



## Canadian Sediment Quality Guidelines for the Protection of Aquatic Life

## NONYLPHENOL AND ITS ETHOXYLATES

**N**onylphenol (NP) is a term used to refer to a group of isomeric compounds each consisting of a nine-carbon alkyl chain attached to a phenol ring, with the chemical formula  $C_{15}H_{24}O$ . The various isomers can differ both in the degree of alkyl chain branching and in the position on the phenol ring at which the alkyl chain is attached. Most NP produced commercially is in the form of 4-nonylphenol (i.e., with the alkyl chain attached at the *para*-position) with varied alkyl chain branching. Major derivatives of NP are nonylphenol ethoxylates (NPEs) and nonylphenoxy acetic acids (also called nonylphenol ethoxycarboxylates [NPECs]).

NPEs consist of a phenol group attached to both a nine-carbon alkyl chain and an ethoxylate chain. NPEs have the general formula  $C_9H_{19}-C_6H_4O(CH_2CH_2O)_nH$ , where  $n$  may range from 1 to 100, but with most commercially produced NPEs containing between 6 and 12 ethoxylate groups. NPEs have an amphipathic nature, that is, the alkylphenyl moiety is hydrophobic, while the ethoxylate chain is hydrophilic. Most commercial and industrial applications of NPEs utilize this characteristic surfactancy.

NPECs have a chemical structure similar to NPEs, but with a carboxylate group ( $-COOH$ ) on the end of the ethoxylate chain. The chemical formula for NPECs is  $C_9H_{19}-C_6H_4O(CH_2CH_2O)_nCH_2COOH$ , where  $n$  again refers to the number of ethoxylate groups. NPECs are formed during the aerobic biodegradation of NPEs (Ahel et al., 1994b).

NP, NPEs, and NPECs are synthetic compounds that do not occur naturally, hence environmental concentrations can be solely attributed to human activities. The majority of NP in production is used to produce NPEs; however, it is also used as a formulant in pesticides, as a lubricating oil additive, and as a catalyst in the curing of epoxy resins (CIS 1990; Lorenc et al. 1992).

NPEs are present in many consumer products, including laundry detergents, shampoos, cosmetics, household cleaners, latex paints, and spermicides (Metcalf et al. 1996). They have a broad range of industrial uses as surfactants, detergents, wetting agents, dispersants, defoamers, de-inkers, and antistatic agents. Major Canadian industrial users are textile mills and the pulp

and paper industry; other applications include plastics and elastomer manufacturing, oil extraction and production, metal processing, leather processing, industrial and institutional cleaners, paint and protective coatings, and pesticides (Metcalf et al. 1996).

NP and its ethoxylates may potentially enter the environment during production, use, storage, transportation, or through accidental spillage. They are primarily released to the environment through the discharge of municipal and industrial wastewaters into surface waters. Pathways to the terrestrial environment include the spraying of pesticides that contain NP or NPEs as formulants, landfilling of sludges, or the application of sewage sludge or pulp and paper mill sludge to agricultural soils. Runoff from these terrestrial sources is another pathway to aquatic systems.

Once NP and NPEs have entered aquatic systems, they may be adsorbed to suspended sediment particles in the water column that may eventually settle out, or they may be directly adsorbed to sediment particles at the surface of the sediment–water interface. Short-chain NPEs and NP are more hydrophobic than NPECs and longer-chain NPEs and therefore have a higher tendency to adsorb to sediments. This may involve both hydrophobic interactions with the organic matter in the sediment and hydrophilic interactions with the mineral components (John et al. 2000).

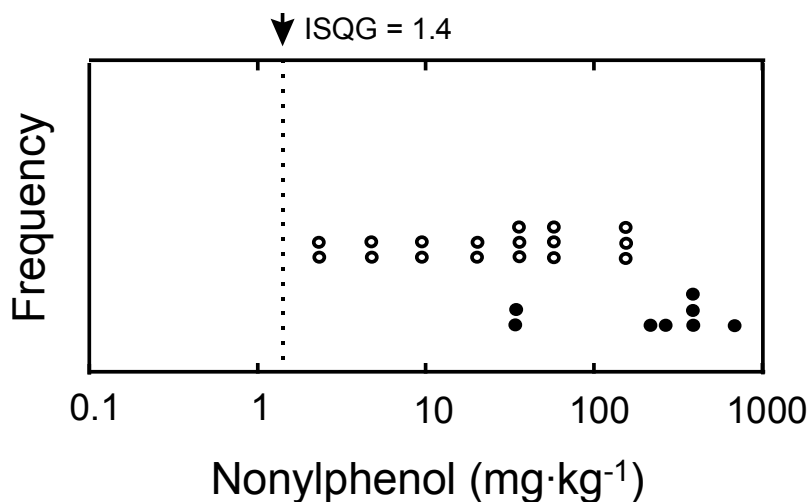
Partitioning of NPEs between the water and sediment phases and achievement of adsorption equilibrium occur rapidly under artificial and mesocosm conditions (Heinis et al. 1999; John et al. 2000). Nonylphenol may persist in sediments for many months. For example, in a freshwater mesocosm study, the half-life for dissipation of nonylphenol from sediments was estimated at 66 d, and the time required for dissipation of 95% of the NP was

**Table 1. Interim sediment quality guidelines (ISQGs) for nonylphenol and its ethoxylates ( $mg \cdot kg^{-1} dw^*$ ).**

	Freshwater	Marine/estuarine
ISQG	1.4 <sup>†</sup>	1.0 <sup>†</sup>

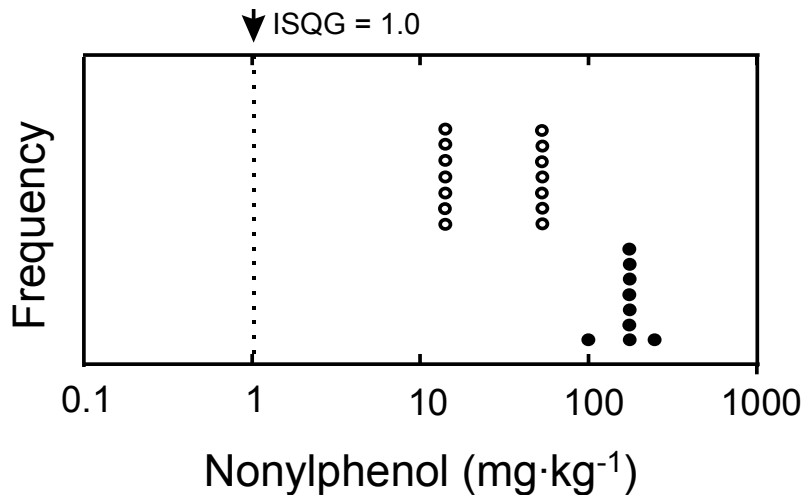
\* Expressed on a TEQ basis using NP TEFs; assumes 1% TOC.

† Provisional; use of equilibrium partitioning approach.



**Figure 1. Distribution of nonylphenol concentrations in freshwater sediments that are associated with adverse biological effects (●) and no adverse effects (○).**

The provisionally recommended Canadian freshwater interim sediment quality guideline (ISQG) is indicated by the dotted line.



**Figure 2. Distribution of nonylphenol concentrations in marine and estuarine sediments that are associated with adverse biological effects (●) and no adverse effects (○).**

The provisionally recommended Canadian marine interim sediment quality guideline (ISQG) is indicated by the dotted line.

estimated at 401 d (Heinis et al. 1999). NPEs (with 1–14 ethoxylate units) applied to estuarine intertidal plots as part of a herbicide formulation showed decreasing concentrations in the sediments during the first 14 d posttreatment, but then stabilized and remained constant even after 119 d (Paveglio et al. 1996). This indicates that regular NP inputs to a site will result in the accumulation of NP over time.

Biodegradation is the most important process affecting the fate of NP and NPEs (Ahel et al. 1994b). NPEs undergo a two-stage degradation process. First, step-wise removal of ethoxy groups results in the formation of intermediate biodegradation products (short-chain NPEs and NPECs, and NP). The second stage involves the ultimate conversion of the molecules to CO<sub>2</sub>, H<sub>2</sub>O, and inorganic salts (Swisher 1987). The intermediate degradation products are more biorefractory, that is, they are less readily biodegraded than the parent NPEs, and therefore ultimate biodegradation occurs more slowly (Ahel et al. 1994a). Rates of biodegradation increase with increasing temperature (Ahel et al. 1994b; Manzano et al. 1999).

Nonylphenolic substances in the sediment are taken up by benthic organisms. NP and NPEs with 1–3 ethoxylate groups show mild bioaccumulation in aquatic organisms, with the majority of calculated bioaccumulation factors (BAFs) and bioconcentration factors (BCFs) ranging between 30 and 1 000 (McLeese et al. 1981; Granmo et al. 1991; Ward and Boeri 1991; Ahel et al. 1993; Brooke 1993; Lewis and Lech 1996). In general, BAFs decrease with increasing ethoxylation. There is no evidence to suggest that NP or NPEs biomagnify in the food chain.

Canadian provisional interim sediment quality guidelines (ISQGs) for nonylphenol and its ethoxylates in freshwater and marine sediments were developed using an equilibrium partitioning (EqP) approach (Table 1). Provisional ISQGs of 1.4 and 1.0 mg·kg<sup>-1</sup> dw, expressed in nonylphenol toxic equivalent units (NP TEQs) and normalized to 1% total organic carbon (TOC), are recommended for the protection of freshwater and marine life, respectively. The ISQGs refer to total concentrations of nonylphenol and nonylphenol equivalents in surficial sediments (i.e., top 5 cm) on a dry weight basis, as quantified by extraction using standard analytical methods (e.g., soxhlet extraction, steam distillation, or supercritical fluid extraction) followed by detection with high pressure liquid chromatography or gas chromatography/mass spectrometry. The ISQGs represent

levels of NP and NPEs in sediments below which adverse biological effects are not expected to occur.

Because of insufficient data, freshwater and marine ISQGs could not be developed for NP and its ethoxylates using any of the three approaches described in the protocol (i.e., the modified National Status and Trends Program approach, the spiked-sediment toxicity test approach, or the adoption of guidelines from other jurisdictions) (CCME 1995). Justification for use of the EqP approach is provided in Environment Canada (2002).

Through the EqP approach, provisional ISQGs were derived from the Canadian water quality guidelines (WQGs) for nonylphenol and its ethoxylates using the following equation (Di Toro et al. 1991; MacDonald et al. 1992):

$$\text{ISQG} = \text{WQG} \cdot K_p \quad (1)$$

where

WQG = water quality guideline (µg·L<sup>-1</sup>)

K<sub>p</sub> = sediment/water partition coefficient (L·kg<sup>-1</sup>)

The TOC content of the sediments is a major factor controlling the partitioning between water and sediment of alkylphenols (Johnson et al. 1998; Hecht et al. 2000) and short-chain NPEs (Urano et al. 1984; John et al. 2000). The sediment/water partition coefficient can therefore be related to the organic carbon/water partition coefficient by the following equation (Di Toro et al. 1991):

$$K_p = K_{oc} \cdot f_{oc} \quad (2)$$

where

K<sub>oc</sub> = organic carbon/water partition coefficient (L·kg<sub>oc</sub><sup>-1</sup>)

f<sub>oc</sub> = fraction of organic carbon

Therefore, by substituting equation (2) into equation (1), the ISQG may be calculated as follows:

$$\text{ISQG} = \text{WQG} \cdot K_{oc} \cdot f_{oc} \quad (3)$$

A K<sub>oc</sub> for NP of 141 254 L·kg<sub>oc</sub><sup>-1</sup> (i.e., the geometric mean of estimated log K<sub>oc</sub> values) (Sekela et al. 1999) and a WQG for NP of 1.0 µg·L<sup>-1</sup> (Environment Canada 2002) yields a freshwater ISQG of 141 mg NP per kilogram of organic carbon. The ISQG was normalized to 1% TOC (i.e., f<sub>oc</sub> = 0.01) resulting in a freshwater ISQG of 1.4 mg·kg<sup>-1</sup> dw.

Using the marine Canadian WQG of  $0.7 \mu\text{g}\cdot\text{L}^{-1}$  (Environment Canada 2002), an ISQG of 99 mg NP per kilogram of organic carbon was derived for marine sediments. This value was normalized to 1% TOC to give a marine ISQG of  $1.0 \text{ mg}\cdot\text{kg}^{-1} \text{ dw}$ .

It is recommended that these guidelines be adjusted based on local levels of TOC to obtain site-specific objectives. This is discussed in more detail under Additional Considerations.

## Toxicity

Field studies that evaluated the effects of sediment-associated nonylphenol or nonylphenol ethoxylates on freshwater and marine organisms were not available. However, a number of spiked-sediment toxicity tests have been conducted to evaluate the effects of sediment-associated nonylphenol in freshwater and marine ecosystems (Environment Canada 2002, Appendixes IIIa and IIIb).

The toxicity of NP in sediment has been determined for a single freshwater invertebrate species, larvae of the midge *Chironomus tentans*, over a 14-d exposure period (England and Bussard 1993). Adverse biological effects included increased mortality and reduced growth, with a LOEC of  $34.2 \text{ mg}\cdot\text{kg}^{-1} \text{ (dw)}$  reported for both. Another freshwater spiked-sediment toxicity test was conducted by Ward and Boeri (1992), who examined the toxicity of NP to tadpoles of the bullfrog *Rana catesbiana* over a 30-d exposure period. Reported endpoints for mortality included a NOEC of  $155 \text{ mg}\cdot\text{kg}^{-1}$  and a LOEC of  $390 \text{ mg}\cdot\text{kg}^{-1}$ , with a calculated  $\text{LC}_{50}$  of  $260 \text{ mg}\cdot\text{kg}^{-1}$ . The authors also examined sublethal endpoints such as growth, lethargy, hemorrhaging, and occurrence of tail lesions. For all of these sublethal effects, the NOECs were  $155 \text{ mg}\cdot\text{kg}^{-1}$ , the LOECs were  $390 \text{ mg}\cdot\text{kg}^{-1}$ , and the  $\text{EC}_{50}$ s were calculated at  $220 \text{ mg}\cdot\text{kg}^{-1}$  (Ward and Boeri 1992).

Two spiked-sediment studies have examined the toxicity of NP to marine invertebrates. One study evaluated NP lethality to the amphipod *Ampelisca abdita* (Fay et al. 2000). A 10-d  $\text{LC}_{50}$  of  $98.7 \text{ mg}\cdot\text{kg}^{-1}$  was observed, with 100% mortality occurring at a concentration of  $243 \text{ mg}\cdot\text{kg}^{-1}$ . The other study evaluated sublethal effects of NP on the polychaete *Capitella* sp. I over a 78-d

exposure period (Hansen et al. 1999). Sublethal endpoints examined included time to first reproduction, time between reproductive events, number of broods per reproductive individual, number of eggs per individual and per reproductive individual, population growth rate, and size at maturity. For all of these endpoints, the NOEC was  $52 \text{ mg}\cdot\text{kg}^{-1}$  and the LOEC was  $174 \text{ mg}\cdot\text{kg}^{-1}$  (Hansen et al. 1999). The results of this study, however, should be treated with caution because standard sediment-spiking procedures were not followed.

A comparison of the ISQGs with the available sediment toxicity data indicates that these concentrations are likely to be protective of sediment-dwelling organisms (Figures 1 and 2). For example, the lowest effect concentrations observed in freshwater and marine sediment toxicity tests with nonylphenol were  $34.2 \text{ mg}\cdot\text{kg}^{-1}$  and  $174 \text{ mg}\cdot\text{kg}^{-1}$ , respectively, as previously discussed. The guidelines are lower than these effect concentrations and are therefore considered protective.

## Toxic Equivalency Factors

NP, NPEs, and NPECs typically occur together in the aquatic environment as mixtures. Therefore, for the purpose of assessing acceptable levels of these substances in sediment, it is more useful to develop guidelines that consider their combined effects. This can be accomplished through the use of a toxic equivalency (TEQ) approach. The guidelines in this fact sheet were developed based on toxicity data for NP, however, they should also be applied to NPEs and NPECs through the use of the TEQ approach.

The TEQ approach is used to characterize the toxicity of a mixture of related compounds by expressing the toxicities of each individual compound in common terms and summing them. Application of the TEQ approach requires that all components of a mixture have a common mode of toxicity such that their effects are additive. Nonylphenolic substances meet this requirement. At typical environmental concentrations and for conventional toxicity endpoints, the mode of toxicity for NP and for at least the lower ethoxylate chain length NPEs and NPECs is narcosis (Schüürmann 1991; Fay et al. 2000; Servos et al. 2000). Additivity of their effects (as opposed to synergism or antagonism) is therefore most likely (Servos et al. 2000).

Relative toxicity values, or NP toxic equivalency factors (TEFs), have been estimated for various nonylphenolic compounds (Table 2). The total concentration of NP and NP equivalents in a sample can be calculated using the following equation:

$$\text{Total TEQ} = \sum_{i=1}^n (C_i \times \text{TEF}_i)$$

where

TEQ = concentration of the mixture of nonylphenolic compounds expressed as toxic equivalent of NP

n = number of nonylphenolic compounds

i = 1, 2, 3, ..., n

C<sub>i</sub> = concentration of compound i

TEF<sub>i</sub> = toxic equivalency factor for the compound i (unitless)

The application of the TEQ approach during the assessment of sediment quality requires expressing sediment concentrations of NPEs and NPECs in terms of NP equivalents. The sum of these concentrations are then compared with the ISQGs. If octylphenol (OP), octylphenol ethoxylates (OPEs), or octylphenol ethoxycarboxylates (OPECs) are present in sediments, it is recommended that these substances also be considered in the calculation of total NP equivalents. OP and OPEs are very similar in structure and behaviour to NP and NPEs and likely act through a similar mode of action (Servos et al. 2000).

## Concentrations

Concentration data for NP and NPEs in Canadian freshwater and marine sediments only exist for the Great Lakes–St. Lawrence system and locations in British Columbia (Environment Canada 2002). Most of the sediments that have been analyzed for NP and NPEs were collected from locations near wastewater treatment plant outfalls or industrialized areas. Therefore, the data reported here likely represent the upper range of concentrations present in Canadian sediments.

Reported freshwater bed sediment concentrations of NP range from below detection limits to 110 mg·kg<sup>-1</sup> on a dry weight basis (Lee and Peart 1995; Bennie et al. 1997; Brewer et al. 1999; Bennett and Metcalfe 1998, 2000; Sabik et al. 2000). Sekela et al. (1999) reported NP concentrations in suspended solids of the Fraser River, British Columbia, at 0.021–0.087 mg·kg<sup>-1</sup> dw. Canadian freshwater bed sediment concentrations of NP1EO and NP2EO fell within the ranges of <0.015–75 mg·kg<sup>-1</sup> and

**Table 2. Toxic equivalency factors (TEFs) for NP, NPEs, NPECs, OP, OPEs, and OPECs (Servos et al. 2000; Environment Canada 2002).**

Chemical	TEFs (relative to NP)
NP	1
NP <sub>n</sub> EO (1 ≤ n ≤ 8)	0.5
NP <sub>n</sub> EO (n ≥ 9)	0.005
NP1EC	0.005
NP2EC	0.005
OP	1
OP <sub>n</sub> EO (1 ≤ n ≤ 8)	0.5
OP <sub>n</sub> EO (n ≥ 9)	0.005
OP1EC	0.005
OP2EC	0.005

<0.015–18.0 mg·kg<sup>-1</sup> dw, respectively (Bennie et al. 1997; Bennett and Metcalfe 2000; Sabik et al. 2000).

Concentrations of NP in surficial marine sediments from the Strait of Georgia, British Columbia, ranged from 0.122 to 0.541 mg·kg<sup>-1</sup> dw (mean = 0.273 mg·kg<sup>-1</sup>; n = 21) (Shang et al. 1999; R.W. MacDonald and M.G. Ikononou 2001, Institute of Ocean Sciences, Sidney, B.C., pers. com. [concerning unpublished raw data used in Shang et al 1999]). Surficial marine sediment concentrations of NP1EO and NP2EO were measured at 0.065–0.558 mg·kg<sup>-1</sup> dw (mean = 0.279 mg·kg<sup>-1</sup>; n = 21) and 0.034–0.198 mg·kg<sup>-1</sup> dw (mean = 0.069 mg·kg<sup>-1</sup>; n = 21), respectively. Total concentrations of NP<sub>n</sub>EO (3 ≤ n ≤ 19) were measured at 0.29–1.42 mg·kg<sup>-1</sup> dw (mean = 0.78 mg·kg<sup>-1</sup>, n = 21). Sediment cores from these same marine sites did not show significant differences with depth in either the total amount of NP<sub>n</sub>EO, or the profile of relative concentrations for individual NP<sub>n</sub>EO ethoxamers (Shang et al. 1999).

## Additional Considerations

Regardless of the origin of NP and its ethoxylates in sediments, aquatic organisms may be adversely affected by exposure to elevated levels. However, the occurrence of adverse biological effects from exposure to NP and NPEs cannot be precisely predicted from concentration data alone. The likelihood of adverse biological effects occurring at a particular site depends on the sensitivity of individual species, the endpoints examined, as well as a variety of physicochemical (e.g., temperature), geochemical (e.g., TOC and particle size), and biological

(e.g., feeding behaviour and life stage) factors that affect the bioavailability of NP and NPEs (Environment Canada 2002). With the exception of TOC, few studies have examined the effects of these factors on the bioavailability of NP and NPEs, though they are expected to play a role.

Physicochemical characteristics of NP and NPEs themselves, such as octanol–water partition coefficients ( $K_{ow}$ ) and water solubility, also have the potential to influence their bioavailability to aquatic organisms. Reported estimates of the log  $K_{ow}$  for NP and NPEs generally fall between 4.1 and 4.4 (McLeese et al. 1981; Barber et al. 1988; Freij 1991; Wild and Jones 1992; Ahel and Giger 1993b). Water solubility of NP is  $5.43 \text{ mg}\cdot\text{L}^{-1}$  (at  $20.5^\circ\text{C}$ ), and solubility of NPEs generally increases with increasing ethoxylation (Ahel and Giger 1993a; Hall et al. 1989).

As previously mentioned, TOC is an important factor influencing the bioavailability of NP and NPEs. Hence, the ISQGs should be adjusted for site-specific levels of TOC. The provisional ISQGs in Table 1 were normalized to 1% TOC ( $f_{oc} = 0.01$ ). In jurisdictions where the percentage of organic carbon in sediments is known to be consistently higher or lower than 1%, local estimates of TOC may be used to set site-specific sediment objectives. This is done by multiplying the ISQG by the percentage of organic carbon. For example, for freshwater sediments with 2% TOC, the freshwater ISQG of  $1.4 \text{ mg}\cdot\text{kg}^{-1}$  would be multiplied by 2 to obtain a site-specific objective of  $2.8 \text{ mg}\cdot\text{kg}^{-1}$ . With lower levels of organic carbon there will be less binding of contaminants to the sediments and consequently greater bioavailability. Therefore, it is important that the  $f_{oc}$  value used be based on the lowest sediment organic carbon concentration that might possibly occur, such that all aquatic life in the designated jurisdiction will be protected. It is not recommended that  $f_{oc}$  values outside of the range 0.002–0.12 be used in deriving site-specific objectives. For sediments that contain <0.2% or >12% TOC, other factors that the EqP method does not account for may influence the contaminant partitioning (USEPA Science Advisory Board 1992). Further generic guidance regarding the implementation of Canadian sediment quality guidelines (SQGs), the interpretation of sediment chemistry in comparison to ISQGs, and other sediment assessment tools is provided elsewhere (CCME 1995).

It should be noted that because these guidelines were derived using an EqP approach, they represent sediment concentrations that will be protective of sediment-dwelling biota when the sediment and interstitial water

are in equilibrium. Their use requires the assumptions that there are no changes in the thermodynamics of the system (i.e., constant temperature and pressure with no input/output of energy or mass), and that there has been sufficient time for the NP and/or NPEs to distribute throughout the system (Spacie et al. 1995). It is possible that nonequilibrium conditions could exist in some cases, such as immediately following a spill, in river channels where the sediments are unstable, or in active dredge disposal areas (NYSDEC 1999). The USEPA Science Advisory Board (1992) cautions against the use of guidelines based on equilibrium partitioning in areas of rapid sediment deposition or erosion. Where partitioning of nonylphenolic compounds between the sediment and interstitial water is not in equilibrium, it is possible that the sediment guidelines may not be protective against adverse effects to benthos.

The ISQGs recommended for NP and NPEs reflect the current state of knowledge regarding the concentrations, environmental fate, and biological effects of sediment-associated NP and NPEs. These ISQGs provide reference points that can be used in conjunction with other scientific tools to evaluate the potential for adverse biological effects of NP and NPEs in sediments. In addition, these guidelines provide interim targets that can be used in the risk management of these substances or in the remediation of impacted sediments. In the above context, it is anticipated that the recommended ISQGs for NP and NPEs will be useful in assessing the ecotoxicological significance of NP and NPEs in sediments. Although these ISQGs are considered provisional at this time, they should not be used differently than they would be if they were full SQGs. The revision and re-assessment of these provisional ISQGs will be possible when sufficient data become available to support the use of the modified NSTP or SSTT approaches as described in the formal protocol (CCME 1995).

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