

NONYLPHENOL AND ITS ETHOXYLATES 2002

his fact sheet provides Canadian soil quality guidelines for nonylphenol and its ethoxylates for the protection of environmental and human health (Table 1). A supporting scientific document is also available (Environment Canada 2002).

Background Information

Nonylphenol (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. The CAS registry number for a mixture of various branched isomers of NP is 84852-15-3. NP is a viscous liquid with

a molecular weight of 220.3, a water solubility of 5.43 mg·L⁻¹ at 20.5°C, a vapour pressure of 4.55 mPa at 25°C, and a Henry's law constant of 11.02 Pa·m³·mol⁻¹ (Reed 1978; Romano 1991; Ahel and Giger 1993; European Commission 1999). Synonyms for NP include nonyl phenol and isononylphenol. 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.

Table 1. Soil quality guidelines for nonylphenol and its ethoxylates (mg TEQ·kg⁻¹).

	Land use			
	Agricultural	Residential/ parkland	Commercial	Industrial
Guideline	5.7 ^a	5.7 ^a	14 ^a	14 ^a
SQG_{HH}	NC ^b	NC ^b	NC ^b	NC ^b
Limiting pathway for SQG_{HH}	ND	ND	ND	ND
Provisional SQG_{HH}	NC ^c	NC ^c	NC ^c	NC ^c
Limiting pathway for provisional SQG_{HH}	ND	ND	ND	ND
SQG _E	5.7	5.7	14	14
Limiting pathway for SQG _E	Soil contact	Soil contact	Soil contact	Soil contact
Provisional SQG_E	NC ^d	NC ^d	NC ^d	NC ^d
Limiting pathway for provisional SQG_E	ND	ND	ND	ND
Interim soil quality criterion (CCME 1991)	No value	No value	No value	No value

Notes: NC = not calculated; ND = not determined; $SQG_E = soil$ quality guideline for environmental health; $SQG_{HH} = soil$ quality guideline for human health.

^aData are sufficient and adequate to calculate only an SQG_E. An interim soil quality criterion (CCME 1991) was not established for these substances, therefore, the SQG_E becomes the soil quality guideline.

^bThere is no SQG_{HH} for this land use at this time.

^CThere is no provisional SQG_{HH} for this land use at this time.

^dBecause data are sufficient and adequate to calculate an SQG_E for this land use, a provisional SQG_E is not calculated.

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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. Therefore, 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). In the form of tris(4-nonylphenyl) phosphite, it may be used as an antioxidant in the stabilization of rubber, vinyl polymers, polyolefins, and styrenics (Lorenc et al. 1992).

NPEs are present in many consumer products, including laundry detergents, shampoos, cosmetics, household cleaners, latex paints, and spermicides (Metcalfe 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 (Metcalfe et al. 1996).

NP and its ethoxylates may potentially enter the environment during production, use, storage, or 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.

Only one study has looked at Canadian soil concentrations of nonylphenolic substances. In that study, soil from a single site that had been amended with sludge was found to contain 2.72 mg·kg⁻¹ of NP, <0.538 mg·kg⁻¹ each of NP1EC and NP2EC, and only trace amounts of NP1EO, NP2EO, and NP*n*EO (Water Technology International Corporation 1998).

Environmental Fate and Behaviour in Soil

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 CO₂, H₂O, 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).

Ultimate biodegradation of NPEs has been observed in soil under aerobic conditions. For example, during a 9-week experimental period, 57% of the initially applied NP9EO was mineralized, including breakdown of the benzene ring (Hughes et al. 1996). Mineralization of NP has also been observed in a variety of soil types, including agricultural soils of various textures, noncultivated temperate soils, and soils from the Canadian tundra (Topp and Starratt 2000). The fact that biodegradation occurred in the arctic soils indicates that the microorganisms already possessed the biochemistry for NP metabolism without prior exposure and adaptation. The authors suggested that exposure of microorganisms to phenolic substances released by the decomposition of plant matter may have enabled them to degrade NP (Topp and Starratt 2000).

Mineralization of NP by soil bacteria can be inhibited by either an overabundance or a lack of water content (Topp and Starratt 2000). Temperature has also been observed to affect rates of NP mineralization in soil. Although biodegradation will occur over the temperature range of 4 to 30°C, at lower temperatures the rate decreases and there is a greater lag period (Topp and Starratt 2000). Overall, the authors conclude that NP in land-applied sewage sludge would readily undergo biodegradation under the soil conditions of a normal Canadian growing season.

NPEs with longer ethoxylate chains are nonpersistent in the environment and undergo a rapid primary biodegradation. However, there are conflicting reports on the persistence of their degradation products in soil. Some researchers report that under aerobic conditions, nonylphenolic compounds are not persistent and degradation occurs readily (Wild and Jones 1992;

Maguire 1999). For example, concentrations of NP introduced into agricultural soils through sludge application decreased by 90% in 10 d (Kirchmann et al. 1991) and by 92% in 3 months (Dierexsens and Tarradellas 1987). In contrast, there is also evidence of persistence with complete degradation taking considerably longer. Researchers have observed a biphasic mineralization of NP, NP1EO, and NP2EO in agricultural soil (Marcomini et al. 1988; Topp and Starratt 2000). In the initial phase, lasting 10 d, rapid mineralization occurred, with >30% of the NP converted to carbon dioxide (Topp and Starratt 2000). This was followed by a much slower second phase such that low concentrations of the nonylphenolic compounds were still present after 360 d (Marcomini et al. 1988). Under anaerobic conditions, such as in landfills, it appears that nonylphenolic compounds are persistent in soil (Maguire 1999). For example, in a survey of several German sludge-only landfills, concentrations of NP and NP1EO of 4 and 19 mg·kg⁻¹ dw, respectively, were detected in samples of sludge that had been landfilled 30 years previously (Marcomini et al. 1989).

In addition to biodegradation, other processes may also influence the fate and behaviour of NP and NPEs. The abiotic fate processes affecting NP and NPEs in soils, in decreasing order of importance, include particle adsorption, infiltration to groundwater, and volatilization.

Based on physical/chemical properties, Wild and Jones (1992) speculated that NP in sludge applied to agricultural soils would likely have high adsorption to soil particles. Various NPEs have also exhibited high adsorption on soil (Beigel et al. 1998). The relative degree of adsorption among NPE oligomers appears to increase with decreasing number of ethoxylate units, to a maximum soil affinity at about NP9EO, after which the tendency for adsorption decreases with decreasing number of ethoxylate units (Beigel et al. 1998). It is suggested that adsorption increases due to increasing hydrophobicity, but with the shorter ethoxylate chains, that have a lower critical micelle concentration, there may be a preference for surfactant-surfactant interactions and micelle formation, rather than surfactant-soil surface interactions (Beigel et al. 1998).

Although the high adsorption of nonylphenolic compounds to soil particles suggests that there would be a low probability of leaching (Wild and Jones 1992), numerous studies have shown evidence of infiltration of nonylphenolic compounds into groundwater from municipal landfills (Ahel 1991) from contaminated rivers and wastewater canals (Zoller et al. 1990; Ahel 1991;

Ahel et al. 1996) and due to sewage disposal through rapid infiltration (Barber et al. 1988; Rudel et al. 1998). Because of their higher water solubility and mobility, NPECs are thought to pose a greater risk as groundwater contaminants than other nonylphenolic compounds (Ahel et al. 1996). Although NP, NP1EO, and NP2EO are more effectively retained in the soil, significant concentrations of these substances have been measured in groundwater at distances >100 m from the original source (Ahel 1991; Ahel et al. 1996).

NP is not likely to volatilize from soils in significant quantities. For example, of the initial 1000 mg·kg⁻¹ NP that was spiked into a compost–sandstone mixture, only 0.22% was lost through volatilization over a period of 40 d (Trocmé et al. 1988).

Behaviour and Effects in Biota

The mode of toxicity for NP and at least the lower chain length NPEs and NPECs is likely narcosis (Schüürmann 1991; Fay et al. 2000; Servos et al. 2000). Toxicity from longer chain length NPEs may be due to physical surfactancy, particularly at high concentrations above the critical micelle concentration. Nonylphenolic substances may also act as estrogen agonists to disrupt endocrine functions (White et al. 1994).

Soil Microbial Processes

Observations of both decreased and increased respiration of soil microorganisms (as measured through CO₂ evolution) due to NP exposure have been documented. For example, in a 40-d test, Trocmé et al. (1988) observed no effect of CO₂ evolution when NP was present in the soil at a concentration of 100 mg·kg⁻¹, but at 1000 mg·kg⁻¹ there was a significant reduction in CO2 evolution of 64%. In a 3-month assay, Kirchmann et al. (1991) observed no effect on CO2 evolution at an NP concentration of 10 mg·kg⁻¹. At 500 mg·kg⁻¹, Kirchmann et al. (1991) did see an effect, but in contrast with the results of Trocmé et al. (1988), the effect was an increase in CO₂ evolution by 26%. Kirchmann et al. (1991) also examined effects of NP on the nitrogen-cycling of microorganisms. No effects on nitrification were observed at an NP concentration of 10 mg·kg⁻¹. With 500 mg·kg⁻¹, no effect on nitrogen mineralization occurred, but a 35% decrease in nitrification was observed.

Terrestrial Plants

Based on the physical and chemical properties of NP, Wild and Jones (1992) predicted a high retention of NP at root surfaces, with only moderate uptake and translocation occurring within plants. This theory is supported by studies that found no evidence of increased NP concentrations in wheat grains (Kirchmann and Tengsved 1991) or tomato shoots (Harms 1992) when roots of the plants were exposed to NP. In contrast, translocation of NP or its metabolites from roots to shoots within wheat, tomato, and garden orache plants has been observed by other researchers (Bokern et al. 1998). Uptake and metabolism of NP have also been observed to occur in cell suspension cultures of over a dozen crop species (Bokern et al. 1996: Bokern and Harms 1997). Metabolites were ß-glucosides of differently hydroxylated NP, some of which were further acylated with glucuronic acid or malonic acid (Bokern and Harms 1997). Although most plant species readily take up NP (when exposed in vitro) and metabolize it, no mineralization of NP to CO₂ appears to occur (Bokern et al. 1998).

NP toxicity tests using soil (as opposed to culture medium) have been conducted for five crop species. For turnip, *Brassica rapa*, a 14-d EC₅₀ for growth reduction of 10 mg·kg⁻¹ was calculated (Günther and Pestemer 1992). Other reported endpoints are one to two orders of magnitude higher. In 21-d tests with NP, soybeans (*Glycine max*), sunflowers (*Helianthrus rodeo*), and sorghum (*Sorghum bicolor*) all showed no growth effects at 100 mg·kg⁻¹, but had EC₅₀s for growth reduction at 1000 mg·kg⁻¹ (Windeatt and Tapp 1986). EC₅₀s for growth reduction have also been reported for lettuce (*Lactuca sativa*) at 559 mg·kg⁻¹ after 7 d of exposure, and at 625 mg·kg⁻¹ after 14 d of exposure (Hulzebos et al. 1993).

Terrestrial Invertebrates

Toxicity tests with NP have been conducted for three soil-dwelling invertebrates. From a 28-d exposure of the springtail *Folsomia candida*, Madsen et al. (1998) reported that the EC₅₀ for reproduction falls between 16 and 30 mg·kg⁻¹, and the LC₅₀ is >30 mg·kg⁻¹. Researchers have also studied survival and reproduction in another species of springtail, *Folsomia fimetaria*. In 21-d exposures with this second species, Krogh et al. (1996) reported an LC₁₀ of 75.1 mg·kg⁻¹ and an LC₅₀ of 151.3 mg·kg⁻¹. For reproduction in *F. fimetaria*, Krogh et al. (1996) reported a 21-d EC₁₀ of 23.6 mg·kg⁻¹, while M. Holm (N.d., Department of Terrestrial Ecology,

National Environmental Research Institute, Denmark, pers com. [cited in Jensen et al. 1997]) reported EC₁₀s of 27 mg·kg⁻¹ and 48 mg·kg⁻¹. The EC₅₀s for reproduction in F. fimetaria were reported at 65.5 mg·kg⁻¹ (Krogh et al. 1996) and 39 mg·kg⁻¹ and 59 mg·kg⁻¹ (M. Holm N.d., Terrestrial Department of Ecology, Environmental Research Institute, Denmark, pers com. [cited in Jensen et al. 1997]). The 21-d LC₅₀ for NP in the earthworm Apporectodea calignosa occurred at >40 mg·kg⁻¹ (Krogh et al. 1996). The EC₅₀ for growth reduction was 23.9 mg·kg⁻¹. The authors calculated the EC₁₀ and EC₅₀ for effects on reproduction as 3.44 mg·kg⁻¹ and 13.7 mg·kg⁻¹, respectively (Krogh et al. 1996).

Experimental Animals

The only mammals that have been tested for NP toxicity ingestion are laboratory through rats. multigenerational study, Chapin et al. (1999) examined the effects of NP administered through dosed food on Sprague Dawley rats (Rattus norvegicus). At a diet concentration of 650 mg·kg⁻¹ (i.e., a dose of 30-108 mg·kg⁻¹ bw), vaginal opening at an earlier age was observed in the F1 generation. Significant effects observed at a diet concentration of 2000 mg·kg⁻¹ (i.e., a dose of 100-360 mg·kg⁻¹ bw) included increased relative kidney weights and decreased adult ovary weights in the F1 generation and increased estrous cycle length in both the F1 and F2 generations. Laws et al. (2000) reported LOECs for increased uterine weight and earlier vaginal opening both at a diet concentration of 50 mg·kg⁻¹, and disruption of estrous cyclicity at a diet concentration of 100 mg·kg⁻¹. A LOEC of 75 mg·kg⁻¹ bw per day for increased uterine weight in ovariectomized female rats has been reported by Odum et al. (1999).

Toxic Equivalency Factors

NP, NPEs, and NPECs typically occur together in the environment as mixtures. Therefore, for the purpose of assessing acceptable levels of these substances in soil, 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 TEO approach requires that all components of a mixture have a common mode of toxicity such that their effects are additive. Nonylphenolic meet this requirement. At substances 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:

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

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_i = concentration of compound i

TEF = toxic equivalency factor for the compound i (unitless).

The application of the TEQ approach during the assessment of soil quality requires expressing soil concentrations of NPEs and NPECs in terms of NP equivalents. The sum of these concentrations are then compared with the guidelines developed in this document. If octylphenol (OP), octylphenol ethoxylates (OPEs), or octylphenol ethoxycarboxylates (OPECs) are present in soils, 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). It should be noted, however, that these NP TEQs are not to be confused with the TEQs used for dioxins, furans, and PCBs. They are based on different modes of toxicity and therefore cannot be combined.

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

Chemical	TEFs (relative to NP)
NP	1
NP n EO $(1 \le n \le 8)$	0.5
NP n EO ($n \ge 9$)	0.005
NP1EC	0.005
NP2EC	0.005
OP	1
OP n EO $(1 \le n \le 8)$	0.5
OP n EO ($n \ge 9$)	0.005
OP1EC	0.005
OP2EC	0.005

Guideline Derivation

Canadian soil quality guidelines are derived for different land uses following the process outlined in CCME (1996) using different receptors and exposure scenarios for each land use (Table 1). Detailed derivations for NP and NPEs soil quality guidelines are provided in Environment Canada (2002).

Soil Quality Guidelines for Environmental Health

Environmental soil quality guidelines (SQG_Es) are based on soil contact using data from toxicity studies on plants and invertebrates. In the case of agricultural land use, soil and food ingestion toxicity data for mammalian and avian species are included. To provide a broader scope of protection, a nutrient and energy cycling check is calculated. For industrial land use, an off-site migration check is also calculated.

For all land uses, the preliminary soil contact value (also called threshold effects concentration [TEC] or effects concentration low [ECL], depending on the land use) is compared to the nutrient and energy cycling check. If the nutrient and energy cycling check is lower, the geometric mean of the preliminary soil contact value and the nutrient and energy cycling check is calculated as the soil quality guideline for soil contact. If the nutrient and energy cycling check is greater than the preliminary soil contact value, the preliminary soil contact value becomes the soil quality guideline for soil contact.

For agricultural land use, the lower of the soil quality guideline for soil contact and the soil and food ingestion guideline is recommended as the SQG_E.

For the residential/parkland and commercial land uses, the soil quality guideline for soil contact is recommended as the SQG_E .

For industrial land use, the lower of the soil quality guideline for soil contact and the off-site migration check is recommended as the SQG_E.

In the case of NP and NPEs, the recommended SQG_E for all land use categories is based on the soil contact guideline determined using the weight of evidence approach (Table 3).

Soil Quality Guidelines for Human Health

There are no human health guidelines or check values available at this time (Table 3).

Soil Quality Guidelines for NP and NPEs

The soil quality guidelines are usually the lower of the SQG_E and the interim soil quality criteria (CCME 1991). However, since no interim soil quality criteria were established for NP and NPEs, the SQG_Es are the soil quality guidelines for all land uses (Table 1).

These guidelines should be applied to all nonylphenolic (and octylphenolic) substances present in soil, through use of the TEQ approach. Further guidance on using the TEQ approach is provided in Environment Canada (2002). CCME (1996) and Environment Canada (2002) provide guidance on potential modifications to the recommended soil quality guidelines when setting site-specific objectives.

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Table 3. Soil quality guidelines and check values for nonylphenol and its ethoxylates (mg TEQ·kg⁻¹).

	Land use				
	Agricultural	Residential/ parkland	Commercial	Industria	
Guideline	5.7 ^a	5.7 ^a	14 ^a	14 ^a	
Human health guidelines/check values					
SQG_{HH}	NC^b	NC^b	NC^b	NC^{b}	
Soil ingestion guideline	NC^{b}	NC^b	NC^b	NC^{b}	
Inhalation of indoor air check	NC^b	NC^{b}	NC^b	NC^{b}	
Off-site migration check	_	_	_	NC^{b}	
Groundwater check (drinking water)	NC^b	NC^b	NC^b	NC ^b	
Produce, meat, and milk check	NC^b	NC^b	_	_	
Provisional SQG_{HH} Limiting pathway for provisional SQG_{HH}	NC ^b ND	NC ^b ND	NC ^b ND	NC ^b ND	
Environmental health guidelines/check values					
SQG_E	5.7 ^c	5.7 ^c	14 ^c	14 ^c	
Soil contact guideline	5.7	5.7	14	14	
Soil and food ingestion guideline	NC^d	_	_	_	
Nutrient and energy cycling check	NC^d	NC^d	NC^d	NC^d	
Off-site migration check	_	_	_	82	
Groundwater check (aquatic life)	21 ^e	21 ^e	21 ^e	21 ^e	
Provisional SQG_E Limiting pathway for provisional SQG_E	NC ^f ND	NC ^f ND	NC ^f ND	NC ^f ND	
Interim soil quality criterion (CCME 1991)	No value	No value	No value	No value	

Notes: NC = not calculated; ND = not determined; SQG_E = soil quality guideline for environmental health; SQG_{HH} = soil quality guideline for human health. The dash indicates a guideline/check value that is not part of the exposure scenario for this land use and therefore is not calculated.

 $^{^{}a}$ Data are sufficient and adequate to calculate only an SQG_E. An interim soil quality criterion (CCME 1991) was not established for these substances. Therefore the SQG_E becomes the soil quality guideline for this land use.

bThere is no value for this land use at this time.

^cThe SQG_E is based on the soil contact guideline.

dData are insufficient/inadequate to calculate this value for this land use.

^eThe environmental groundwater check (aquatic life) value has not been applied in determining the soil quality guideline. The applicability of the groundwater check (aquatic life) value should be determined on a site-specific basis.

 f_B Because data are sufficient and adequate to calculate an SQG_E for this land use, a provisional SQG_E is not calculated.

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