

BINATIONAL FRAMEWORK FOR IDENTIFYING SUBSTANCES TO BE ADDRESSED IN THE GREAT LAKES BASIN

Test Case: Nonylphenol and its Ethoxylates (NPEs)

I. FEEDERS FOR SUBSTANCE IDENTIFICATION

National Chemical Management Programs¹

Canada

NPEs have been assessed as toxic under the Canadian Environmental Protection Act (CEPA, 1999) and were added to CEPA 1999 Schedule 1- List of Toxic Substances in 2002 (Environment Canada and Health Canada 2001). As a result, two [Pollution Prevention \(P2\) Planning Notices \(Textile Mills that Use Wet Processing and Nonylphenol and its Ethoxylates Contained in Products\)](#) were developed under CEPA 1999 to manage risks associated with NPEs.

United States

EPA Inventory Update Rule (IUR) - EPA requires that chemical manufacturers periodically update production information for compounds listed in the TSCA Chemical Substance Inventory under the IUR.

NP and NPEs are High Production Volume (HPV) chemicals (Emerging Chemicals 2005).

Great Lakes Monitoring and Surveillance

NPEs were not involved in the Great Lakes Screening Project (Muir et al. 2007).²

Other Sources of Information

United States

USEPA Region 5 developed low-level detection analytical methods for NP, NPE1, NPE2 and OP and procured analytical standards for NP1EO and NP2EO, etc. in support of the implementation of the WQC for NP and to support Region 5 monitoring activities.

USEPA Region 5 and the Great Lakes National Program Office (GLNPO) has supported several University and Federal Agency Studies through grants and participated in numerous collaborative sampling efforts to assess the presence of APEs in the region, demonstrate fate and transport of the chemicals, and assess toxicity of the chemicals.

¹ For purposes of the framework analysis, “National Chemical Management Programs” refer to federal programs that assess, manage, and share information on chemicals, including the Chemicals Management Plan (CMP) under the Canadian Environmental Protection Act (CEPA), US EPA’s Chemical Assessment and Management Program (ChAMP), US EPA Inventory Update Rule (IUR), US EPA High Production Volume (HPV) and Medium Production Volume (MPV) programs, etc.

² NPE has a BCF below the threshold criteria used by Muir and Howard to determine bioaccumulation potential (threshold of 1000).

European Union

A risk assessment was completed by the European Union for 4-nonylphenol (branched) and nonylphenol in 2002 and the assessment was presented to the Organization for Economic Co-operation and Development (OECD) as a contribution to Chapter 19, Agenda 21 goals for evaluating chemicals (European Chemicals Bureau 2002).

In 2003, the EU passed a Directive which restricts the marketing and use in Europe of certain products and product formulations that contain more than 0.1% of NPE or NP which came into force January 2005 (European Parliament 2002).

OSPAR (The Convention for the Protection of the Marine Environment of the North-East Atlantic)
Nonylphenol Ethoxylate is on the OSPAR List of Chemicals for Priority Action (2001, updated 2004).

II. CONSIDERATIONS FOR SUBSTANCE SELECTION

Monitoring and Surveillance

Monitoring and surveillance studies are available which provide data on the occurrence of NPEs, and APEs, in the Great Lakes Basin. Examples of such information are provided below. Note that most of the data provided below was conducted prior to implementation of risk management actions (Pollution Prevention Plans) in Canada in 2004.

- USEPA Region 5 & GLNPO are participating in numerous collaborative sampling efforts to assess the presence of APEs in the region.
- Bennie *et al.* 1997, “Occurrence of alkylphenols and alkylphenol mono- and diethoxylates in natural waters of the Laurentian Great Lakes basin and the upper St. Lawrence River”.
 - Reports concentrations in surface water & sediments.
 - Of the surface water samples, 58% were found to contain NPE₁ and 32% contained NPE₂. Their concentrations ranged from <0.020 to 7.8 µg/L for NPE₁ and from <0.020 to 10 µg/L for NPE₂.
 - A total of 66% of sediments had detectable amounts of NPE₁ and concentrations ranged from <0.015 to 38 µg/g (dry weight), 66% of sediments had detectable amounts of NPE₂ and values ranged from <0.015 to 6.0 µg/g (dry weight).
- Bennett and Metcalfe (1998), “Distribution of Alkylphenol Compounds in Great Lakes Sediments, United States and Canada”.
 - Sediment samples were collected from both contaminated and pristine areas of Lakes Huron, Erie, and Ontario and analyzed for alkylphenol compounds. The data indicated that alkylphenols are distributed widely in sediments in the lower Great Lakes. However, concentrations of NP were present at high (mg/g) levels only in sediments near urban and industrialized centers.
- Bennie 1999, “Review of the environmental occurrence of alkylphenols and alkylphenol ethoxylates”.

- Reports concentrations in Canadian and U.S. river, lake and harbour water & sediments.
- Sites in the US indicate nonylphenol levels of less than 0.1 to 0.6 µg/L in river water and from less than 0.003 to 3.0 µg/g (dry weight) in sediments.
- Nonylphenol levels in Canadian river water range from <0.01 to 0.9 µg/L.
- Nonylphenol levels in Canadian river sediments similar to US sediments except samples (containing up to 72 µg/g dry weight) from Hamilton Harbor (Lake Ontario) near the discharge of the Burlington sewage treatment plant.
- Levels of nonylphenol ethoxylates with 3 to 17 ethoxylate units range from less than 1.6 to 15 µg/L in US river water samples.

- Lee *et al.* 2000, “Endocrine-disrupting chemical in industrial wastewater samples in Toronto, Ontario”.
 - Reports concentrations of NP & NPEO from <0.1 to 253 µg/L and <2 to 117,570 µg/L for NPEO in wastewater generated in Toronto (from 97 samples from 40 facilities in 10 different industry classes).

- Bennett and Metcalfe (2000), “Distribution of Degradation Products of Alkylphenol Ethoxylates Near Sewage Treatment Plants in the Lower Great Lakes, North America”.
 - Reports concentrations in sediment samples near sewage treatment plants (Hamilton Harbour & Detroit River).
 - Reports concentrations of APEO degradation products in mussels and semipermeable membrane devices at sites close to sewage treatment plant (STP) discharges (see table below).
 - Data showed that concentrations of these compounds declined rapidly as a function of distance from a point source (primarily waste water treatment plants).

Table 1. Concentrations of alkylphenol ethoxylate (APEO) degradation products and coefficient of variation (% CV) in mussels (lipid normalized; µg/g lipid) and semipermeable membrane devices (µg/g triolein) deployed at sites closest to discharges in the sewage treatment plant(STP) Detroit River (sites 2 and 7) and in Hamilton Harbour

	4-tOP		4-NP		NP1EO		NP2EO	
	Mean	% CV	Mean	% CV	Mean	% CV	Mean	% CV
West Windsor STP								
Outflow Site 2								
Mussel data	3.19	40.3	91.02	11.3	73.4	40.3	21.3	19.7
SPMD data	2.96	*	117.2	*	NA	—	NA	—
Detroit River STP								
Outflow Site 8								
Mussel data	4.92	21.8	134.2	16.1	3.97	16.0	1.09	16.8
SPMD data	0.18	18.2	11.1	4.9	4.39	9.0	2.28	7.3
Burlington STP								
Outflow Site 13								
Mussel data	4.27	12.8	185.6	14.7	2.81	22.0	3.53	21.6
SPMD data	0.52	*	17.95	*	5.93	*	2.25	*

- Environment Canada & Health Canada 2001, “Priority Substance List Assessment Report-Nonylphenol and its Ethoxylates”.
 - Reports concentrations in Canadian freshwater, effluent and sludge from Canadian sewage treatment plants, surface waters & sediments.
 - Report available at: <http://www.ec.gc.ca/substances/ese/eng/psap/final/npe.cfm>.

- Kannon *et al.* (2003) measured NP and NPEs in fish, sediment, and water from the Kalamazoo River.
 - The results showed variable concentrations of NP in fish with only 41% of the fish having detectable concentrations.
 - Detection of NP in sediments and waters was limited to only a few samples with detectable concentrations. NPEs were not detected in any water samples.
- Ying *et al.* (2002) described the environmental fate of alkylphenols and alkylphenol ethoxylates (APEs) in the natural environment.
 - Data showed that wastewater treatment plant discharge is the main source of APEs in the environment.
 - The study also showed that APEs readily degraded to alkylphenol, nonylphenol, and octaphenol, which are persistent in environmental matrices.
- Rice *et al.* (2002) conducted an exploratory study of carp and selected top-predator fish from the Great Lakes and effluent-dominated rivers in Chicago, Detroit, and St. Paul.
 - Carp were found to be excellent accumulators of APEs.
 - Large mouth bass collected from a Detroit river site accumulated only about one-tenth as much total APE as were measured in carp at this site.
 - Carp from the North Branch of the Chicago River had the highest average level of APEs (16 mg/kg fresh wt.).
 - Walleye collected from the St. Paul metropolitan outfall had the next highest average level of APEs (6.1 mg/kg fresh wt.).
 - Average levels of APEs in carp at the other contaminated sites ranged from 1.6 to 2.3 mg/kg fresh weight.
 - Low but detectable levels of APEs were measured in Lake trout obtained from Lake Michigan, near Saugatuck (24 ug/kg-NP only).
- Rice *et al.* (2003) measured alkylphenol and alkylphenol-ethoxylates (manufactured as surfactants in detergents) in the Cuyahoga River in Ohio.
 - General concentration trends showed higher concentrations in the river around urban areas, with the highest concentration detected by a wastewater treatment plant effluent discharge site.
- Sabik *et al.* (2003) analyzed surface waters, municipal effluents, sediment, and mussels from the St Lawrence River for APEs and its degradation products.
 - The results showed that many of the target chemicals were present in all matrices studied: in water, at ppt and ppb levels, and reaching ppm levels in sediments and mussels.
 - Concentrations were significantly higher downstream from the municipal effluent discharge point.
- International Joint Commission, 2006, "Priorities 2003-2005: Priorities and Progress under the Great Lakes Water Quality Agreements".

- Reports concentrations in Great Lakes species (carp, 1998-2000; carp and walleye, 1998-1999; 7 fish species, 1999; herring gull liver); sediments & water, 1998.

CHEMICAL	YEAR	SPECIES OR MEDIUM	LAKE OR LOCATION	CONCENTRATION in parts per billion unless indicated	CITATIONS
NP + NPEOb	98-00	Carp	Cuyahoga & Detroit R.	32 -920	Rice <i>et al.</i> 2003, Schmitz-Afonso <i>et al.</i> 2003
	98-99	Carp and walleye	Near WWTP outlet	4750	Schmitz-Afonso <i>et al.</i> 2003
	1999	7 fish species	Kalamazoo R.	< 3- 29	Keith <i>et al.</i> 2001
		Herring gull liver	Lower Great Lakes	225-464	Grasman <i>et al.</i> in preparation.
	1998	Sediments	Cuyahoga R.	250-1020	Rice <i>et al.</i> 2003
	1998	Sediments	Detroit and Rouge R.	<10-60000	Kannan <i>et al.</i> 2001b
	1998	Water	Cuyahoga R.	0.13 -5.1	Rice <i>et al.</i> 2003

- Mayer *et al.* (2007) analyzed APEs at a Great Lakes marsh land where effluent from a wastewater treatment plant is discharged.
 - Concentrations in sediment and biota were measured, and the results revealed transfer of alkylphenolics from sediments to biota and accumulation in the invertebrate tissue.
- Klecka *et al.* (2007) Exposure analysis of C8- and C9-alkylphenols, alkylphenol ethoxylates, and their metabolites in surface water systems within the United States.
 - This paper presents a statistical analysis of monitoring data provided by 19 investigations of alkylphenol ethoxylates (APE) and their metabolites in US surface waters conducted between 1990 and 2005.

Environmental Benchmarks

Yes, environmental quality benchmark criteria are available from both Canada and the United States.

Canada

Canadian Environmental Quality Guidelines for Nonylphenol and its Ethoxylates

Environmental Media	Media Type	Guideline Type	Guideline Value *
Water	Freshwater	Full	1.0 µg·L ⁻¹
	Marine	Interim	0.7 µg·L ⁻¹
Sediment **	Freshwater	Provisional Interim	1.4 mg·kg ⁻¹
	Marine	Provisional Interim	1.0 mg·kg ⁻¹
Soil	Agricultural	SQGE ***	5.7 mg·kg ⁻¹
	Residential/Parkland	SQGE	5.7 mg·kg ⁻¹
	Commercial	SQGE	14 mg·kg ⁻¹
	Industrial	SQGE	14 mg·kg ⁻¹
	Groundwater	Check value	21 mg·kg ⁻¹

Source: Environment Canada 2002.

* all guidelines are expressed in nonylphenol toxic equivalent units

** these guideline values are for sediments containing 1% total organic carbon

*** environmental soil quality guideline; a full soil quality guideline would also consider protection for human health (CCME 1996)

United States

U.S. Water Quality Criteria for Nonylphenol in Fresh and Marine Water

Environmental Media	Media Type	Guideline Value
Water	Fresh water	- A four day average of 6.6µg/L not exceeded more than once every three years - A one hour average of 28µg/L not exceeded more than once every three years
	Marine water	- A four day average of 1.7µg/L not exceeded more than once every three years - A one hour average of 7.0µg/L not exceeded more than once every three years

Source: USEPA 2005.

Note: The USEPA's WQC for nonylphenol does not consider toxicity related to the short chain nonylphenol ethoxylates, in particular the mono- and di- nonylphenol ethoxylates.

US EPA finalized ambient Water Quality Criteria for NP in 2006 (USEPA 2005). EPA WQC provide guidelines to states for the development of state Water Quality Standards under the Clean Water Act. When monitoring indicates an exceedance of state WQS, regulatory mechanisms exist under the National Pollutant Discharge Elimination System (NPDES) to enact controls.

Environmental Levels and Trends

Bennie *et al.* 1997 reported NPE concentration ranges for surface water and sediment samples from the Laurentian Great Lakes Basin. Of the surface water samples, 58% were found to contain NPE₁ and 32% contained NPE₂. Their concentrations ranged from <0.020 to 7.8 µg/L for NPE₁ and from <0.020 to 10 µg/L for NPE₂. A total of 66% of sediments had detectable amounts of NPE₁, and concentrations ranged from <0.015 to 38 µg/g (dry weight), 66% of sediments had detectable amounts of NPE₂, and values ranged from <0.015 to 6.0 µg/g (dry weight).

The highest range of NPE (NPE₁ & NPE₂) surface water concentrations exceeded both the Canadian freshwater guideline value of 1.0 µg/L and the United States freshwater four day average of 6.6 µg/L.

The results of Bennie *et al.* 1997 are over 10 years old and do not reflect changes that may have occurred since Canadian and U.S. management actions have taken place.

Source/Use/Release/Exposure Information

Source/use/release/exposure information available primarily from Environment Canada & Health Canada's Priority Substance List Assessment Report - Nonylphenol and its Ethoxylates (2001).

Source/Use

Nonylphenol ethoxylates (NPEs) are surfactants that have been in commerce for over 50 years. Products containing NPEs are used in many sectors, including textile processing, pulp and paper processing, paints, resins and protective coatings, oil and gas recovery, steel manufacturing, pest control products and power generation. A variety of cleaning products, degreasers and detergents are also available for institutional and domestic use. These products have numerous applications, including controlling deposits on machinery, cleaning equipment, scouring fibres, as wetting and de-wetting agents, in dyeing, in machine felt cleaning and conditioning and in product finishing.

NPEs have also been used in a wide range of consumer products, including cosmetics, cleaners and paints, and in a variety of applications. Use of NPEs in consumer cleaning and detergent applications is expected to have declined significantly in recent years due to governmental risk management measures implemented in Canada in 2005 and market pressures in North America generally.

The amount of NP/NPE available for use in Canada was 23 800 tonnes in 1995 and 19 000 in 1996.

In the United States NP production was 147.2 million pounds (66 800 tonnes) in 1980 (USITC 1981), 201.2 million pounds (91.300 tonnes) in 1988 (USITC 1989), 230 million pounds (104 000 tonnes) in 1998 (Harvilicz 1999).

More recently, North American consumption of NPE surfactants has been estimated between 300 and 400 million pounds per annum (Colin A. Houston & Associates, Inc. 2007).

Release

In the United States the vast majority of NPE are disposed and treated in industrial and/or publicly owned wastewater treatment plants (WWTP) prior to entering the aquatic environment. Their fate and treatability in WWTP has been well studied and reviewed (Drewes *et al.* 2005; Loyo-Rosales *et al.* 2007; Melcer *et al.* 2007; Esperanza *et al.* 2004). The composition of the mixture of degradation intermediates can differ considerably among the various effluents, depending on the source and the degree and type of treatment. Textile mill effluents have been a major source of NPEs to the environment. In Canada, reported releases from textile mills have decreased significantly as a result of government risk management measures implemented in Canada in 2005. Municipal effluents are a significant source of NPEs and are widespread across Canada.

Exposure:

In Canada, these chemicals have been found in fresh water, sediment, fish and beluga whale tissue, textile mill effluents, pulp and paper mill effluents, MWWTP influents, effluents and sludges, and soil to which municipal sludges had been applied.

Environmental and Health Data

Environmental and health information is available from Environment Canada & Health Canada's Priority Substance List Assessment Report - Nonylphenol and its Ethoxylates (2001).

CEPA 1999 64(a): Based on the available data, it is concluded that nonylphenol and its ethoxylates are entering the environment in a quantity or concentration or under conditions that have or may have an immediate or long-term harmful effect on the environment or its biological diversity. Therefore, nonylphenol and its ethoxylates are considered to be "toxic" as defined in CEPA 1999 Paragraph 64(a).

CEPA 1999 64(b): Based on the available data, it is concluded that nonylphenol and its ethoxylates are not entering the environment in a quantity or concentration or under conditions that constitute or may constitute a danger to the environment on which life depends. Therefore, nonylphenol and its ethoxylates are not considered to be "toxic" as defined in CEPA 1999 Paragraph 64(b).

CEPA 1999 64(c): On the basis of consideration of the margin of exposure between effect levels and reasonable worst-case estimates of intake from environmental media, nonylphenol and its ethoxylates are not considered a priority for investigation of options to reduce public exposure through control of sources that are addressed under CEPA 1999.

Other Reasons for Concern

Evidence from scientific journals suggests that NPEs may have endocrine disrupting properties (European Chemicals Bureau 2002; Klecka *et al.* 2007; Servos 1999). Disruption of the endocrine system can occur in various ways. Some chemicals mimic a natural hormone, fooling the body into over-responding to the stimulus (e.g., a growth hormone that results in increased muscle mass), or responding at inappropriate times (e.g., producing insulin when it is not needed). Other endocrine disrupting chemicals block the effects of a hormone from certain receptors (e.g., growth hormones required for normal development). Still others directly stimulate or inhibit the endocrine system and cause overproduction or underproduction of hormones (e.g., an over or underactive thyroid). In many situations involving environmental chemicals, an endocrine effect is not desirable (USEPA 2009).

EPA's WQC Document for NP notes that some studies suggest that NP has weak estrogenic activity. The document addresses reproductive, developmental and estrogenic effects of NP in aquatic species (pp. 26-28). Twenty four related studies were incorporated and conventional endpoints were found to be the more sensitive (e.g., growth, survival, reproduction) than estrogenic biomarker effects (e.g. vitellogenin induction). The EPA WQC document concludes "the ability of nonylphenol to induce estrogenic effects has seldom been reported at concentrations below the freshwater Final Chronic Value of 6.5965 ug/L" (USEPA 2005).

Nichols *et al.* (2001) tested the commercial product NPE9 and found no induction of VTG at any concentration tested in a 42-day flow-through study with fathead minnows (*P. promelas*).

Balch and Metcalfe (2006) exposed Japanese medaka (*Oryzias latipes*) to NP, NP1E, NP4E, NP9E, and NP1EC for 100 days post hatch and measured the incidences of altered sex ratios, mixed secondary sex characteristics, and gonadal intersex. Based on these endpoints, the relative estrogenic potency of NP1E was found to be 0.083. No estrogenic effects were observed for NP4E, NP9E, or NP1EC at the concentrations used in this study.

III. PRESENT MANAGEMENT STATUS

Canada

There are two Pollution Prevention (P2) Planning notices under CEPA developed to manage risks associated with NPEs (both effective December 04, 2004) (Environment Canada 2008):

- Notice Requiring the Preparation and Implementation of Pollution Prevention Plans in Respect of Nonylphenol and its Ethoxylates Contained in Products.
 - Phase 1 sets a reduction target of 50 percent from base year levels (typically 1998), of the total mass of NP and NPEs used in the manufacturing of products or imported annually. Phase 2 sets a target of 95% reduction from base year levels of the total mass of NP and NPEs used in the manufacturing of products or imported annually.

- *Status:* In 2006, facilities reported annual reductions of 63% of nonylphenol (NP) and its ethoxylates (NPEs) used in the manufacturing of products and 81% in imports since preparing and implementing their P2 plans. Most facilities are a year ahead of schedule for meeting the first phase of reduction targets.
- Notice Requiring the Preparation and Implementation of Pollution Prevention Plans in Respect of Nonylphenol and its Ethoxylates Used in the Wet Processing Textile Industry and Effluents From Textile Mills that Use Wet Processing.
 - Affected persons must prepare and implement a P2 plan that takes into consideration the following two objectives: 1. reduce the annual use of NP and NPEs by at least 97% relative to the annual use for 1998 by 2009, and; 2. reduce the toxicity of effluent to a maximum acute toxicity of 13 % IC₅₀ (50% inhibiting concentration by 2009.
 - *Status:* Textile mills surpassed the 97% reduction targets for annual use of nonylphenol and its ethoxylates. Despite these significant reductions, effluent tests reveal that 78% of the facilities are still struggling to maintain acceptable toxicity levels.

United States

EPA's Office of Pollution Prevention and Toxics (OPPT), Design for the Environment Program (DfE) Formulators Program - The EPA DfE Formulator Program is a voluntary program in which EPA partners with cleaning product manufacturers and others in the design of products with a more positive health and environmental profile. Under the Formulators Program, DfE assesses the potential health and environmental effects of each ingredient in the approved formulations.

EPA Office of Pollution Prevention and Toxics (OPPT), Design for the Environment Program (DfE) Formulators Program Safer Detergent Stewardship Initiative - Building on its Formulator work, DfE launched SDSI, a high-level recognition program for companies (manufacturers, formulations, end users, and sellers) who commit to switch completely to "safer surfactants". NPEs are provided as an example of a surfactant class that does not meet the SDSI definition of a safer surfactant.

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