Canadian Water Quality Guidelines for the Protection of Aquatic Life

CARBARYL

Carbaryl (CAS Registry Number 63-25-2) is a carbamate insecticide which exerts its toxic effect through cholinesterase inhibition. Its chemical formula is $C_{12}H_{11}NO_2$. Carbaryl is a white, odourless and crystalline solid with a molecular mass of 201 g•mol^{-1} (IPCS 1994).

Carbaryl was first introduced in 1956 and was the first carbamate insecticide to be successfully marketed for agricultural and household use. The current registrant of technical grade carbaryl is Bayer CropScience and the insecticide is registered in Canada, the United States, Madagascar, South Africa, Tanzania, Australia, India, New Zealand, Philippines, Hungary, Portugal, and the United Kingdom. In Canada, carbaryl is registered for and available in multiple end-use formulations including dusts, bait dusts, granules, soluble concentrates, wettable powders and ready-to-use spray formulations (PMRA 2007).

Uses: Carbaryl is a broad spectrum insecticide with agricultural use for pest control on crops, residential use on lawns and gardens, and domestic use for control of fleas and lice on household pets (CCOHS 2008). Carbaryl controls more than 100 insect species on a variety of crops including fruit, nuts, ornamentals and shade trees as well as on poultry and livestock (EXTOXNET 1993).

The PMRA registers carbaryl for the application methods of dusting, as well as air and ground spraying. Specific application rates of carbaryl depend on the application target and the formulation. The formulation Sevin SL (43% technical active) has application rates for vegetable crops ranging from 1.25 to 6.4 L•ha^{-1}, for tobacco ranging from 2 to 5.25 L•ha^{-1}, and for tree fruit crops ranging from 2000-3000 L•ha^{-1} for dilute sprays and from 300-1000 and 100-200 L•ha^{-1} for concentrate and aerial sprays, respectively (PMRA 2007). The formulation Chipco Sevin RP2 (22.5% technical active) has typical spray volumes of 800 to 1600 L•ha^{-1} for vegetables crops and 1100-3400 L•ha^{-1} for small fruit crops (PMRA 2007).

Sources to the environment: Direct application of carbaryl to soil, vegetation, and animals can result in exposure to non-target organisms. In Canada, carbaryl is not registered for direct application to the water. Carbaryl can enter the aquatic environment through spray drift and run-off from application. Accidental spills, dumping of tank residues or washing of application equipment can cause elevated but transient levels of carbaryl in the environment.

Fate, behaviour and partitioning: Technical carbaryl is lipophilic and sparingly soluble in water, with a solubility of 40 mg•L-1 at 30°C (IPCS 1993). It is soluble in ethanol, petroleum ether, diethyl ether, chloroform, and dimethyl sulfoxide and moderately soluble in petroleum oils, dimethyl formamide, acetone, isophorone and cyclohexanone. The rate of hydrolysis for carbaryl is highly influenced by pH. At pH 7 the half-life of carbaryl is 10-16 days, at pH above 8 the half-life is in the range of several hours and at pH 6 the half-life of carbaryl is 406 days (IPCS 1994). The main products of hydrolysis for carbaryl in water are 1-naphthol and carbon dioxide. In surface water, carbaryl can be broken down by bacteria (EXTOXNET 1993).

Environmental factors influence the rate of degradation of carbaryl in soil, for example soil type, soil aeration and soil temperature. At room temperature (23-25°C) and aerobic conditions, light textured soil has a half-life of 11 days compared to 21-27 days for heavy textured soil. Lowering the temperature to 15°C causes a 2-fold increase in the half life of carbaryl (Khasawinah 1978). Carbaryl has low volatility from moist soils and surface water based on a Henry's Law constant of 5.3 x 10-6 (IPCS 1994). The average Koc value for carbaryl adsorption for silty clay loam, sandy loam, sediment and silt loam was 211, while the average Koc value was 624 for desorption. Using KOC values to predict leaching potential, carbaryl is anticipated to have medium mobility in silty clay loam, sandy loam, sediment and silt loam. In sand soils of low

Table 1. Canadian water quality guidelines (CWQG),for carbaryl for the protection of aquatic life (µg a.i.·L^{-1}).

	Long-Term Exposure	Short-Term Exposure
Freshwater	0.20*	3.3 **
Marine	0.29*	5.7*

* value calculated from low-effect data using lowest endpoint approach

** value calculated from LC_{50} data using the SSD approach organic matter, the mobility is predicted to be high as desorption K values are positively correlated with the percent organic matter (Skinner 1994).

Carbaryl is not likely to bioaccumulate significantly in aquatic organisms. Bioconcentration factors have been found to be between 14 and 75 for freshwater fish species (IPCS 1994). Because carbaryl is rapidly metabolized and degraded, and because of the low octanol/water partition coefficient (log K_{ow} 1.59- 2.3), it is not likely to pose a bioaccumulation risk in alkaline water, however the risk increases under conditions below neutrality as the half-life of carbaryl increases (EXTOXNET 1993).

Analytical methods: The majority of analytical methods used to quantify carbaryl and its metabolites are based on separation by chromatographic techniques including gas-liquid chromatography (GLC), thin-layer chromatography (TLC) and high pressure liquid chromatography (HPLC) using detectors such as ultraviolet (UV), mass spectrometry (MS) and diode array detector (DAD) (Zhu et al. 2008). The detection limits of the aforementioned techniques can be below 0.001 µg•L^{-1} and recovery is generally greater than 80% (IPCS 1994). New methods which are less costly and more efficient include chemiluminescence with detection limits of 0.0039-0.0367 µg•L^{-1}, enzyme-linked immunoassays which can be used for field assays and have detection limits of 10 µg•L^{-1} and biosensors with detection limits of 30 µg•L^{-1} (Perez-Ruiz et al. 2007; Wang et al. 2005; Suwansa-ard et al. 2005).

Ambient concentrations: The presence and level of priority pesticides in select aquatic ecosystems in Canada was monitored in a project by Environment Canada Pesticides Science Fund. Carbaryl was monitored in the Atlantic region and the region of Québec between 2003 and 2005. Surveillance monitoring did not detect any level of carbaryl in surface waters of New Brunswick, Prince Edward Island and Nova Scotia (detection limits unreported). No detectable levels of the insecticide were found in surface waters of the Québec region over the course of the study; detection limits were 0.01 to 0.03 µg•L^{-1} (Cantox Environmental 2006). Between 1998 and 2006 the Québec government carried out monitoring of pesticide concentrations along the St. Lawrence River including the tributaries L'Assomption, Bayonne, Maskinongé and du Loup. In 2006, carbaryl was detected at a frequency of 3% in the tributary Bayonne and at a frequency of 4% in the tributary Maskinongé. The concentration of carbaryl detected at Maskinongé in July 2006 was 0.07 µg•L^{-1} with a method detection limit of 0.07 µg•L^{-1} (Giroux 2007).

Mode of action: The primary mechanism of toxicity for carbamate pesticides, including carbaryl, is cholinesterase (ChE) inhibition. Degradation of the neurotransmitter acetylcholine is inhibited causing it to build up and over stimulate the central nervous system. The effects on pests are exerted after carbaryl is ingested into the stomach or absorbed through direct contact. Hydrolysis and ring hydroxylation are the principal metabolic pathways. The major degradation product is 1-naphthol (IPCS 1993).

Toxicity: In the following sections, all concentrations of carbaryl expressed in µg•L^{-1} refer to µg of active ingredient (a.i.) per litre. In general, invertebrate species are more sensitive to carbaryl exposure than vertebrate, amphibian and plant/algal species, which is consistent with its selective target toxicity to insect species.

Short-term toxicity values for freshwater fish ranged from a 48-h LC_{50} of 15.83 µg a.i.•L^{-1} for the spotted snakehead (*Channa punctata*) to a 24-h TL_m of >32 000 µg a.i.•L^{-1} for the fathead minnow (*Pimephales promelas*) (Bhattacharya, 1993; Henderson et al. 1960). Other physiological symptoms of short-term carbaryl toxicity include changes to the rate of oxygen consumption in *Tilapia mossambica* exposed to carbaryl at one third of the LC_{50} (5495 µg a.i.•L^{-1}) for 48 hours, decreased heart rate in rainbow trout (*Oncorhynchus mykiss*) exposed to the LC_{95} for carbaryl for 24 and 48 hours, accumulation of lactic acid in various tissues of the catfish (*Clarias batrachus*) exposed to sublethal concentrations of carbaryl, and neurological injury in juvenile medaka (*Oryzias latipes*) exposed to sublethal concentrations of carbaryl (Basha et al. 1984; McKim et al. 1987; Sharma 1995; Carlson et al. 1998).

Long-term toxicity values for freshwater fish exposed to carbaryl ranged from a 9-month LC_{20} of 32.46 µg a.i.•L^{-1} to a 7-d LOEC for growth of 4000 µg a.i.•L^{-1} for the fathead minnow (*Pimephales promelas*) (Carlson 1971). Sastry et al. (1988) exposed the spotted snakehead (*Channa punctatus)* to carbaryl at 10500 µg a.i.•L^{-1} for 96 hours and at 1050 µg a.i.•L^{-1} for 120 days. During both exposures fish were hyperglycaemic, hyperlactemic and had depleted glycogen levels in the liver and muscles. Other haematological and enzymatic effects included altered levels of lactic acid and altered activity levels of hexokinase, lactate dehydrogenase, pyruvate dehydrogenase and succinate dehydrogenase in various tissues (Sastry et al. 1988).

Among the most sensitive short-term endpoint values for freshwater invertebrate species exposed to carbaryl was a 96-h NOEC for mortality of 3.4 µg a.i.•L^{-1} for the larval stage stonefly (*Chloroperla grammatica*) (Schafers 2002). The lowest LC_{50} for an invertebrate species was 4.075 µg a.i.•L^{-1} for an adult cladoceran (*Bosmina fatalis*) for an exposure duration of 24-h (Sakamoto et al. 2005). The most tolerant species were the paramecium (*Paramecium aurelia*) with a 24-h LC_{50} of 46000 µg a.i.•L^{-1} (Edmiston et al. 1984) followed by the pondmussel (*Ligumia subrostrata*) with a 24-h LC_{50} of 43100 µg a.i.•L^{-1} for the glochidia life stage and the paper pondshell mussel (*Utterbackia imbecellus*) with a 24-h LC_{50} of 40200 µg a.i.•L^{-1} for the glochidia life stage (Milam et al. 2005). The damselfly (*Xanthocnemis zealandica*) was exposed to carbaryl for 48-h at various stages in the life cycle and it was found that the most sensitive life stage was the 2nd instar (48-h LC_{50} of 156.6 µg a.i.•L^{-1}) and the most tolerant was the 10th instar (48-h LC_{50} of 770 µg a.i.•L^{-1}). An overall pattern was observed that earlier life stages were more sensitive to the toxicity of carbaryl (Hardersen and Wratten 2000).

Many invertebrate species demonstrated high sensitivity to carbaryl during long-term exposure including the zooplankton *Daphnia ambigua* which had an EC_{50} for survival of 2 µg a.i.•L^{-1} for an exposure period of the 1st to 6th instar stage (Hanazato 1991). Adult waterfleas (*Daphnia magna*) had a 21-d MATC for survival and reproduction of >3.3 µg a.i.•L^{-1} (Springborn Bionomics 1985), and the zooplankton (*Daphnia galeata*) had a 7-d IC_{70} of 5 µg•L^{-1} for abundance (Havens 1995). The midge (*Chironomus riparius*) was a relatively more tolerant species with a 28-d NOEC of 147.25 µg a.i.•L^{-1} and a 28-d LOEC of 318.31 µg a.i.•L^{-1} for larval development (Ebeling and Radix 2002).

The most sensitive amphibian species to the short-term toxicity of carbaryl was the African clawed frog (*Xenopus laevis*) with a 24-h EC_{50} for development of 110 µg a.i.•L^{-1} (Elliott-Feeley and Armstrong 1982) and the most tolerant species was the green frog (*Rana clamitans*) with short-term LC_{50}s for tadpoles ranging from 11320 to 26010 µg a.i.•L^{-1}. The most sensitive amphibian species in a long-term toxicity test was the gray tree frog (*Hyla versicolor*) which had a 10-d EC_{60} for survival of 50 µg a.i.•L^{-1} for tadpoles (Relyea and Mills 2001).

Limited data were available concerning the short-term and long-term toxicity of carbaryl to plants. Water lettuce (*Pistia stratiotes*) and water spinach (*Ipomoea aquatica*) had a 96-h EC_{50} for chlorophyll content of 785 000 and 996 000 µg a.i.•L^{-1}, respectively (Booyawanich et al. 2001). The most sensitive endpoint for long-term exposure was a 5-d EC_{10} of 140 µg a.i.•L^{-1} for blue-green alga (*Anabaena flos-aquae*) (Lintott 1992b) while the least sensitive endpoint was an IC_{48} of 5000 µg a.i.•L^{-1} for green algae (*Scenedesmus bijugatus*) (Megharaj et al. 1989).

Toxicity data for marine species were limited. The most sensitive fish tested in acute saltwater conditions was the striped bass (*Morone saxatilus*) with a 96-h LC_{50} of 2300 µg a.i.•L^{-1} (Palawski et al. 1985), while the least sensitive was the mosquitofish (*Gambusia affinis*) with a 96-h TL_m of 31800 µg a.i.•L^{-1} (Chaiyarach et al. 1975). Among marine invertebrates, the most sensitive species was the protozoan *Euplotes sp.* with a 24-h LC_{50} of 1 µg a.i.•L^{-1} (Weber et al. 1982) while the least sensitive species was the macrid clam (*Rangea cuneata*) with a 96-h TL_m of 125000 µg a.i.•L^{-1} (Chaiyarach et al. 1975).

Toxicity Modifying Factors: There are insufficient data regarding the effects of pH, temperature, hardness and UV radiation on the toxicity of carbaryl to reliably identify patterns of toxicity modifying effects or to normalize toxicity data.

Water Quality Guideline Derivation: The Canadian Water Quality Guidelines (CWQGs) for short-term and long-term exposures for carbaryl for the protection of aquatic life were developed based on the CCME protocol (CCME 2007). The short-term freshwater guideline was developed using the statistical (Type A) approach with a Species Sensitivity Distribution (SSD). The long-term freshwater guideline was developed using the lowest-endpoint (Type B2) approach. The short and long-term marine guidelines were also developed using the lowest endpoint (Type B2) approach.

Short-term Freshwater Quality Guideline: Short-term exposure guidelines provide information on the impacts of severe but transient events and are derived using severe effects data (such as lethality) of defined short-term exposure periods (see CCME 2007 for exposure period definitions). These guidelines identify estimators of severe effects to the aquatic ecosystem and are intended to give guidance on the impacts of severe, but transient, situations (e.g., spill events to aquatic receiving environments and infrequent releases of short-lived/nonpersistent substances). Short-term exposure guidelines *do not* provide guidance on protective levels of a substance in the aquatic environment, as short-term exposure guidelines are levels which *do not* protect against adverse effects, but rather indicate the level where severe effects are likely to be observed.

The minimum data requirements for the Type A guideline approach were met. Toxicity studies meeting the requirements for primary and secondary data, according to CCME (2007) protocol, were considered in the derivation of the short-term SSD. Each species for which appropriate short-term toxicity data were available was ranked according to sensitivity, and its centralized position on the SSD was determined using the Hazen plotting position (estimate of the cumulative probability of a data point). Intra-species variability was accounted for by taking the geometric mean of the studies considered to represent the most sensitive lifestage and endpoint.

According to CCME (2007), datasets may display bimodal distributions on account of differential toxicological sensitivities across taxa due to the toxic mode of action of the substance. In such situations, separate SSD curves may be plotted for different taxa or functional groups and the most sensitive taxonomic level may be used to derive the guideline. Concerning carbaryl, a bimodal distribution was apparent when all taxa were plotted together (Figure 1), with arthropods demonstrating increased sensitivity due to the selective toxicity of carbaryl to insects. The SSD plotted for arthropods separately (split distribution)(Figure 2) is hence appropriate and can be used to derive the short-term guideline value.The final dataset used to generate the fitted SSD for carbaryl can be seen in Table 2.

Table 2. Arthropod endpoints used to determine the short-term CWQG for carbaryl.

Species	**Endpoint**	**Concentration (μg a.i.•L^{-1})**
B. fatalis	24-h LC_{50}	4.075
C. grammatica	96-h LC_{50}	5.8
B. longirostris	24-h LC_{50}	8.597
D. similis	48-h EC_{50} (immobility)	8.8
C. dubia	48-h LC_{50}	11.6
C. sphaericus	48-h EC_{50} (immobility)	12.4
D. magna	48-h EC_{50} (immobility)	16
M. lamarrei	96-h LC_{50}	19
G. fossarum	96-h LC_{50}	31
S. vittatum	48-h EC_{50} (immobility)	32.43*
E. danica	96-h LC_{50}	153
M. relicta	96-h LC_{50}	230
P. hoyi	96-h LC_{50}	250
A. aegypti	24-h LC_{50}	510

***value shown is the geometric mean of comparable values**

The Fisher-Tippett model provided the best fit of the twelve models tested (Figure 2). The equation of the fitted Fisher-Tippett model is in the form of

$$f(x) = e^{-e^{\frac{(L-x)}{s}}}$$

Where *x* is the log (concentration) and *F(x)* is the proportion of species affected.

Summary statistics for the short-term SSD are presented in Table 3. The concentration 3.3 µg a.i.•L^{-1}, is beyond the range of the data (to which the model was fit). Therefore, the 5th percentile and its fiducial limits (FL) (boundaries within which a parameter is considered to be located) are extrapolations.

Table 3. CWQG for short-term exposure for carbaryl resulting from the SSD method.

	Concentration
SSD 5th percentile	3.31 µg a.i.•L^{-1}
SSD 5th percentile, LFL (5%)	1.98 µg a.i.•L^{-1}
SSD 5th percentile, UFL (95%)	5.53 µg a.i.•L^{-1}

Therefore, the short-term exposure benchmark concentration indicating the potential for severe effects (e.g. lethality or immobilization) to sensitive freshwater/marine life during transient events is 3.3 µg a.i.•L^{-1}, for carbaryl.

Long-term Freshwater Quality Guideline

Long-term exposure guidelines identify benchmarks in the aquatic ecosystem that are intended to protect all forms of aquatic life for indefinite exposure periods.

Although the persistence of carbaryl in water may be limited under field conditions on account of its rapid degradation, aquatic organisms may experience long-term exposure to the pesticide. Aquatic organisms may be chronically exposed to carbaryl if they inhabit the waters receiving pesticide input from multiple sources, or multiple applications.

The acceptable long-term studies identified in this review consisted of three algal species, three fish species, and three invertebrate species. Based on the minimum data requirements; there were insufficient data to derive a long-term SSD for carbaryl according to CCME (2007) protocol, as no long-term study regarding a salmonid was available. There were also insufficient data to derive a long-term guideline using the lowest endpoint approach (Type B1). Therefore, following the tiered approach, the lowest endpoint approach (Type B2) guideline method was used to develop the long-term CWQG.

Using the Type B2 guideline method to derive the long-term CWQG, the critical (lowest acceptable) endpoint was identified as a 24h-LC_{50} of 4.075 µg a.i.•L^{-1}, for the cladoceran *Bosmina fatalis* (Sakamoto et al. 2005). A safety factor of 20 was applied to the lowest data to derive the Type B2 guideline for carbaryl.

Therefore, the long-term exposure CWQG for the protection of freshwater life is 0.20 µg a.i.•L^{-1}, for carbaryl.

Marine Water Quality Guideline

The acceptable short-term studies identified in this review consisted of four invertebrate species and three fish species. Based on the minimum data requirements; there were insufficient data to derive a short and long-term SSD for carbaryl according to the CCME (2007) protocol. There were also insufficient data to derive a short and long-term exposure guideline using the lowest endpoint approach (Type B1). Therefore, following the tiered approach, the lowest endpoint approach (Type B2) guideline method was used to develop the short and long-term marine CWQG.

Using the Type B2 guideline method to derive the short-term CWQG, the critical endpoint was identified as a 96-h LC_{50} of 5.7 µg a.i.•L^{-1} for the mysid *Mysidopsis bahia* (24-h old) (Lintott 1992a). A safety factor of 10 was applied to the critical endpoint to derive a Type B2 guideline. The critical endpoint for the long-term marine guideline was the same 96-h LC_{50} for the mysid as above. A safety factor of 20 was applied to the lowest data to derive the long-term Type B2 guideline for carbaryl.

Therefore, for carbaryl, the short-term exposure benchmark concentration indicating the potential for severe effects (e.g. lethality or immobilization) to sensitive marine life during transient events is 0.57 µg a.i.•L^{-1} and the long-term exposure CWQG for the protection of marine life is 0.29 µg a.i.•L^{-1}.

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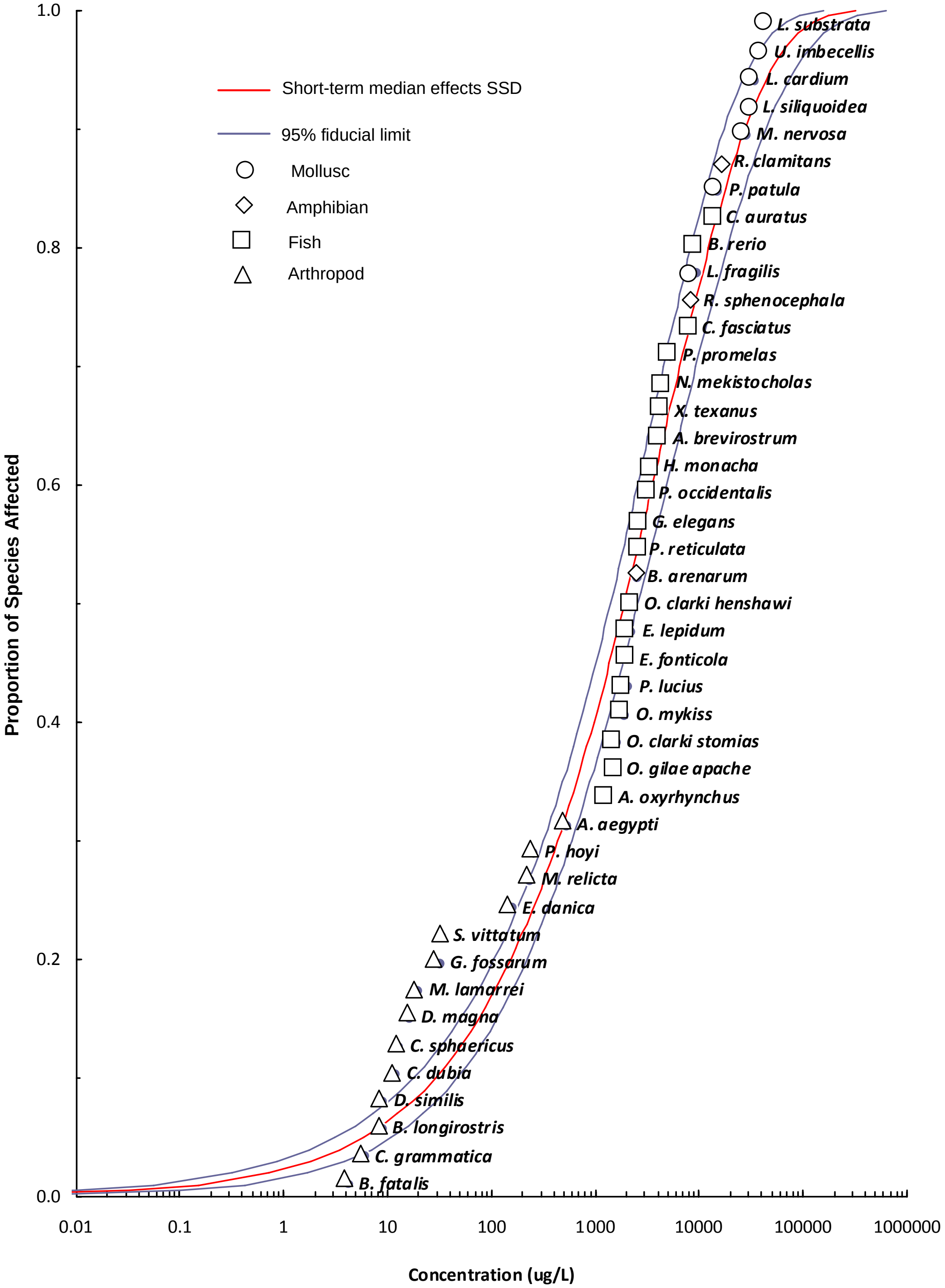

Figure 1. Short-term SSD representing the toxicity of carbaryl in freshwater consisting of acceptable short-term LC_{50}s of aquatic species versus proportion of species affected.

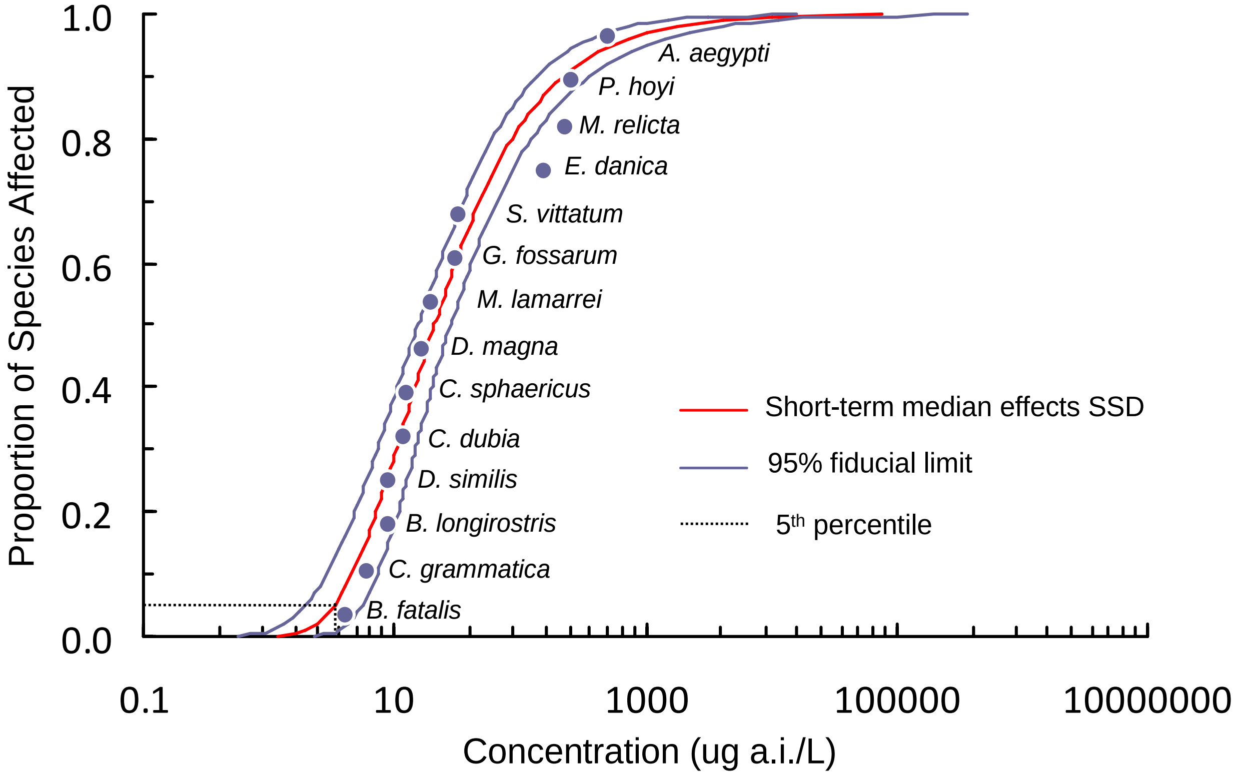

Figure 2. Short-term SSD representing the toxicity of carbaryl in freshwater consisting of acceptable short-term LC_{50}s of arthropod species versus proportion of species affected.

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For further scientific information, contact:

Environment Canada
National Guidelines and Standards Office
Fontaine
200 Sacre-Cœur Blvd.
Gatineau, QC K1A 0H3
Phone: (819) 953-1550
E-mail: ceqg-rcqe@ec.gc.ca
Internet: http://www.ec.gc.ca/ceqg-rcqe

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