland soils, land-applied sludge, or other land-based materials (*e.g.*, gravel) (Long and MacDonald 1998). The SQT values should also be used with caution for any sediments containing large amounts of gravel, coarse sand, tar, slag, metal ore (*e.g.*, taconite pellets), paint chips, coal chunks, fly ash, or wood chips (Long and MacDonald 1998). The presence of the aforementioned materials may render some chemicals unavailable to aquatic organisms. In addition, the age of the contaminated sediments may affect the bioavailability of PAHs and other hydrophobic organic contaminants, as partitioning studies with soils have shown these chemicals to become less bioavailable with time (Alexander 2000). The decreased desorption of these compounds as soils age may control bioavailability and, hence, degradation (Carmichael *et al.* 1997). In addition, the SQTs were derived in units of dry weight sediments; therefore, they do not directly account for the potential effects of geochemical factors in sediments that may influence contaminant bioavailability (Long and MacDonald 1998). Despite these potential limitations, the mean PEC-Qs are predictive of sediment toxicity across a variety of geographic areas in North America. Guidance on the applications of using SQTs and other sediment quality metrics (*e.g.*, sediment toxicity tests, benthic community analyses, and/or bioaccumulation tests) to assess sediment quality conditions in the St. Louis River AOC is provided in a companion paper (Crane and MacDonald 2002).

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