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Munition constituents: Preliminary sediment screening criteria for the protection of marine benthic invertebrates

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ABSTRACT

Sediment screening criteria for many munition constituents (MC) are not available in sources typically used in regulatory-driven ecological risk assessments for contaminated sediment sites. Preliminary sediment quality benchmarks (SQBs) for MC were developed for screening potential risks to marine benthic invertebrates at a munitions contaminated sediment site in Puget Sound, WA, USA. SQBs were developed for 2,4,6-trinitrotoluene (TNT) and 13 breakdown products; six other explosive nitroaromatic compounds and nitramines (e.g., RDX, HMX); and five propellants, plasticizers, and stabilizers. The SQBs were developed using freshwater and limited marine aquatic toxicity values (and hence are considered preliminary) and equilibrium partitioning theory to relate water concentrations of the compounds to sediment concentrations. The SQBs are derived from the lowest available aquatic toxicity values for aquatic invertebrates from published reviews, original studies, and database sources; ranges of $\log K_{ow}$ and K_{oc} values from published reviews and database sources, and some K_{oc} values calculated from $\log K_{ow}$. SQBs are presented for 25 MC as organic carbon-normalized values and as ranges of dry weight values for various levels of organic carbon content of sediments. Comparison of the preliminary SQBs with method detection limits and sample detection limits achieved at the contaminated sediment site demonstrates their utility in risk screening of benthic invertebrates.

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1. Introduction

Previous investigations in the vicinity of naval facilities in Puget Sound, Washington, USA, have detected munition constituents (MC)¹ in marine sediments. As an initial screening step in ecological risk assessment (ERA), detected concentrations of contaminants in sediment would typically be compared with risk-based sediment screening criteria to assess the potential for risk to benthic invertebrates. However, screening criteria for many MC have not been published within literature or database sources normally used for screening sediment chemistry, such as NOAA (2008) SQUIRT, State of Washington sediment management standards (Ecology, 2001), USEPA (1996) EcoTox thresholds, or other compilations (e.g., MacDonald et al., 1999).

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¹ Munition constituents (MC) are defined as any materials originating from unexploded ordnance, discarded military munitions, or other military munitions, including explosive and nonexplosive materials, and emission, degradation, or breakdown elements of such ordnance or munitions (US Code, 2004. 10 USC 2710 (e) (4)).

Preliminary sediment quality benchmarks (SQBs) for protection of benthic invertebrates were developed for MC for use in a baseline ERA (USEPA, 1997) at the Jackson Park Housing Complex/Naval Hospital Bremerton (JPHC) Superfund Site, Bremerton, Washington, USA. The site contains a marine sediment operable unit in Ostrich Bay, a shallow estuarine bay of Puget Sound, that is contaminated with MC. The JPHC site, formerly US Naval Ammunition Depot (NAD) Puget Sound, was used for the storage, production, and refurbishment of naval ammunition. Ordnance fillers included 2,4,6-trinitrophenylmethyl nitramine (tetryl), trinitrotoluene (TNT), Explosive D/ammonium picrate, Composition A containing hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), smokeless powder (nitroglycerine and/or fibrous nitrocellulose), black powder (nitrates), and magnesium. A list of 26 MC was developed based on compounds identified as used or potentially present at the former NAD Puget Sound facility, as listed in Table 1: TNT and 13 environmental breakdown products; six other explosive nitroaromatic compounds and nitramines (e.g., RDX, HMX); and six propellants, plasticizers, and stabilizers. Facility-related chemicals contaminated Ostrich Bay via direct discharge of wash-down wastewater through outfall drainage pipes, seeps from contaminated groundwater, storm drains, spills, and surface soil runoff. Preliminary

Table 1
Aquatic toxicity data for munition constituents.

Munition constituent	Talmage et al. (1999)		Nipper et al. (2001)		Selected toxicity value ($\mu\text{g L}^{-1}$)	Reference	Notes on selected toxicity values
	Freshwater FAV/SAV ^a ($\mu\text{g L}^{-1}$)	Freshwater FCV/SCV ^a ($\mu\text{g L}^{-1}$)	Marine FAV/NOEC ^b ($\mu\text{g L}^{-1}$)	Marine FCV/LOEC ^b ($\mu\text{g L}^{-1}$)			
2,4,6-Trinitrotoluene (TNT)							
2,4,6-Trinitrotoluene (TNT)	570	93	85.4/476 ^c	28.4/171 ^c	28.4	Nipper et al. (2001)	Preliminary FCV marine
Environmental transformation products of TNT							
2-Amino-4,6-dinitrotoluene (2-ADNT)	350	19	–	–	19	Talmage et al. (1999)	SCV freshwater
4-Amino-2,6-dinitrotoluene (4-ADNT)	–	–	–	–	30	Johnson et al. (1994)	LC ₅₀ Turbellarian flatworm (<i>Dugesia dorocephala</i>), 300 $\mu\text{g L}^{-1}$, 96 h; renewal; EPA (2008b) ECOTOX database
2,4-Diamino-6-nitrotoluene (2,4-DANT)	–	–	–	–	19	Talmage et al. (1999)	Toxicity data not available; the SCV value for 2-ADNT was used as a surrogate based on similarity in chemical structure
2,6-Diamino-4-nitrotoluene (2,6-DANT)	–	–	–	–	19		
2,4-Dinitrotoluene (2,4-DNT)	–	–	2400	2400	2400	Nipper et al. (2001)	NOEC polychaete (<i>Dinophilus gyrociliatus</i>) reproduction, 7 d, initial measured concentration
2,6-Dinitrotoluene (2,6-DNT)	–	–	1800	1800	1800	Nipper et al. (2001)	NOEC polychaete (<i>Dinophilus gyrociliatus</i>) reproduction, 7 d, initial measured concentration
2-Nitrotoluene	–	–	–	–	3400	Maas-Diepeveen and Van Leeuwen (1986)	LC ₅₀ <i>Daphnia magna</i> , 3400 $\mu\text{g L}^{-1}$; 21 d
3-Nitrotoluene	–	–	–	–	750	Maas-Diepeveen and Van Leeuwen (1986)	LC ₅₀ <i>Daphnia magna</i> , 7.5 mg L ⁻¹ , 48 h; static
4-Nitrotoluene	–	–	–	–	320	Sloof and Canton (1983)	NOEC freshwater snail (<i>Lymnaea stagnalis</i>), 0.32 mg L ⁻¹ , 40 d reproduction
1,3-Dinitrobenzene (1,3-DNB)	215	17	2400	4400	17	Talmage et al. (1999)	SCV freshwater
1,3,5-Trinitrobenzene (TNB)	60	11	350	610	11	Talmage et al. (1999)	SCV freshwater
3,5-Dinitroaniline (3,5-DNA)	460	59	–	–	59	Talmage et al. (1999)	SCV freshwater
Nitrobenzene	–	–	–	–	2700	USEPA (1978)	LC ₅₀ <i>Daphnia magna</i> , 27,000 $\mu\text{g L}^{-1}$, 48 h, static
Other nitroaromatic compounds							
2,4,6-Trinitrophenol (picric acid)	–	–	9200	20,600	9200	Nipper et al. (2001)	NOEC Mysid shrimp (<i>Americamysis bahia</i> , formerly <i>Mysidopsis bahia</i>), juvenile survival
2-Amino-4,6-dinitrophenol (picramic acid - transformation product of picric acid)	–	–	–	–	6980	Goodfellow et al. (1983).	LC ₅₀ American oyster (<i>Crassostrea virginica</i>) 69.8 mg L ⁻¹ (6-d survival)
2,4-Dinitrophenol (DNP, transformation product of picric acid)	–	–	–	–	62	USEPA (1978)	LC ₅₀ Bluegill sunfish (<i>Lopomis macrochirus</i>) 620 $\mu\text{g L}^{-1}$, 96 h. Conditions of bioassay not specified. Listed in HSDB.
2,4,6-Trinitrophenylmethylnitramine (tetryl)	–	–	15	26	15	Nipper et al. (2001)	NOEC polychaete (<i>Dinophilus gyrociliatus</i>) reproduction, 7 d, initial measured concentration
Nitramines							
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	3750	330	–	–	330	Talmage et al. (1999)	SCV freshwater
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1390	186	11,900	23,700	186	Talmage et al. (1999)	SCV freshwater
Propellants/plasticizers/stabilizers							
Nitrocellulose	–	–	–	–	–	Bentley et al. (1976)	No acute toxicity observed with fish, invertebrates, algae; 579 mg L ⁻¹
Nitroglycerin	–	–	–	–	3230	Burton et al. (1993)	NOEC cladoceran (<i>Ceriodaphnia dubia</i>), 3.23 mg L ⁻¹ , 7 d survival
Nitroguanidine	–	–	–	–	260,000	Burton et al. (1993)	NOEC cladoceran (<i>Ceriodaphnia dubia</i>), 260 mg L ⁻¹ , 7 d reproduction
Diphenylamine (DPA)	–	–	–	–	120	Dave et al. (2000)	EC ₅₀ <i>Daphnia magna</i> , 1.2 mg L ⁻¹ , 48 h
N-Nitrosodiphenylamine (NDPA)	–	–	–	–	9.2	Verschuereen (1996)	NOEC Bluegill sunfish (<i>Lopomis macrochirus</i>) 9.2 $\mu\text{g L}^{-1}$, 14 d
Pentaerythritol tetranitrate (PETN)	–	–	–	–	850,000	Bentley et al. (1975)	LC ₅₀ <i>Daphnia magna</i> , 8500 mg L ⁻¹ , 48 h; EPA (2008b) ECOTOX database

– = Not reported in Talmage et al. (1999) or Nipper et al. (2001).

FAV = freshwater acute value.

FCV = freshwater chronic value.

SAV = secondary acute value.

SCV = secondary chronic value.

^a Freshwater toxicity values calculated using EPA guidelines as Tier I FAV and FCV for TNT, or as Tier II SAV and SCV for remaining MC.

^b Preliminary marine FAV and FCV for TNT calculated using EPA guidelines. Insufficient data were available to derive SAVs or SCVs for the remaining MC, for which NOECs and LOECs are shown, and which represent the minimum detected concentration in toxicity tests of sea urchin embryo development, polychaete survival and reproduction, redfish larval survival, or mysid survival.

^c FAV and FCV values are the mean of initial and final water concentrations.

SQB_s are developed for 25 of these MC, for which toxicity data are available.

Previous studies of Ostrich Bay either failed to reliably detect MC in sediment other than picric acid or reported detections at or below method detection limits using non-regulatory analytical methods such that their presence remained suspect (EA, 1998). In addition, toxicity in marine amphipod and bivalve larvae bioassays of Ostrich Bay sediment appeared to be unrelated to the presence of any chemicals detected in the samples, including MC (Carr et al., 2001; Pascoe et al., 2002). Munition constituents are known to undergo degradation in the environment, some to numerous breakdown products, possibly including unknown compounds. Whether such compounds might be contributing to the observed toxicity in Ostrich Bay sediment has been a question. For example, Nipper et al. (2004) found that picric acid spiked to fine-grained marine sediments degrades to numerous identifiable transformation products, including 2,4-dinitrophenol (DNP), 2-amino-4,6-dinitrophenol (picramic acid), 3,4-diaminophenol, amino nitrophenol, and nitrodiaminophenol. In studies of pore water from marine sediments spiked with picric acid, two degradation products, DNP and picramic acid, were found to be more toxic than the parent compound (Nipper et al., 2005), yet the toxicity of these compounds was not sufficient to account for the full toxicity of the spiked picric acid.

In addition to the present study, the baseline ERA for the JPHC site also included the development of analytical methods to achieve lower sediment method detection limits (MDLs) for MC that were previously uncharacterized in Ostrich Bay sediment but were suspected of being present based on past site use (TtEC, 2009). The MDLs and sample detection limits (DLs) for sediments collected from the operable unit are compared with the preliminary SQB_s to evaluate their sufficiency to detect MC at levels below potential risks to benthic invertebrates.

2. Methods

2.1. Sediment quality benchmark development

Sediment quality benchmarks were developed for MC using the equilibrium partitioning (EqP) approach to relate water concentrations to sediment concentrations, as described in Di Toro et al. (1991) and USEPA (1993b, 2008a). USEPA (2008a) advocates the use of EqP as a conservative approach to developing sediment screening values. The EqP method for deriving SQB_s uses partitioning theory to relate the sediment concentration to the equivalent free chemical concentration in pore water and in sediment organic carbon, and as such are organic carbon-based SQB_s (SQB_{OC}_s) for comparison with organic carbon-normalized sediment chemistry data. The porewater concentration would typically be the effects concentration for benthic species exposure. As Di Toro et al. (1991) states, the use of water toxicity test data or water quality criteria relative to the water concentration assumes that the sensitivities of benthic species and species tested are similar, and that the levels of protection afforded by water toxicity tests are appropriate for benthic organisms. Data available for examination by Di Toro et al. (1991) suggest that, on average, benthic and water column species are similarly sensitive, which supports the use of water toxicity test data to derive SQB_s for the protection of infaunal and epifaunal species.

Sediment spiking experiments (e.g., Green et al., 1999; Lotufo et al., 2001; Nipper et al., 2002, 2004; Rosen and Lotufo, 2005) were not used in the development of screening criteria for MC due to the uncertainties associated with experimental design and interpretation of results. As discussed in a recent review (Lotufo et al., 2009), variability among spiked sediment studies is likely

due to rapid degradation of MC in sediment (e.g., transformation is more likely to occur during spiking and mixing than in water-only tests) and inaccuracies in quantifying exposure concentrations. Water-only toxicity tests were considered to provide greater quality control over solubility and stability of the test chemicals compared to sediment spiking studies.

Toxicity values consisted of available chronic water screening benchmarks and/or no-observed effects concentrations (NOECs) from water toxicity tests. The NOEC value is a concentration below which no adverse effect from exposure to a specific chemical was observed. Chronic water screening benchmarks for eight MC were taken from the review of Talmage et al. (1999), which calculated final or secondary chronic values (FCVs or SCVs) for MC in freshwater following USEPA Tier I and Tier II methods (Stephan et al., 1985; USEPA, 1993a). Nipper et al. (2001) present a preliminary marine FCV for TNT and NOEC/LOEC data on survival and reproduction for seven additional MC using five species of marine organisms, from which the lowest NOECs were selected for SQB derivation. For the remaining MC, toxicity data consist of NOEC, LC₅₀, and EC₅₀ values from freshwater and marine toxicity tests, identified from searches of published literature and databases. The selected chronic toxicity values are consistent with or lower than those in a recent compilation in Nipper et al. (2009), which cites many of the sources used herein. The databases consisted of USEPA (2008b) ECOTOX, the US Army Center for Health Promotion and Preventive Medicine (USACHPPM, 2008), the Los Alamos National Laboratory (LANL, 2005), ToxNet (2009), and Hazardous Substances Data Bank (HSDB) (2009). The remaining NOEC, LC₅₀, and EC₅₀ values represent minimum detected concentrations using combined toxicity data for both acute and chronic experiments that identified significant reductions in survival, growth, or reproduction. NOEC values that were below the analytical detection limits were not used. Toxicity values from acute studies (i.e., LC₅₀ and EC₅₀) were divided by 10 to approximate chronic values. Table 1 presents a summary of the selected toxicity values and sources used for SQB derivation for the 26 MC. A comprehensive toxicity data set for all 26 MC is not reported; however, additional studies that were reviewed for toxicity data and equilibrium partitioning coefficients are listed in an appendix.

In the EqP procedure, the SQB_{OC} is calculated from the FCV or SCV, the NOEC, or the adjusted LC₅₀ or EC₅₀, and the theoretical partitioning between water and sediment. Partitioning is determined by the mass fraction of organic carbon in the sediment and is expressed for nonionic chemicals through the partition coefficient for organic carbon (K_{oc}). The low end of the range of K_{oc} values, the single value where a range was unavailable, and some of the high end values were estimated from $\log K_{ow}$ by Eq. (1) (Di Toro, 1985):

$$\log K_{oc} = 0.00028 + 0.983(\log K_{ow}) \quad (1)$$

Eq. (1) has been previously used by Talmage et al. (1999) to develop a freshwater partitioning-based SQB_{OC} for TNT.

SQB_{OC} values in units of mg kg⁻¹ organic carbon (OC) were then calculated by Eq. (2) (Di Toro et al., 1991):

$$SQB_{oc} = K_{oc} \times \text{chronic toxicity value} \quad (2)$$

where K_{oc} is the organic carbon partitioning coefficient (L kg⁻¹ sediment); and chronic toxicity value is the final or secondary chronic value or NOEC; or LC₅₀/EC₅₀ derived from acute toxicity tests multiplied by 0.1 for conversion to a chronic value (units converted to mg L⁻¹).

Values and sources for the ranges of $\log K_{ow}$ and K_{oc} for the 26 MC are listed in Table 2. The chronic toxicity value for each MC was used with the range of K_{oc} values to derive a range of SQB_{OC}_s. Presentation of a range of SQB_{OC}_s for MC reflects uncertainties in the partitioning parameters, and is consistent with USEPA

Table 2
Partition coefficients for munition constituents.

Munition constituent	Log K_{ow}			K_{oc}		Source of high K_{oc}
	Low	High	Source of K_{ow}	(L kg ⁻¹)		
				Low (calculated) ^a	High	
<i>2,4,6-Trinitrotoluene (TNT)</i>						
2,4,6-Trinitrotoluene (TNT)	1.6	2.7	Talmage et al. (1999)	37.4	451	Calculated ^a
<i>Environmental transformation products of TNT</i>						
2-Amino-4,6-dinitrotoluene (2-ADNT)	1.85	1.94	Elovitz and Weber (1999), Walsh et al. (1995)	65.9	81	Calculated
4-Amino-2,6-dinitrotoluene (4-ADNT)	2.1	–	Elovitz and Weber (1999)	116	–	Calculated
2,4-Diamino-6-nitrotoluene (2,4-DANT)	0.7	–	Elovitz and Weber (1999)	4.88	–	Calculated
2,6-Diamino-4-nitrotoluene (2,6-DANT)	0.7 ^b	–	Elovitz and Weber (1999)	4.88	–	Calculated
2,4-Dinitrotoluene (2,4-DNT)	1.98	–	Hansch et al. (1995)	88.4	300	Environment Agency (2000)
2,6-Dinitrotoluene (2,6-DNT)	2.1	–	Nakagawa et al. (1992)	116	150	Environment Agency (2000)
2-Nitrotoluene	2.3	–	Hansch et al. (1995)	182	–	Calculated
3-Nitrotoluene	2.45	–	Hansch et al. (1995)	256	–	Calculated
4-Nitrotoluene	2.37	–	Hansch et al. (1995)	214	–	Calculated
1,3-Dinitrobenzene (1,3-DNB)	1.49	–	Hansch et al. (1995)	29.2	210	Layton et al. (1987)
1,3,5-Trinitrobenzene (TNB)	1.18	1.4	Hansch and Leo (1985), Talmage et al. (1999)	14.5	77	Layton et al. (1987)
3,5-Dinitroaniline (3,5-DNA)	1.89	–	Zakikhani et al. (2002)	72.1	–	Calculated
Nitrobenzene	1.85	–	Hansch et al. (1995)	65.9	131	Layton et al. (1987)
<i>Other nitroaromatic compounds</i>						
2,4,6-Trinitrophenol (picric acid)	1.6	–	Layton et al. (1987)	37.4	–	Calculated
2-Amino-4,6-dinitrophenol (picramic acid)	0.93	–	Hansch et al. (1995)	8.21	–	Calculated
2,4-Dinitrophenol	1.37	1.75	Hansch et al. (1995)	22.2	53	Calculated
2,4,6-Trinitrophenylmethylnitramine (Tetryl)	1.65	2	Jenkins (1989) (cited in Talmage et al. (1999)), Layton et al. (1987)	41.9	406	Layton et al. (1987)
<i>Nitramine compounds</i>						
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	0.06	0.59	Yoon et al. (2002), Talmage et al. (1999)	1.15	130	Layton et al. (1987)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	0.81	0.9	Talmage et al. (1999), Monteil-Rivera et al. (2004)	6.26	42	Layton et al. (1987)
<i>Propellants/plasticizers/stabilizers</i>						
Nitrocellulose	–	–	–	–	–	–
Nitroglycerin	1.62	2	Hansch et al. (1995), Walsh et al. (1995)	39.2	180	ToxNet (2009)
Nitroguanidine	–0.89	–	Hansch et al. (1995)	0.13	25	ToxNet (2009)
Diphenylamine (DPA)	3.42	–	Drzyzga (2003)	2302	–	Calculated
N-nitrosodiphenylamine (NDPA)	3.13	–	Veith et al. (1980)	1194	–	Calculated
Pentaerythritol tetranitrate (PETN)	1.61	2.0	HSDB (2009), Quinn et al. (2009)	38.3	179	Quinn et al. (2009)

K_{oc} = organic carbon partitioning coefficient (L kg⁻¹).

^a $\log K_{oc} = 0.00028 + 0.983 (\log K_{ow})$.

^b K_{ow} was unavailable; the K_{ow} value for 2,4-DANT was used as a surrogate based on similarity in chemical structure.

(2008a) guidelines on developing sediment benchmarks based on partitioning. Where a range of K_{oc} values is shown in Table 2, the low K_{oc} value represents the value calculated from the lowest log K_{ow} whereas the high value shown reflects either the highest value reported in the literature or the value calculated from the higher listed log K_{ow} . For comparison with SQB_{OCs}, sediment characterization data in $\mu\text{g kg}^{-1}$ dry weight are normalized to organic carbon content of the sample by dividing by the sample organic carbon fraction (f_{oc}).

2.2. Sediment collection

Surface sediment samples were collected as 0–10 cm of sediment from 43 locations in the JPHC marine operable unit within Ostrich Bay, Puget Sound, WA. Nine of these locations were resampled in a second phase to collect material for concurrent toxicity testing and chemical analyses, for a total of 52 samples. Samples were collected in fall of 2009 using a double van Veen grab, following US EPA regional protocols for sediment sample collection and preparation in Puget Sound (PSEP 1997a), as described in more de-

tail elsewhere (TtNUS, 2010). Split samples were collected for chemical analyses and toxicity testing.

2.3. Analytical methods for MC in sediment

Methods of sediment sample storage and preparation followed US EPA regional guidelines for Puget Sound (PSEP, 1997b), with details described elsewhere (TtNUS, 2010). All MC except nitrocellulose were analyzed using EPA Method 8330B, modified by the analytical laboratory (Columbia Analytical Services Inc., Kelso, WA) to achieve MDLs below or as close as possible to the SQBs: nitroguanidine, nitroglycerin, nitrobenzene, 2-NT, 3-NT, and 4-NT by EPA Method 8330B with HPLC-UV; 4-NDPA, dibutylphthalate, diethylphthalate, and DPA by EPA Method 8270 with GC/MS; picric acid, picramic acid, and 2,4-dinitrophenol by EPA Method 8330B with HPLC-MS/MS; HMX, RDX, TNB, 1,3-DNB, tetryl, TNT, 2-ADNT, 4-ADNT, 2,4-DNT, 2,6-DNT, 3,5-DNA, PETN, 2,4-DANT, and 2,6-DANT by EPA Method 8330B with HPLC-UV and LC-MS/MS. Samples for nitrocellulose analysis were prepared following the method of MacMillan et al. (2008) and analyzed by modified EPA Method 353.2.

2.4. Benthic invertebrate toxicity tests

Splits of sediment samples from 35 of the locations in Ostrich Bay were tested for toxicity to marine benthic invertebrates following State of Washington and US EPA regional protocols for Puget Sound (Ecology, 1995, PSEP 1995). Toxicity was tested at NewFields Northwest laboratory (Port Gamble, WA) with the acute 10-d amphipod (*Ampelisca abdita*) mortality test, the acute 48-h bivalve larval mortality/abnormality test (*Mytilus galloprovincialis*), and the chronic 20-d juvenile polychaete survival and growth test (*Neanthes arenaceodentata*). The bivalve larval test was initially found to respond to the fine grains present in the sediment samples, resulting in larval entrainment within the sediment matrix. A subset of the samples was re-tested using a modified procedure where the sediment samples were tested within a screen tube to control for the entrainment, following the procedure of Anderson et al. (2001). Consequently, 20 of the 35 bivalve larval test samples were considered to pass performance criteria for the bivalve larval bioassay (Ecology, 1991a); results for the remaining bivalve larval test samples are not used. Reference area samples were obtained from Carr Inlet, a Washington State Department of Ecology-approved toxicity test reference location (Ecology, 1991b). Details of the toxicity testing methods and results are presented elsewhere (TtNUS, 2010).

3. Results and discussion

3.1. Sediment quality benchmarks for munition constituents

Ranges of organic carbon-normalized preliminary SQB_{OCs} for 25 MC for the protection of benthic invertebrates are presented in

Table 3. Preliminary SQBs could not be calculated for nitrocellulose due to a lack of suitable aquatic toxicity data. For rapid screening purposes with sediment characterization data in dry weight units and for comparison with MDLs, the organic carbon-normalized SQB_{OCs} have been converted to dry weight values based on a range of sediment organic carbon contents (Table 3). The three lowest SQB_{OCs} of the 25 MC were calculated for transformation products of TNT. The SQB_{OCs} at the low end of the range for 2,4-DANT and 2,6-DANT are both calculated at 0.09 mg kg⁻¹ OC, reflecting the use of a surrogate toxicity value from 2-ADNT (Table 1) and a surrogate *K*_{ow} for 2,6-DANT (Table 2). These SQB_{OCs} are more uncertain than those for other MC because of the reliance on surrogate toxicity and partitioning values. The SQB_{OC} for TNB, also a transformation product of TNT as well as an explosive and industrial chemical (Talmage et al., 1999), is calculated at 0.16 mg kg⁻¹ OC, which is one-seventh the calculated SQB_{OC} for TNT (1.1 mg kg⁻¹ OC). The SQB_{OCs} for HMX (0.38 mg kg⁻¹ OC), 1,3-dinitrobenzene (0.5 mg kg⁻¹ OC), and tetryl (0.6 mg kg⁻¹ OC) are somewhat higher than the lowest three SQB_{OCs} but lower than the TNT SQB_{OC}. The SQB_{OCs} for the remaining MC are higher than the SQB_{OC} for TNT.

The compiled toxicity values in Table 1 illustrate the higher freshwater toxicity of the TNT transformation products 2-ADNT, 4-ADNT (and possibly 2,4-DANT and 2,6-DANT based on assumed toxicity), 1,3-DNB, and TNB relative to the parent TNT. This higher toxicity is ultimately reflected in lower SQBs for 2-ADNT, 2,4-DANT, 2,6-DANT, 1,3-DNB, and TNB relative to TNT. This trend of higher toxicity in the TNT transformation products was noticed in the review of Talmage et al. (1999). Tetryl and NDPA also appear to exhibit higher aquatic toxicity than TNT, based on limited studies.

Most of the SQBs in Table 3 are derived from freshwater aquatic toxicity values (Table 1), and for some MC only single studies were

Table 3
Sediment quality benchmarks for munition constituents.

Munition constituent	SQB _{OC}		SQB dry weight (μg kg ⁻¹ dw)							
	(mg kg ⁻¹ OC)		0.5% OC		1% OC		2% OC		3% OC	
	Low	High	Low	High	Low	High	Low	High	Low	High
2,4,6-Trinitrotoluene (TNT)	1.1	12.8	5.5	64	11	128	22	256	33	384
<i>Environmental transformation products of TNT</i>										
2-Amino-4,6-dinitrotoluene (2-ADNT)	1.3	1.5	6.5	7.5	13	15	26	30	39	45
4-Amino-2,6-dinitrotoluene (4-ADNT)	3.5	–	18	–	35	–	70	–	105	–
2,4-Diamino-6-nitrotoluene (2,4-DANT)	0.09	–	0.45	–	0.9	–	1.8	–	2.7	–
2,6-Diamino-4-nitrotoluene (2,6-DANT)	0.09	–	0.45	–	0.9	–	1.8	–	2.7	–
2,4-Dinitrotoluene (2,4-DNT)	210	720	1050	3600	2100	7200	4200	14 400	6300	21 600
2,6-Dinitrotoluene (2,6-DNT)	210	270	1050	1350	2100	2700	4200	5400	6300	8100
2-Nitrotoluene	620	–	3100	–	6200	–	12 400	–	18 600	–
3-Nitrotoluene	190	–	950	–	1900	–	3800	–	5700	–
4-Nitrotoluene	68	–	340	–	680	–	1360	–	2040	–
1,3-Dinitrobenzene (1,3-DNB)	0.5	3.57	2.5	18	5.0	36	10	71	15	107
1,3,5-Trinitrobenzene (TNB)	0.16	0.85	0.80	4.3	1.6	8.5	3.2	17	4.8	26
3,5-Dinitroaniline (3,5-DNA)	4.3	–	21.3	–	42.6	–	85.2	–	127.8	–
Nitrobenzene	180	350	900	1750	1800	3500	3600	7000	5400	10,500
<i>Other nitroaromatic compounds</i>										
2,4,6-Trinitrophenol (picric acid)	340	–	1700	–	3400	–	6800	–	10,200	–
2-Amino-4,6-dinitrophenol (picramic acid)	57.0	–	285	–	570	–	1140	–	1710	–
2,4-Dinitrophenol	1.4	3.3	7.0	17	14	33	28	66	42	99
2,4,6-Trinitrophenylmethylnitramine (tetryl)	0.6	6.1	3	30.5	6	61	12	122	18	183
<i>Nitramine compounds</i>										
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	0.38	42.9	1.9	215	3.8	429	7.6	858	11.4	1287
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1.2	7.8	6.0	39	12	78	24	156	36	234
<i>Propellants/plasticizers/stabilizers</i>										
Nitrocellulose	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC
Nitroglycerin	127	581	633	2907	1265	5814	2530	11 628	3795	17 442
Nitroguanidine	35	6500	174	32 500	347	65 000	694	130 000	1041	195 000
Diphenylamine (DPA)	280	–	1400	–	2800	–	5600	–	8400	–
N-nitrosodiphenylamine (NDPA)	11.0	–	55	–	110	–	220	–	330	–
Pentaerythritol tetranitrate (PETN)	32 533	152 150	162 665	760 750	325 330	1521500	650 660	3043 000	975 990	4564 500

OC = organic carbon. – = Not calculated. NC = not calculable; toxicity data not available.

found. As such, the available data on which to base SQBs for MCs is very limited. Nipper et al. (2001) developed marine toxicity values for eight MC: TNT, 2,4-dinitrotoluene, 2,6-dinitrotoluene, 1,3-DNB, TNB, tetryl, picric acid, and RDX. A marine aquatic toxicity value was also found for picramic acid (Table 1). Nipper et al. (2001) suggests that comparison of marine and freshwater organism toxicity values for TNT indicates the higher sensitivity of marine organisms to the toxicity of MC than freshwater organisms. Nonetheless, the freshwater toxicity values for DNB, TNB, and RDX reported in Talmage et al. (1999) are lower than the marine values in Nipper et al. (2001). Lotufo et al. (2009) recently discussed the uncertainty in existing laboratory sediment toxicity data for developing sediment benchmarks for MC. Due to the limited availability of toxicological data, the SQBs developed in this study are based on the lower of available toxicity values, whether marine or freshwater. Because of the lack of a comprehensive or more robust toxicity data set for MC and uncertainties in partitioning coefficients, the SQBs for the 25 MC are considered preliminary. As new toxicological and physicochemical data become available for MC, the SQBs can be revised and updated.

Table 3 also presents ranges of dry weight-based SQBs for four levels of total organic carbon content in sediments (e.g., 0.5%, 1%, 2%, and 3% organic carbon), similar to the presentation format in USEPA (2008a). In sediment with total organic carbon greater than 0.2%, organic carbon is the predominant phase for chemical sorption and application of organic carbon normalization to sediments above this range is considered to be valid (Di Toro et al., 1991; Michelsen and Bragdon-Cook, 1993). Normalization at organic carbon contents between 0.1% and 0.2% may be appropriate for some data (Di Toro and De Rosa, 1998).

The partitioning model of Di Toro et al. (1991) was developed from data on non-polar nonionic organic chemicals with $\log K_{ow} > 1$, and the model is considered to be most effective for chemicals with $\log K_{ow} > 2$ (USEPA, 2008a). Partitioning behavior of MC with $\log K_{ow} < 2$, such as 2,4-DANT, 1,3-DNB, TNB, picric acid, picramic acid, 2,4-dinitrophenol, HMX, RDX, nitroglycerine, nitroguanidine, and PETN, may deviate from this model. Fuchsman (2003) demonstrates that equilibrium partitioning may inaccurately predict the bioavailable concentration of organic compounds with $\log K_{ow} < 3$. The reasoning is that the equilibrium partitioning equation assumes an association with sediment organic carbon and that the amount of chemical in the dissolved phase is negligible. This assumption may not hold for chemicals with low K_{ow} , for which a substantial fraction may be in the dissolved phase, and for which the SQB_{OC}s in Table 3 may be unnecessarily low. Based on Fuchsman (2003), USEPA (2008a) provides a modification to the equilibrium partitioning equation to account for the fraction of solids in the sediment sample, which can be applied to derive a sample-specific dry weight SQB_{dw} for MC as:

$$\text{Sample SQB}_{dw} = \text{chronic toxicity value} \times \left[\frac{f_{OC}K_{OC}}{((1 - f_{Solids}) \div f_{Solids})} \right] \quad (3)$$

where the sample-specific SQB_{dw} is in units of $\mu\text{g kg}^{-1}$ dry weight sediment and f_{Solids} is the fraction of sediment present as solids. The sample-specific SQB_{OC} would be determined by dividing the SQB_{dw} by the fraction of organic carbon in the sediment sample (f_{OC}). With either low f_{OC} (0.002) or low f_{Solids} (0.2), the modified sample SQB_{OC} would increase from the SQB_{OC} derived from Eq. (2) for those MC with a $\log K_{ow}$ of approximately 2.5 (USEPA, 2008a).

To illustrate the increase in SQB_{OC} based on the modification of Fuchsman (2003), modified sample-specific SQB_{OC}s were calculated for select MC with low SQB_{OC}s. Sample-specific SQB_{OC}s were calculated using Eq. (3) and the f_{OC} and f_{Solids} data for the 52 sediment samples collected from Ostrich Bay, with f_{OC} ranging from 0.001 to 0.035 (i.e., 0.1–3.5% organic carbon) and f_{Solids} ranging

from 0.28 to 0.85. For 2,4-DANT, the resultant sample-specific SQB_{OC}s range from 0.7 to 3.9 mg kg^{-1} OC, which are higher than the Low SQB_{OC} of 0.09 mg kg^{-1} OC in Table 3. For 1,3-DNB, the sample-specific SQB_{OC}s range from 1.1 to 3.9 mg kg^{-1} OC, compared with the Low SQB_{OC} of 0.5 mg kg^{-1} OC. For TNB, the sample-specific SQB_{OC}s range from 0.5 to 2.4 mg kg^{-1} OC, compared with the Low SQB_{OC} of 0.16 mg kg^{-1} OC. And for HMX, the sample-specific SQB_{OC}s range from 11.3 to 66.5 mg kg^{-1} OC, compared with the Low SQB_{OC} of 0.38 mg kg^{-1} OC.

For polar nonionic chemicals such as nitroaromatics and nitramines, the target lipid model of Kipka and Di Toro (2009) may be useful for future modeling efforts. Employing the target lipid model may necessitate the identification of solute parameters and use of the polyparameter linear free energy relationship approach described in Nguyen et al. (2005).

SQB_{OC}s have previously been developed for a limited number of MC by Oak Ridge National Laboratory (Hovatter et al., 1997; Talmage et al., 1999), using the EqP model of Di Toro as used herein. The values in Talmage et al. (1999) are updated from Hovatter et al. (1997), and the SQB_{OC}s are similar. Many of the FCVs and SCVs reported in Talmage et al. (1999) are used in the present study as toxicity values for the SQB derivation. Comparison of the SQB_{OC} values from Talmage et al. (1999) with the ranges derived herein is presented in Table 4. In general, the low ends of the ranges of SQB_{OC}s calculated herein are slightly lower than the values presented in Talmage et al. (1999). With the use of the high ends of the ranges of K_{ow} and K_{OC} values, the high ends of the ranges of calculated SQB_{OC}s are higher than those reported in Talmage et al. (1999), with some values substantially higher (e.g., the High preliminary SQB_{OC} for HMX in Table 4 is almost 100-fold higher). Differences between the SQB_{OC}s published by Oak Ridge and those derived herein can be attributed to slight differences in chronic toxicity values and to the use of a range of partition coefficients.

Sediment toxicity values for nine MC based on sediment spiking experiments have been compiled in Lotufo et al. (2009), and the NOECs are compared in Table 4 with the dry weight SQB_{dw} values derived in this study. The spiked sediment NOECs for all MC except 2,6-DNT are substantially higher than the SQB_{dw} values, for similar OC content. The greatest differences are seen for those MC with SQB_{dw}s derived from freshwater SCVs reported in Talmage et al. (1999) and with spiked sediment NOECs derived from exposures of the freshwater amphipod *Hyalella azteca* or the freshwater midge *Chironomus tentans*. For example, the spiked sediment NOECs range from slightly more than two to over five orders of magnitude higher than the Low SQB_{dw} values for 2,ADNT, 2,4-DANT, TNB,

Table 4
Comparison of available sediment quality benchmarks for munition constituents.

Munition constituent	Carbon-normalized SQBs developed in present study ^a (mg kg^{-1} OC)		Talmage et al. (1999) ^b (mg kg^{-1} OC)
	Low	High	
2,4,6-Trinitrotoluene (TNT)			
2,4,6-Trinitrotoluene (TNT)	1.1	13	9.2
<i>Environmental transformation products of TNT</i>			
1,3-Dinitrobenzene (1,3-DNB)	0.5	3.57	0.67
1,3,5-Trinitrobenzene (TNB)	0.16	0.85	0.24
<i>Nitramine compounds</i>			
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	0.38	43	0.47
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1.2	7.8	1.3

^a Based on marine preliminary FCV for TNT (Nipper et al., 2001) and freshwater SCVs for remaining MC (Talmage et al., 1999) using ranges of K_{OC} values.

^b Based on freshwater FCV for TNT and freshwater SCVs for remaining MC.

HMX, and RDX (Table 4), with the spiked sediment NOECs for RDX and HMX at 17 000 and 60 000-fold higher than their corresponding Low SQB_{dw} values. Much smaller differences are seen for MC with SQB_{dw,s} based on marine NOECs from Nipper et al. (2001) and the spiked sediment NOECs based on marine amphipods; e.g., spiked sediment NOECs are less than one to two orders of magnitude greater than the SQB_{dw} values for 2,6-DNT, picric acid, and tetryl (Table 4). The differences between the equilibrium partitioning-based SQBs and the spiked sediment NOECs may be related to uncertainties in quantifying exposures in spiked sediment due to chemical and biological transformation (Lotufo et al., 2009) and in the validity of assumed steady state equilibrium in both approaches.

3.2. Protectiveness of SQBs

The preliminary SQBs were used to determine whether method detection limits (MDLs) for MC in sediment are sufficient to show an absence of risk to benthic invertebrates, and to screen sample detection limits (DLs) and sediment characterization data from Ostrich Bay as part of the baseline ERA for the JPHC site. Undetected MC with MDLs or sample DLs above their SQBs would present

uncertain levels of risk to benthic invertebrates. The comparison of SQBs with MDLs and sample DLs in Table 5 demonstrates the effectiveness of the SQBs to be risk protective, i.e., the MDLs and sample DLs for most MC are below their respective SQBs, with the exception of 2,4-DANT, 2,6-DANT, 4-nitrotoluene, 1,3-DNB, 1,3,5-TNB, HMX, and nitroglycerin for which MDLs are higher than their Low SQB_{dw,s}. However, the MDLs for 1,3-DNB, 1,3,5-TNB, HMX, and nitroglycerin are below their High SQB_{dw,s}, leaving only 2,4-DANT, 2,6-DANT, and 4-nitrotoluene with MDLs above their SQB_{dw,s} (Table 5).

The comparison of MDLs with sample-specific SQB_{dw} values calculated with Eq. (3) above results in far fewer exceedances of the SQB_{dw} values by the MDLs. For example, the MDL of 15 µg kg⁻¹ dw for 2,4-DANT would exceed the sample-specific SQB_{dw}, which ranges from 3.6 to 51.8 µg kg⁻¹, in 33% of the samples rather than in all of them. For 2,6-DANT, the MDL of 4.9 µg kg⁻¹ dw would exceed the sample-specific SQB_{dw} for 8% of the samples. The MDL of 6.2 µg kg⁻¹ dw for 1,3-DNB would exceed the sample-specific SQB_{dw}, which ranges from 4.5 to 60.7 µg kg⁻¹ dw, in 10% of the samples. The MDL of 2.8 µg kg⁻¹ dw for TNB would exceed the sample-specific SQB_{dw,s}, which ranges from 2.4 to 33.7 µg kg⁻¹ dw, in 8% of the samples. And lastly, the MDL of 2.5 µg kg⁻¹ dw for

Table 5
Sediment method detection limits, sample detection limits, and detected concentrations for munition constituents.

Munition constituent	Method detection limit ^a µg kg ⁻¹ dw	Ranges of sample detection limits for Ostrich Bay sediment		Maximum detected concentration at Ostrich Bay		SQB (µg kg ⁻¹ dw)		SQB _{OC} (mg kg ⁻¹ OC)		
		µg kg ⁻¹ dw	mg kg ⁻¹ OC	µg kg ⁻¹ dw	mg kg ⁻¹ OC ^b	Low ^c	High ^c	Low	High	
2,4,6-Trinitrotoluene (TNT)	1.1	1.1 – 3.1	0.034–0.22	ND	ND	5.5	64	1.1	12.8	
<i>Environmental transformation products of TNT</i>										
2-Amino-4,6-dinitrotoluene (2-ADNT)	0.9	0.9 – 2.6	0.031–0.18	ND	ND	6.5	7.5	1.3	1.5	
4-Amino-2,6-dinitrotoluene (4-ADNT)	1.6	1.6 – 4.5	0.058–0.32	ND	ND	18	NC	3.5	NC	
2,4-Diamino-6-nitrotoluene (2,4-DANT)	15	15–42	0.54–3.0	ND	ND	0.45	NC	0.09	NC	
2,6-Diamino-4-nitrotoluene (2,6-DANT)	4.9	4.9–14	0.16–0.97	ND	ND	0.45	NC	0.09	NC	
2,4-Dinitrotoluene (2,4-DNT)	12	12–34	0.37–2.4	ND	ND	1050	3600	210	720	
2,6-Dinitrotoluene (2,6-DNT)	6.9	6.9–20	0.22–1.4	ND	ND	1050	1350	210	270	
2-Nitrotoluene	330	330–440	10–65	ND	ND	3100	NC	620	NC	
3-Nitrotoluene	390	390–520	11–77	ND	ND	950	NC	190	NC	
4-Nitrotoluene	530	530–710	17–105	ND	ND	340	NC	68	NC	
1,3-Dinitrobenzene (1,3-DNB)	6.2	6.2–18	0.2–1.2	ND	ND	2.5	18	0.5	3.57	
1,3,5-Trinitrobenzene (TNB)	2.8	2.9–7.8	0.09–0.58	ND	ND	0.8	4.3	0.16	0.85	
3,5-Dinitroaniline (3,5-DNA)	1.2	1.2–3.4	0.041–0.24	ND	ND	21.3	NC	4.3	NC	
Nitrobenzene	170	170–230	5–33	ND	ND	900	1750	180	350	
<i>Other nitroaromatic compounds</i>										
2,4,6-Trinitrophenol (picric acid)	0.75	0.74–0.94	0.024–0.15	2.7	0.1	1700	NC	340	NC	
2-Amino-4,6-dinitrophenol (picramic acid)	0.19	0.19–0.46	0.006–0.04	0.21	0.01	285	NC	57	NC	
2,4-Dinitrophenol	0.25	0.25–10	0.008–1.22	0.42	0.02	7	17	1.4	3.3	
2,4,6-Trinitrophenylmethyl nitramine (tetryl)	2.3	2.1–6.5	0.069–0.48	ND	ND	3	30.5	0.6	6.1	
<i>Nitramine compounds</i>										
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2.5	2.5–7	0.078–0.5	ND	ND	1.9	215	0.38	42.9	
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1.8	1.8–5.1	0.062–0.36	ND	ND	6	39	1.2	7.8	
<i>Propellants/plasticizers/stabilizers</i>										
Nitrocellulose	1500 ^d	1500–15 000	50–1937	12,000	1440	NC	NC	NC	NC	
Nitroglycerin	670	670–890	20–133	ND	ND	630	2910	127	581	
Nitroguanidine	130	130–160	4–27	650	36	174	32 500	35	6500	
Diphenylamine (DPA)	1.4	1.4–48	0.062–1.5	ND	ND	1400	NA	280	NC	
N-nitrosodiphenylamine (NDPA)	1.6	1.6–55	0.072–1.7	ND	ND	55	NA	11	NC	
Pentaerythritol tetranitrate (PETN)	2	2–5.6	0.07–0.4	ND	ND	162 665	760 750	32 533	152 150	

OC = organic carbon. NC = not calculatable due to lack of toxicity data or alternative K_{ow}/K_{oc} values, or due to TOC < 0.5% for concentration data normalized to OC. NA = not applicable. ND = Not detected.

^a EPA Method 8330 as modified by Columbia Analytical Services (CAS) for the US Navy, Engineering Field Command, Poughkeepsie, NY, except for nitrocellulose.

^b Maximum detection limit for those samples with total organic carbon (TOC) > 0.5%.

^c Assumes 0.5% OC.

^d MDL based on MacMillan et al. (2008) and EPA Method 353.2.

HMX would not exceed the range of sample-specific SQB_{dws} , 58.1–858 $\mu\text{g kg}^{-1}$ dw, in any of the samples.

The protectiveness of the preliminary SQBs is also evaluated by comparison of the SQB_{OC} values with organic carbon-normalized concentration data for MC in sediments collected from Ostrich Bay, coupled with toxicity test results on split samples. Five MC were detected in Ostrich Bay sediment samples, with picric acid detected in eight of the 35 samples that were tested for toxicity, picramic acid also detected in eight of the samples, 2,4-dinitrophenol in 14 of the samples, nitrocellulose in 15 of the samples, and nitroguanidine in 15 of the samples. Detected organic carbon-normalized concentrations for all five MC were below their respective SQB_{OC} values in all samples (Table 5), and 34 of the 35 sediment samples were tested as not toxic in all three bioassays (i.e., no differences from control and reference area sediment responses). The single location with significant toxicity (OU2-SS-41-P2, mean percent normal development was 83% of reference area samples in the bivalve larval test) was located in the mouth of Ostrich Bay at the northern boundary of the operable unit. The concentrations of the two MC detected in that sample (nitrocellulose and nitroguanidine) were within the ranges of concentrations in non-toxic samples, the organic carbon-normalized concentration of nitroguanidine was below its lowest SQB_{OC} and was within the range of concentrations detected in two of the reference area locations, and no other detected chemicals or detection limits of undetected chemicals exceeded Washington State sediment quality standards (Ecology, 2001). With the lack of sediment quality guideline exceedances, the cause of the sediment toxicity in OU2-SS-41-P2 is uncertain, but evidence suggests that it is unrelated to the presence of MC. Together, these results are indicative of the protectiveness and applicability of the preliminary SQBs for screening benthic invertebrate risks from MC contamination of sediments.

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