

# Aquatic Toxicology Notes: Diquat<sup>1</sup>

P. Chris Wilson and Jun Wu<sup>2</sup>

## Introduction

This document is a companion to EDIS publication SL236/SS455, *Aquatic Toxicology Notes: Predicting the Fate and Effects of Aquatic and Ditchbank Herbicides* (Wilson 2006; <http://edis.ifas.ufl.edu/ss455>). This publication introduces users of diquat to the physical, chemical, environmental, and ecological properties of this herbicidal active ingredient relative to the aquatic environment. Readers should refer to SL236/SS455 for a more detailed introduction to the various concepts mentioned. These physical and chemical properties directly influence the fate, bioavailability, and potential toxicity of diquat to target and non-target organisms in the environment. Understanding the toxicological properties of diquat will provide users with an idea of which organisms and organism groups may be at greatest risk of negative impacts in a given treatment situation. Knowledge of these properties will allow users to identify high-risk situations for herbicide use. This information is discussed in detail in the following sections. As always, users should consult the product label for specific restrictions and allowed uses.

## Chemical Description

Diquat was first manufactured in England, and then exported for use in other countries. It was first sold in the United States in 1967. As a non-selective contact herbicide and crop desiccant, diquat damages plant tissues quickly, causing plants to wilt and desiccate within several hours of application (Vencill 2002). Affected plants appear frostbitten because of cell membrane destruction (CA EPA 1994).

Diquat also reduces plant photosynthetic activity (Ware 1978). It is applied as a dibromide salt, which dissociates to the active, cationic form (without the bromide counterions) (Table 1). Table 1 contains a summary of chemical and physical properties of diquat. These properties influence the transport, fate, and toxicity of this herbicide in the environment.

## Environmental Fate

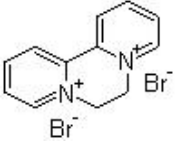
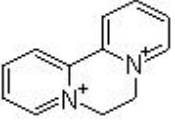
Diquat is applied directly to plants along ditch banks and within aquatic systems. It may also be applied to ponds, lakes, and drainage ditches to control algae and submersed aquatic weeds (Vencill 2002). Using the chemical properties of diquat (Table 1), it is possible to qualitatively predict its fate in the environment. These qualitative predictions are useful for understanding what happens to diquat once it is applied (i.e., where does it go and does it break down into nontoxic compounds) and how long it may be present in water and sediments. This information is useful to managers for predicting exposures and possible toxicity to non-target plants and animals within the treated system. The environmental compartments considered here are air, water, sediments, and biota.

## Air

Diquat is not volatile as indicated by its low vapor pressure (Table 1). While it does not exist in the vapor phase, it may exist in the air as aerosols during and shortly following spray applications depending on sprayer and environmental conditions (e.g., small droplet size and low humidity).

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2. P. Chris Wilson, associate professor, Soil and Water Science Department, Indian River Research and Education Center, Fort Pierce, FL; and Jun Wu, visiting scholar, Indian River Research and Education Center, Fort Pierce, FL; Florida Cooperative Extension Service, Institute of Food and Agricultural Sciences, University of Florida, Gainesville, FL 32611.

Table 1. Chemical nomenclature and chemical/physical properties for the herbicide diquat.

Property	Description	
Common Name	Diquat	
Chemical Name	6,7-dihydrodipyrido(1,2- $\alpha$ :2',1'-c)pyrazinediium ion	
Chemical Family <sup>1</sup>	Bipyridylium, dipyridylium	
Trade Names	Reglone®, Reward®, Weedtrine-D®	
Molecular Formula <sup>1</sup>	Dibromide salt: C <sub>12</sub> H <sub>12</sub> Br <sub>2</sub> N <sub>2</sub> (formulated product) Cation: C <sub>12</sub> H <sub>12</sub> N <sub>2</sub> (active form)	
Molecular Weight <sup>1</sup>	Dibromide salt: 344.05 Cation: 184.24	
CAS* Number <sup>1</sup>	Dibromide salt: 85-00-7 Cation: 2764-72-9	
Structure <sup>2</sup>	Dibromide salt (formulation)	Cation (active form)
		
Physical Appearance <sup>3</sup>	Colorless to yellow crystals, reddish in solution, formulated as aqueous solution	
Density <sup>1</sup>	1.61 g/mL at 25°C	
Melting Point <sup>1</sup>	325°C	
Vapor Pressure <sup>1</sup>	<1×10 <sup>-8</sup> mm Hg at 25°C	
Solubility <sup>1</sup> (Water)	718,000 mg/L	
pKa <sup>1</sup>	Does not dissociate	
LogK <sub>ow</sub> <sup>1</sup>	-4.25	
LogK <sub>oc</sub> <sup>4</sup>	6	
Sorption (K <sub>d</sub> ) <sup>3</sup>	15 (sediment) 36 (sand) 1,882 (sandy loam) 4,895 (sandy clay loam) 10,740 (loam)	
Henry's law constant <sup>3</sup>	<6.3×10 <sup>-14</sup> atm·m <sup>3</sup> /mol at 20–25°C	
Half-life (T <sub>1/2</sub> )	Photolysis: 74 days (aqueous) <sup>5</sup> Hydrolysis: stable at pH 5 and 7; >9 months at pH 9 <sup>5</sup> Soil dissipation: >1000 days (field) <sup>6</sup> Water dissipation: 1–2 days <sup>5</sup>	

\*Chemical Abstracts Service, <sup>1</sup>Vencill 2002, <sup>2</sup>Chemblink 2012, <sup>3</sup>CA EPA 1994, <sup>4</sup>Wauchope et al. 1992, <sup>5</sup>U.S. EPA 1995, <sup>6</sup>Kamrin 1997.

Previous research illustrated that diquat concentrations in air samples decreased 90% (from 0.6 mg/m<sup>3</sup> to 0.06 mg/m<sup>3</sup>) in a treated field 10 minutes after spraying, and no diquat was detectable 20 minutes after spraying. Moreover, diquat was not detected at a distance of 400 m from the treated field (WHO 2004; HSDB 2012). Diquat may also be re-distributed into air as fine soil particle-bound and dust-bound residues (CA EPA 2000).

## Water

Despite diquat's high water solubility, water concentrations are typically very low because of its cationic structure and high affinity for insoluble, negatively-charged constituents

(i.e., sediment, organic matter, etc.). Shorter half-lives should be expected in turbid water with visible solids and dense plants. Diquat concentrations in treated pond water in the Piedmont Plateau of North Carolina were reported to decrease from 1 ppm (treatment concentration) to below the detection limit (3 ppb) within 30–33 hours of treatment (Langeland and Warner 1986). The T<sub>1/2</sub> for diquat applied to Florida ponds near Gainesville was 1–2 days in the water column, with sorption to sandy clay loam and sandy sediments being the primary removal mechanism (U.S. EPA 1995). In addition to monitoring residues in treated ponds, a few studies have monitored diquat residues in water bodies that receive runoff/drainage water from terrestrial

sites where diquat is used. Ibáñez, Picó and Mañes (1996) reported a diquat concentration of 0.8 µg/L in surface water from L'Albufera Natural Park (València, Spain). Ibáñez, Picó and Mañes (1997) also detected diquat in surface water at the Torreblanca Nature Park (Castelló, València, Spain) at concentrations ranging from 0.1 to 0.3 µg/L. Fernández et al. (1998) monitored diquat in surface water from seven sampling points in Prat Cabanes-Torreblanca wetland (Castelló, Spain) once per month for a year. They reported that diquat concentrations tended to be highest during July, August, and September. The average concentration for diquat was 0.09 µg/L, with a maximum concentration of 3.10 µg/L (Fernández et al. 1998). Diquat does not degrade by hydrolysis in pH 5 and 7 water, but a small amount (8%) has been reported to hydrolyze in water at pH 9 after 30 days incubation in the dark (CA EPA 1994).

## Soil/Sediment

Although diquat is very soluble in water (Table 1), it is rapidly removed from the water column in treated aquatic systems primarily by sorption to sediments, plants, and turbidity-causing constituents. Sorption to turbidity-causing constituents can severely limit the efficacy of the herbicide for controlling submersed aquatic weeds and algae. Diquat sorbed to negatively-charged soil particles is not bioavailable to plants or microbes, thus little to no degradation occurs (U.S. EPA 1995). As a result, diquat concentrations in soil/sediment may remain relatively constant or increase as a result of repeat applications (WHO 2004). An EPA technical report indicated that concentrations of diquat ion in the upper 15-cm of loam and clay-loam soils ranged from 0.01 to 0.32 ppm in field plots three years after application in New York; and ranged from 0.01 to 0.13 ppm in the upper 35-cm soil depth three years following application to loam soil plots in Idaho (U.S. EPA 1995). Strong sorption of diquat to soil particles likely results in very low losses from treated soils in the dissolved phase. However, losses of soil-bound diquat may be significant if erosion prevention measures are not sufficient. Diquat is resistant to degradation by sunlight (photodegradation), chemical, and biological mechanisms (U.S. EPA 1995).

## Biota

Diquat is very quickly absorbed into target plant tissues (>50% within 1 hour) to which it is applied (Vencill 2002; Brian 1967). While diquat may be transported in the xylem of exposed plants, rapid plant desiccation usually prevents significant transport from the treated leaves (Vencill 2002). Even though diquat is tightly bound to negatively-charged surfaces, it is not expected to bioaccumulate in living plants and animals given its very low octanol:water partitioning

coefficient (Log  $K_{ow}$ , Table 1). Bioconcentration factors measured in fish exposed to 1.03 mg/L diquat dibromide were 0.7x (edible tissues), 2.5x (nonedible tissues), and 1.03x (whole fish) (U.S. EPA 1995). Approximately 50% of the accumulated diquat was eliminated during a three-day depuration period, further indicating non-significant bioaccumulation in fish (U.S. EPA 1995). The maximum bioconcentration factors reported for invertebrates are 8.3x for *Daphnia magna* (1-day post-exposure to 10 µg/L diquat dibromide); 32x for mayfly nymphs (1-day post-exposure to 1 mg/L diquat dibromide); and 5.5x for oysters (14-day exposure to 0.1 mg/L diquat dibromide monohydrate) (U.S. EPA 1995).

## Ecotoxicology

Ecotoxicology refers to the study of the effects of toxic substances and pollutants on plants, animals, and processes in the environment. The effects of diquat on aquatic species are varied. Because diquat is an herbicide, target and non-target plants are especially at risk of negative effects. Data are available indicating that diquat may also be toxic to a variety of fish and invertebrate species. A summary of these effects follows.

## Plants

Plants are at highest risk of negative impacts because of diquat's specific mode of action targeting them. Diquat is quickly absorbed into treated foliage, and it acts on plants by accepting electrons from photosystem I. This reduces the cationic diquat to diquat radical (electrons gained). The reduced diquat radical is next oxidized (electrons lost) by molecular oxygen to form a superoxide anion radical, and the cationic diquat is regenerated (Vencill 2002). The free radicals (diquat radical and superoxide anion) are molecules bearing an unpaired electron and are extremely reactive, engaging in rapid chain reactions that destabilize other molecules and generate many more free radicals (Dictionary.com 2011). The superoxide oxide anion then facilitates production of other free radicals that all result in the destruction of protein functions, membrane fatty acids, and chlorophyll. Destruction of membrane fatty acids leads to leakage of cytoplasm into intercellular spaces, resulting in rapid leaf wilting and desiccation (Vencill 2002).

Figure 1 shows a species sensitivity distribution (SSD) constructed using the 50% effective concentrations ( $EC_{50}$ ) for growth of aquatic plants and algae. The  $EC_{50}$  values correspond to the concentrations at which a 50% reduction in growth of the species was observed. For aquatic plants, the SSD intersects the lower axis (diquat concentration) at



0.0006 mg/L, indicating that no species should be impacted at concentrations lower than 0.0006 mg/L. The lack of a margin of safety can be great for an aquatic system being treated with diquat. According to the Weedtrine-D® label, the maximum treatment concentration for algal control is 1.5 mg/L. As seen in Figure 1 (\*\*), nearly 88% of plant species would be expected to be negatively impacted (growth reduced by at least 50%) at the highest treatment concentration. In contrast, the maximum labeled treatment concentration for Reward® (\*) is 0.4 mg/L. In this case, approximately 74% of species would be expected to be negatively impacted. Using the target treatment concentrations as a guide, it is easy to visualize the impacts if treatment concentrations were inadvertently higher than expected because of application errors. A similar analysis can be conducted for water bodies that were not treated but that receive runoff from watersheds where diquat is used. Using the concentrations from Ibáñez, Picó and Mañes (1997) and Fernández et al. (1998) as surrogates for the expected environmental concentrations, there is no margin of safety at the highest concentration reported (Figure 2). Approximately 18% of the aquatic plant species within the area would be expected to be adversely impacted by the highest diquat concentration of 3.1 µg/L (Figure 2).

Figure 3 shows a SSD constructed using available Lowest Observable Effects Concentrations (LOEC) for aquatic macrophytes and algae. LOECs correspond to the lowest concentration where observable effects are seen. The 3.1 µg/L concentration reported in the literature is greater than the LOEC for approximately 12% of plant species,

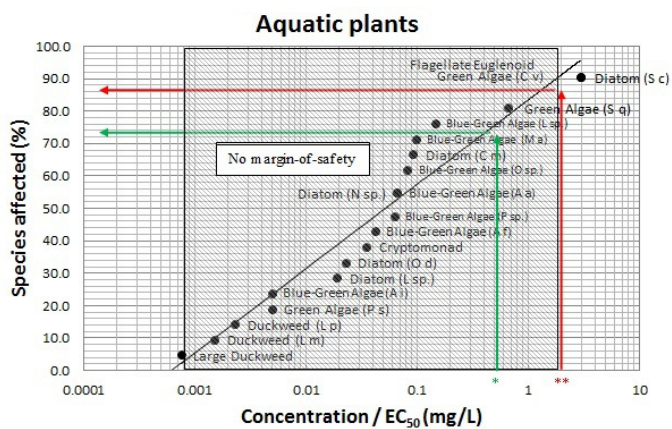


Figure 1. Species sensitivity distribution for aquatic plants exposed to diquat. The straight line represents the model fit to all of the data ( $y = 11.285\ln(x) + 83.41$ ;  $R^2 = 0.9373$ ).

\*Maximum target concentration for Reward® is 0.4 mg/L, indicating a lack of a margin of safety for approximately 74% of species.

\*\*Maximum target concentrations for Weedtrine-D® is 1.5 mg/L, indicating a lack of a margin of safety for approximately 88% of species.

indicating that some toxicity should be expected for those plants, while minimal effects would be expected on the other 88% of aquatic plant species exposed. Toxic effects would be less intense than those at the  $EC_{50}$  for the plants.

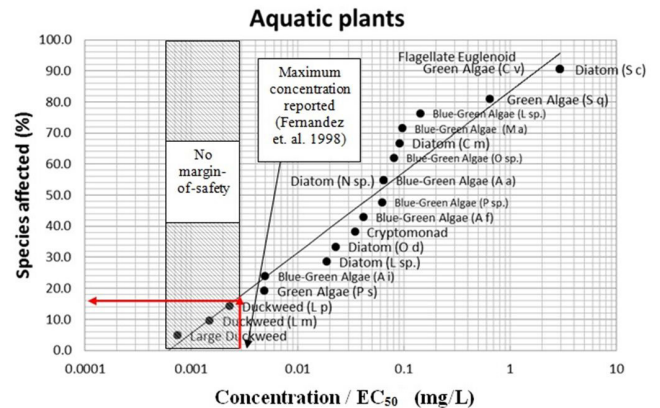


Figure 2. Species sensitivity distribution for aquatic plants exposed to expected environmental concentrations of diquat (not treatment concentrations). The straight line represents the model fit to all of the data ( $y = 11.285\ln(x) + 83.41$ ;  $R^2 = 0.9373$ ). Maximum aqueous concentrations from the literature are higher than the most sensitive species, indicating a lack of a margin of safety for approximately 18% of species.

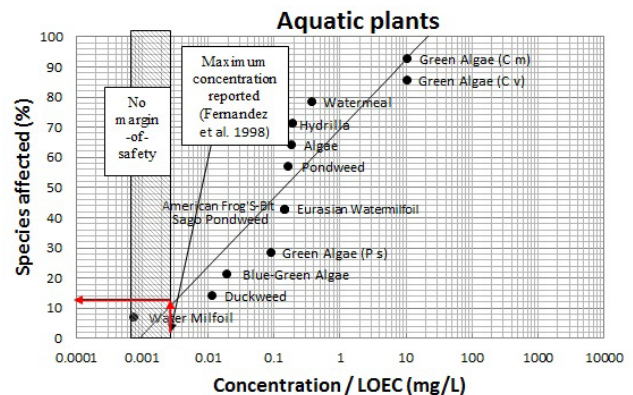


Figure 3. Species sensitivity distribution (plants, based on LOEC) for aquatic plants exposed to diquat. The straight line represents the model fit to all of the data ( $y = 9.953\ln(x) + 69.641$ ;  $R^2 = 0.8228$ ). Maximum aqueous concentrations from the literature are higher than the most sensitive species, indicating a lack of a margin of safety for approximately 12% of species.

## Aquatic Animals

Although plants are the primary environmental resources affected by diquat, other organisms may also be affected if exposed to high enough concentrations for a long enough duration. Diquat is either applied directly to the plants for emergent macrophyte control or to the water for submerged aquatic macrophyte and algae control. When applied directly to the water body, diquat rapidly binds to plants, sediments, and other negatively-charged and non-polar

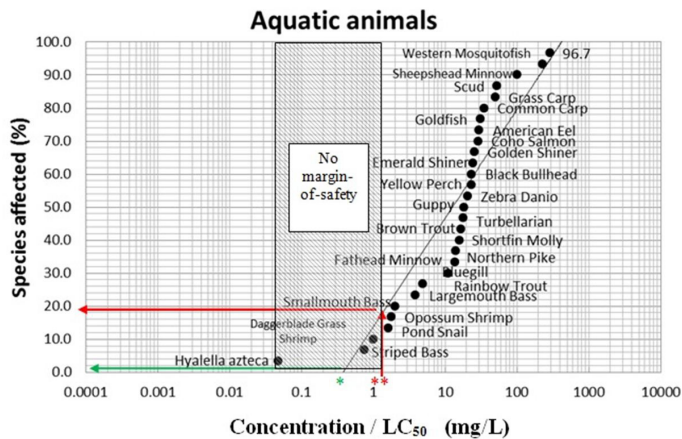


Figure 4. Species sensitivity distribution for animals exposed to diquat. The straight line represents the model fit to all of the data ( $y = 14.291\ln(x) + 13.734$ ;  $R^2 = 0.826$ ).

\*Maximum target concentration for Reward® is 0.4 mg/L, indicating minimal effects on aquatic animals (i.e., 0.4 mg/L is lower than  $LC_{50}$  for most species).

\*\*Maximum target concentration for Weedtrine-D® is 1.5 mg/L, indicating a lack of a margin of safety for approximately 80% of species.

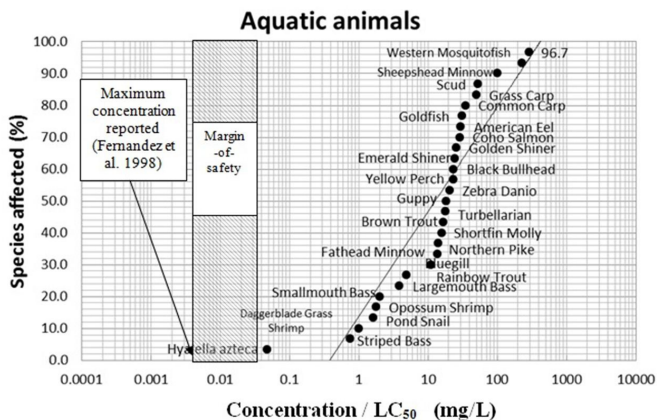


Figure 5. Species sensitivity distribution for animals exposed to diquat. The straight line represents the model fit to all of the data ( $y = 14.291\ln(x) + 13.734$ ;  $R^2 = 0.826$ ). Diquat is not usually applied directly to the water except when controlling submerged weeds. Maximum aqueous concentrations from the literature are much lower than the most sensitive species, indicating a likely margin of safety for terrestrial uses.

surfaces, resulting in quick dissipation of the dissolved fraction. As a result, it is difficult to estimate exposure concentrations (dissolved) for organisms in the treated water body. However, using the labeled maximum treatment lake concentrations, we can visualize the potential toxicity and/or margin of safety for aquatic animals within the treated system. Figure 4 shows a SSD constructed using 50% lethal concentration ( $LC_{50}$ ) values for aquatic animals, with the maximum treatment concentrations for two diquat formulations indicated.  $LC_{50}$  values are concentrations at which 50% of the animals exposed to the chemical die. The maximum treatment concentration for Reward® is less

than the  $LC_{50}$  values for the majority of species, except for the invertebrate *Hyalella azteca*. The maximum treatment concentration for Weedtrine® (1.5 mg/L) is greater than the  $LC_{50}$  values for about 20% of the species, indicating more severe impacts on non-target aquatic animals.

A similar analysis can be conducted for water bodies not treated with diquat but that receive runoff from watersheds where diquat is used, with the previously reported values of 0.1–0.3  $\mu\text{g/L}$  (Ibáñez, Picó and Mañes 1997) and 0.09–3.1  $\mu\text{g/L}$  (Fernández et al. 1998) as surrogates for expected environmental concentrations. Figure 5 shows a SSD for acute toxicity to aquatic animals, using the maximum expected environmental concentration (3.1  $\mu\text{g/L}$ ). In this case, the SSD intersects the lower axis at 0.4 mg/L, indicating that no species should be impacted (at  $LC_{50}$ ) at lower concentrations. As seen in Figure 5, there is a margin of safety for all of the aquatic organisms, indicating that negative impacts should be minimal for most aquatic animals. The highest reported concentration in the environment is an order of magnitude lower than the  $LC_{50}$  for the most sensitive species. However, keep in mind that this may not be the case at all locations since the fate of diquat is also influenced by many local environmental conditions. Concentrations shortly after application are likely to be higher than concentrations hours afterwards. Also, note that the sediment/organic matter consuming invertebrates (*Hyalella*, daggerblade grass shrimp, pond snail, opossum shrimp, and bass) are among the most sensitive to diquat.

## Uncertainty

Uncertainty always exists in environmental risk assessments. This analysis is based on several assumptions. First, the SSD assumes that the known species represent the universe of species. Research has shown that this assumption holds for some classes of contaminants, but exhaustive attempts to evaluate it with all classes of contaminants are resource prohibitive. A second assumption is that the acute toxicity  $EC_{50}$  and  $LC_{50}$  measures are protective of species. In fact, 50% of the population or measurement endpoint (e.g., growth, etc.) would be affected at an organism's  $LC_{50}/EC_{50}$  concentration. This analysis also assumes that laboratory-derived  $EC_{50}$  values are representative of the actual  $EC_{50}$  values that would be observed in the environment. Laboratory-derived values can be more conservative since they must eliminate many of the confounding factors that affect the bioavailability and fate of contaminants.

## Summary

Diquat does not remain dissolved in water for long periods of time following application. It quickly adsorbs to negatively-charged surfaces and functional groups. In the sorbed state, the half-life for diquat can be very long, but bioavailability is very low. Because diquat is an herbicide, plants are most susceptible to injury caused by diquat exposures. Benthic and detritus-consuming invertebrates are the most susceptible animals to injury caused by diquat exposures. Care should be taken when treating water bodies where these organisms comprise an integral trophic level.

## Recommendations for Optimal Use/Environmental Protection\*

- **Only apply to water bodies following label restrictions for control of algae and aquatic weeds. For dense weed infestations, treat only 1/3 to 1/2 of the water body (waiting 14 days between treatments) to minimize oxygen loss and fish suffocation due to weed decomposition.**
- Always follow the recommendations printed on the label. Pay special attention to the “Environmental Hazards” section.
- For emergent and ditch bank weed control, apply only to the foliage, not the water. Thorough coverage of the plants will result in optimal control since this is a contact herbicide.
- Use an appropriately sized course nozzle droplet size to minimize drift of fine droplets to non-target areas. Fine droplets are more prone to drift.
- Apply only to calm water bodies without excessive wind and wave action.
- Do not apply to muddy/turbid water. The constituents contributing to turbidity will sorb the diquat, rendering it ineffective for weed control.
- Ineffective weed control will result from applications to plants covered with sediment deposits.
- Do not apply to intertidal areas below the mean water mark.
- Only use proper measuring devices and appropriately calibrated application equipment.

\*Recommendations summarized from herbicide labels for Weedtrine-D® (Applied biochemists, Germantown, WI) and Reward® (Syngenta Crop Protection, Inc., Greensboro, NC).

## Glossary of Terms and Abbreviations

**Density:** Density of a material is defined as its mass per unit volume (Available on <http://en.wikipedia.org/wiki/Density>).

**Melting point:** The temperature at which a solid changes state to liquid (Available on [http://en.wikipedia.org/wiki/Melting\\_point](http://en.wikipedia.org/wiki/Melting_point)).

**Vapor pressure:** The pressure of a vapor in thermodynamic equilibrium with its condensed phases in a closed system (Available on [http://en.wikipedia.org/wiki/Vapor\\_pressure](http://en.wikipedia.org/wiki/Vapor_pressure)).

**Solubility:** The property of a solid, liquid, or gaseous chemical substance (called the solute) to dissolve in a liquid solvent to form a homogeneous solution of the solute in the solvent (Available on <http://en.wikipedia.org/wiki/Solubility>). Water solubility refers to the amount of the pesticide that will completely dissolve in a given volume of water.

**$K_d$ :** The partitioning coefficient ( $K_d$ ) is an indicator of the sorptive properties of a pesticide.

**$K_{ow}$ :** The octanol-water coefficient is defined as the ratio of a chemical's solubility in n-octanol and water at steady state.

**$K_{oc}$ :** The organic carbon partition coefficient is derived as:  $K_{oc} = K_d / F_{oc}$ , where  $F_{oc}$  is the fraction of organic carbon in the soil/sediment.

**pKa:** The symbol for the acid dissociation constant (or called acidity constant) at logarithmic scale (Available on <http://en.wikipedia.org/wiki/pKa>).

**Henry's law constant:** A partition coefficient defined as the ration of a chemical's concentration in air to its concentration in water at steady state.

**Half-life:** The amount of time needed for half of the chemical to disappear.

**$LC_{50}$ :** Median lethal concentration or lethal concentration of 50% refers to the concentration estimated to cause mortality in 50% of a test population over a specific period of time.



**EC<sub>50</sub>**: Median effective concentration or effective concentration of 50% is defined as the concentration of a chemical estimated to cause a specific effect (e.g., behavior) in 50% of a population of test species after a specific length of exposure.

**LOEC**: Lowest observed effect concentration (toxicology).

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