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Guidance proposal for using available DegT50 values for estimation of degradation rates of plant protection products in Dutch surface water and sediment

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J.J.T.I. Boesten, P.I. Adriaanse, M.M.S. ter Horst, A. Tiktak en A.M.A. van der Linden



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Guidance proposal for using available *DegT50* values for estimation of degradation rates of plant protection products in Dutch surface water and sediment

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J.J.T.I. Boesten

P.I. Adriaanse

M.M.S. ter Horst

A. Tiktak

A.M.A. van der Linden

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Abstract

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The degradation rate of plant protection products and their transformation products in surface water and sediment may influence their concentrations in Dutch surface water. Therefore the estimation of these rates may be an important part of the assessment of the exposure of aquatic organisms. We propose a stepped sequence of studies for estimating the rate in water going from simple and conservative to more sophisticated and more realistic studies. The sequence includes (i) studies on hydrolysis and photolysis, (ii) studies with fresh surface water in the dark, (iii) water-sediment studies in the dark or in light, (iv) studies with algae and macrophytes, and (v) outdoor studies in realistic surface water systems. The usefulness of these studies for the exposure assessment in Dutch surface water is discussed. For estimation of the degradation rate in sediments we propose to conduct studies in which the substance is mixed through the sediment.

Key words: plant protection products, degradation, photolysis, hydrolysis, surface water, exposure assessment

Authors:

J.J.T.I. Boesten, P.I. Adriaanse & M.M.S. ter Horst: Alterra, Wageningen UR

A. Tiktak: PBL Netherlands Environmental Assessment Agency

A.M.A. van der Linden: RIVM National Institute for Public Health and Environmental Protection

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P.O. Box 47, 6700 AA Wageningen, The Netherlands

Phone: +31 (0)317 48 07 00; e-mail: info.alterra@wur.nl

Netherlands Environmental Assessment Agency (PBL)

P.O. Box 47, 6700 AA Bilthoven The Netherlands

Phone: +31 (0)70 328 87 00; e-mail: info@pbl.nl

National Institute for Public Health and the Environment, RIVM.

P.O. Box 13720 BA Bilthoven The Netherlands

Phone: +31 (0)30 274 91 11; e-mail: info@rivm.nl

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Statutory Research Tasks Unit for Nature & the Environment, P.O. Box 47, NL-6700 AA Wageningen, The Netherlands
Phone: +31 317 48 54 71; e-mail: info.wnm@wur.nl; Internet: www.wageningenUR.nl/wotnatuurenmilieu

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Summary

The degradation rate of plant protection products and their transformation products in surface water and sediment may influence their concentrations in Dutch surface water. Therefore the estimation of these rates may be an important part of the assessment of the exposure of aquatic organisms. We propose a stepped sequence of studies for estimating the rate in water going from simple and conservative to more sophisticated and more realistic studies. This sequence starts with hydrolysis studies for the relevant pH range. Then the sequence shows a branch: the left branch contains studies in dark and the right branch studies in light. The left branch contains studies with fresh surface water and a suspended-sediment concentration of 10 mg/L in the dark which are followed by studies with fresh surface water and a sediment layer in the dark. The right branch continues with photolysis studies in buffered pure water followed by photolysis studies in fresh surface water (thus including indirect photolysis). Further options in the right branch are studies with water and sediment in light, studies with algae and macrophytes in light and outdoor studies in realistic surface water systems.

Given time limitations, we were unable to collect and analyse statistical data on light intensities in Dutch surface water. Therefore we do not recommend using studies with surface water in light for the Dutch exposure assessment except outdoor studies with the restriction to use expert judgement to assess the relevance of the studies. We recommend analysing available information on the light intensity in Dutch surface water for relevant wavelengths and using this information as a basis for future guidance for the use of laboratory photolysis studies in buffered pure water and laboratory photolysis studies in fresh water.

For estimation of the degradation rate in sediments we propose to conduct studies in which the substance is mixed through the sediment if this rate is a critical factor in the exposure assessment (otherwise the exposure assessment can be carried out assuming no degradation in the sediment).

Monitoring data on the properties of Dutch surface water provide relevant information for assessing the appropriateness of the conditions in degradation rate studies. Some 37 000 O₂ concentration measurements in Dutch surface water at about 3500 locations in 2009 showed that more than 99% of the concentrations were above 0.5 mg/L, indicating aerobic conditions. Some 43 000 pH measurements in Dutch surface water at about 3500 locations in 2009 showed that 96% of the pH values were between 7 and 9.5. Some 11 000 measurements of suspended matter in Dutch surface water at about 1500 locations in 2009 showed a median of 10 mg/L and a 90th percentile of about 40 mg/L.

1 Introduction

Exposure of aquatic organisms to plant protection products is an important part of the aquatic risk assessment in the Netherlands and in the EU. Tiktak *et al.* (2012) described an edge-of-field Dutch surface water scenario for application of plant protection products to arable soil. This scenario aims to estimate 90th percentile PEC concentrations of a certain spatial population of ditches considering multi-year simulations. In this scenario the behaviour of the substances is described with the TOXSWA model. The substances may degrade in surface water or sediment and this degradation may have an effect on the simulated peak and TWA concentrations in this scenario (especially in case of repeated applications). In the TOXSWA model degradation is described with first-order kinetics and with a degradation rate that is a function of temperature based on the Arrhenius equation. The reference temperature is 20°C.

For compounds that get into the higher tiers of the aquatic risk assessment, there is regularly a debate between regulators and applicants on which of the *DegT50* values to be used.

Hydrolysis, photolysis studies in water, and water-sediment studies (in the dark) are standard data requirements, so such studies are available in the dossier. The presence of plants in surface water may influence the degradation rate in water because they may influence e.g. the pH and the temperature. So one may expect that applicants will submit outdoor studies with plants and sediment in the foreseeable future for those plant protection products that need a higher-tier assessment of the degradation rate in water. Let us assume that such a set of *DegT50* values for water is available in a dossier (see Tables 1 and 2 for examples). Thus the problem is: which *DegT50* to select from such a set of values for the scenario calculations for exposure of aquatic organisms ?

To the best of our knowledge, there is at the EU level no written guidance on how the results of such complex *DegT50* sets should be used to assess *DegT50* values for exposure scenarios. FOCUS (2001) provided guidance on *DegT50* values for water and sediment to be used with the FOCUS surface water scenarios. FOCUS (2001) indicates that usually two water-sediment studies are available and recommends taking the average¹ *DegT50* value of the two studies. However, the surface water properties are not considered in this guidance. Let us assume that the *DegT50* for hydrolysis increases with pH as in the example of Table 1. Let us further assume that the two water-sediment studies were conducted with sediments in the low pH range (see examples of Table 1 and 2). In such cases the guidance by FOCUS (2001) is questionable: e.g. what is the certainty that, for the case of Table 2 (not considering the photolysis *DegT50* of 7 d at pH = 9), a *DegT50* of 90 d (*i.e.* average of 80 and 100 d) is valid for ditches with pH = 9 ?

So this small piece of guidance available at the EU level may be difficult to defend scientifically. FOCUS (2006) mentions hydrolysis and photolysis studies but limits itself to guidance on averaging of rates of the same types of study (see p. 238 of FOCUS, 2006).

Therefore it is advisable to start with developing guidance for estimating *DegT50* values to be used as input for the Dutch surface water scenarios from sets of *DegT50* values as shown in Tables 1 and 2. The endpoints resulting from this guidance are *DegT50* values for surface water and sediment to be used as input for the TOXSWA model for calculations with Dutch surface water scenarios. This guidance development is especially important for surface water bodies with low water velocities such as the water body of the edge-of-field scenario developed by Tiktak *et al.* (2012).

¹ After 2006, it has become common practice to use the geometric mean of the *DegT50* based on FOCUS (2006).

The alternative would be to set the *DegT50*s for water and sediment for the Dutch edge-of-field scenario always at conservative (*i.e.* very high) values (e.g. 1000 days) so that degradation does not play a role in the exposure assessment. However, this is difficult to defend for compounds such as captan that show fast hydrolysis between pH 5 and 9 (see Table 3).

As this is a pioneering activity within a limited time frame, the level of detail of the guidance will be limited (and as a consequence the guidance will, where necessary, have to be on the conservative side).

Table 1. First example of a set of DegT50 values in water in a registration dossier. Note that the photolysis study is performed at the pH with the slowest hydrolysis rate as prescribed by OECD (2008).

pH	DegT50 in water (d) at 20°C			
	hydrolysis study	water-sediment study in the dark	study on direct photolysis in water (OECD, 2002)	outdoor plant-water-sediment studies
5	30	20		
7	50	40		20
9	70		10	

Table 2. Second example of set of DegT50 values in water in a registration dossier. Note that the photolysis study is performed at the pH with the slowest hydrolysis rate as prescribed by OECD (2008).

pH	DegT50 in water (d) at 20°C			
	hydrolysis study	water-sediment study in the dark	study on direct photolysis in water (OECD, 2002)	outdoor plant-water-sediment studies
5	300	100		3
7	400	80		
9	>500		7	

Table 3. Hydrolysis half-lives of captan at 20 °C (source: Footprint database accessed at 6 March 2014).

pH	half-life (d)
5	0.15
7	0.6
9	0.004

2 Guidance for estimating the *DegT50* for degradation in Dutch surface water

2.1 Proposed hierarchy for using different types of study to estimate the *DegT50*

Below guidance is described for deriving the *DegT50* in surface water from different types of study via a stepped approach. The rationale for this approach is that (i) such a stepped approach should go from simple studies that give conservative estimates of the degradation rates to more realistic studies that give less conservative estimates of the degradation rates to be used in the exposure calculations, (ii) hydrolysis is only a function of pH and thus will take place also in any of the studies in the higher steps (with rate dependent on the pH), (iii) measurements with fresh surface water in the dark are easier to perform and to interpret than all other studies except the hydrolysis studies, (iv) photolysis studies are easier to interpret than all other studies except the hydrolysis studies and the studies with fresh surface water in the dark.

The hierarchy of these estimates is as follows (see Figure 1):

- the first step is the *DegT50* resulting from hydrolysis
- in the second step, both degradation rate studies with fresh surface water in the dark and photolysis studies can be taken into account
- the third step is a *DegT50* resulting from more sophisticated studies as indicated.

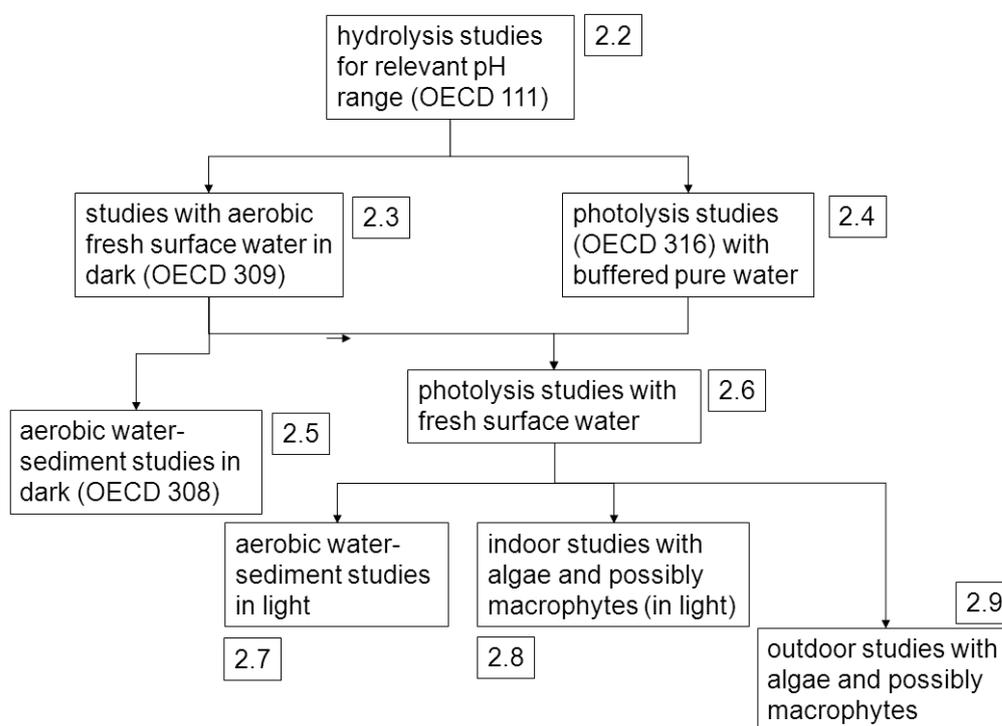


Figure 1. Stepped approach for estimating *DegT50* values for surface water to be used in the Dutch surface water scenarios. The numbers refer to the sections in which the use of the endpoint of the corresponding type of study is discussed.

We provide no guidance for deriving the *DegT50* values themselves from experimental data. So we assume that the available *DegT50* values have been derived in an acceptable way based on e.g. FOCUS (2006).

2.2 Hydrolysis

Considerations

The hydrolysis rate coefficient of many substances depends strongly on the pH and it may both increase and decrease with pH (Deneer *et al.*, 2010). So to estimate this coefficient we need information on the pH in Dutch ditches. Portielje & Roijackers (1995) measured the pH over a four-year period in four Dutch experimental ditches with different nutrient inputs. In the ditch with the highest input, the pH fluctuated between 5 and 11 and in the other three ditches it fluctuated between 7 and 10. Vink (1997, p. 100) collected 11 surface water samples in Southern Flevoland and found a pH range from 7 to 8. About 42,000 pH measurements in Dutch surface waters (Appendix A) showed that only in 2% of the measurements the pH is below 7 and that only in 2% of the measurements the pH is above 9.5. So for a 90th percentile worst case scenario, it seems defensible to assume that the pH is in the range between 7 and 9.5.

Hydrolysis rates are measured in sterile buffered systems as described in guideline OECD-111 (OECD, 2004b). The buffer itself may increase or decrease the measured degradation rate in water. So there is some uncertainty with respect to extrapolating degradation rates measured in buffer solution to rates measured in pure water.

Proposed guidance

An estimate of the half-life resulting from hydrolysis can only be derived from hydrolysis studies that cover the range from 5 to 9 because rates for this range are usually available from the dossier. It is recommended to estimate the longest *DegT50* in the pH range from 7 to 9.5 from the available measurements and to calculate this back to a temperature of 20°C using the temperature dependencies as measured in the hydrolysis studies. If these were not measured, it is recommended to assume an Arrhenius activation energy of 75 kJ/mol, which is the average of the range of 50 to 100 kJ/mol given by Deneer *et al.* (2010). Admittedly, this 75 kJ/mol is a best-guess value with limited underpinning.

Let us consider the example of the hydrolysis half-lives of captan in Table 3. In this case the half-life of 0.6 d is selected for the first step, being the largest *DegT50* value for pH values above or equal to 7.

2.3 Studies in the dark with aerobic fresh surface water only

Considerations

Studies with freshly sampled surface water incubated in the dark may be used to estimate the microbial degradation rate in water (OECD, 2004a). This OECD-309 guideline prescribes that such studies should be conducted in flasks that are filled with a water volume that is at most 1/3 of the total volume so leaving at least 2/3 of the volume for air (to keep the system aerobic, *i.e.* above 0.5 mg O₂/L). It recommends further that studies should preferably start within 1 day after collection of the surface water (allowing storage for at most 4 weeks at 4°C).

One may expect that the oxygen concentration in the surface water has a distinct effect on the degradation rate. As described above, the OECD-309 guideline prescribes aerobic incubation. Some

37000 measurements of oxygen concentrations in Dutch surface waters (Appendix A) show that in more than 99% of the cases concentrations exceeded 0.5 mg/L indicating that the water in nearly all of the Dutch agricultural ditches is also aerobic. So the oxygen concentrations in the water in the OECD-309 test can be considered to be sufficiently close to the conditions in the field

The OECD-309 guideline offers two options for performing the test: a 'pelagic test' or a 'suspended sediment test'. In a pelagic test no suspended sediment is added and in a suspended-sediment test sediment is added at concentrations ranging from 0.01 to 1 g/L. There are more than 10 000 measurements available on the suspended-matter concentration of Dutch surface water and the median of these is about 10 mg/L, *i.e.* 0.01 g/L (Appendix A). So it seems advisable to perform the test as a suspended-sediment test at a sediment concentration of 0.01 g/L.

Vink (1997) found sometimes rapid degradation of aldicarb and MCPA in Dutch surface waters in the first week after application (in studies with fresh surface water in the dark). His study shows that such studies may result in shorter half-lives than those estimated on the basis of hydrolysis studies. So this illustrates that studies with fresh surface water may have added value in the stepped approach of Figure 1.

Proposed guidance

We propose to require studies based on the OECD-309 protocol for a number of surface water samples. The only requirement is that the samples can be considered relevant for Dutch surface water (which does not exclude use of water samples from other countries). The samples should cover the range of relevant pH values (ie from 7 to 9.5). The water should be aerobic and studies should not last longer than a few weeks to ensure adequate microbial activity. We recommend performing the test as a suspended sediment test with a sediment concentration of 0.01 g/L.

In case the temperature of the study differs from 20°C, we recommend to correct the *DegT50* using an Arrhenius activation energy of 75 kJ/mol (same value as for the hydrolysis studies). From all available *DegT50* values (derived from these OECD-309 studies and calculated back to 20°C), the geomean *DegT50* should be calculated.²

2.4 Photolysis studies in buffered pure water (direct photolysis)

Considerations

OECD 316 (2008) provides guidance for studies on direct photolysis of chemicals in buffered pure water. OECD 316 prescribes that for non-ionisable test substances, photolysis studies in water have to be conducted at the pH at which the substance is hydrolytically most stable in the pH range 4-9 (see point 39 of OECD, 2008). As described in OECD 316 it is in principle possible to relate the *DegT50* values to the intensity of the sunlight.

Figure 2 shows that daily global radiation fluctuates strongly in spring in the Netherlands. So any approach to calculate photolysis rates will have to include the dependency of the photolysis rate coefficient on the intensity of the sun light or to use conservative estimates of this global radiation (Figure 2 suggest that minimum values are usually not less than 20% of the maximum).

² The underlying principle of this geomean estimation is that it is the best estimator of the median of the statistical population of *DegT50* values. The guidance for estimating the median of a statistical population is currently under discussion at EU level. Anonymous (2011) recommends to use the geomean if less than 9 values are available and to use the median of the sample population for 9 or more values. EFSA (2012) recommends to use a geomean irrespective of the number of values. It is recommended to use the most recent guidance.

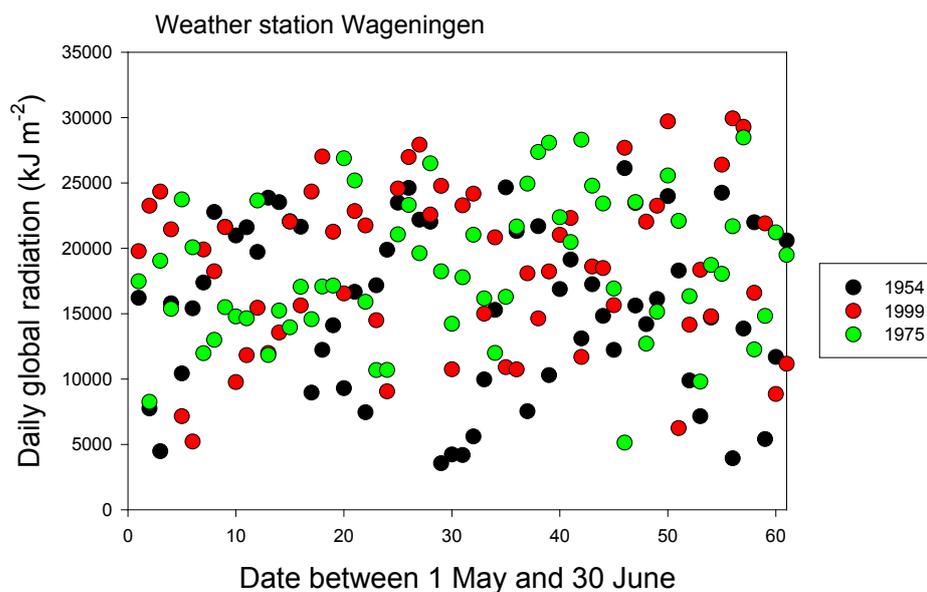


Figure 2. Daily global radiation in spring as measured at weather station Wageningen in three different years. Note that 10000 kJ/m^2 corresponds with 1000 J/cm^2 .

In principle, it would be possible to use direct photolysis rates, as derived from OECD 316 tests, to assess the *DegT50* in Dutch surface water. However, for that purpose it would be necessary to have appropriate information on the light intensity in Dutch surface water systems. One of the issues is that the photodegradation rate decreases with depth in surface water because the light intensity decreases with depth. The light in the so-called UVB range (i.e. 280-320 nm) is usually considered the most relevant part of the light with respect to generating photolysis (e.g. Wanatabe & Takagi, 2000). De Lange (1999) measured the extinction of UVB light with depth in a number of Dutch surface waters including seven ditches in spring 1998. The extinction was characterised by the depth at which still 1% of the UVB light intensity at the water surface was measured. The seven ditches were from three distinctly different locations and included three experimental ditches from one location. This 1%-depth of the seven ditches ranged between 7 and 46 cm and the median 1%-depth was 12 cm. The depth was influenced by the concentrations of (i) humic substances, (ii) organic particulate matter, and (iii) inorganic particulate matter. These measurements show that UVB light usually does not penetrate deep. Another issue is the coverage of Dutch surface water systems with duckweed in spring and summer. This may reduce considerably the amount of light (including UVB) that penetrates into the water at the surface; this is further discussed in Section 2.11.

Deneer *et al.* (2010) reviewed available approaches for describing photodegradation rates in surface water and provided also recommendations for including photodegradation rates in the TOXSWA model. They recommend to perform simulations with a maximum possible photodegradation rate based on optimum conditions and to compare this with simulations without photodegradation, considering that the reality has to be in between. This is an interesting suggestion but does not solve the problem of estimating photolysis rate in the Dutch surface water scenarios.

These considerations apply only to the degradation rate of the parent molecule. For the formation of a photometabolite this reasoning is not relevant. The realistic worst case for formation of a photometabolite is perfectly clear water because the photolysis degradation rate of the parent is highest in such water. Part of the Dutch surface water is likely to be clear.

Proposed guidance

Given the above lack of readily available information and the limited time available to the workgroup, we propose not to use laboratory measurements on photodegradation for estimating the *DegT50* as input for the Dutch surface water scenarios. However, we propose to use such measurements for the identification of photometabolites and the estimation of their formation fractions.

Given the information available it is probably possible to develop methods for using the results from the OECD study to extrapolate to other light conditions and thus to estimate the *DegT50* in Dutch surface water. We recommend developing such methods.

2.5 Studies in aerobic water-sediment systems in the dark

Considerations

Within the context of this guidance, the rationale for conducting water-sediment studies in the dark is to measure a degradation rate in water under more realistic conditions than in the hydrolysis studies which is expected to give a degradation rate in water that is faster than the hydrolysis rate (under otherwise comparable conditions including the pH). This faster rate may be the result of presence of micro-organisms or substances that facilitate degradation in water. So the point is whether the water-sediment studies are in this respect representative of the range of Dutch surface waters.

For many plant protection products the pH is one of the most important properties of surface water influencing the degradation rate of plant protection products. The OECD guideline 308 (OECD, 2002) gives only vague recommendations on the pH of the sediment to be tested. Citation from point 25 of OECD (2002): "Other parameters may be important in the selection of sediments and should be considered on a case-by-case basis. For example, the pH range of sediments would be important for testing chemicals for which degradation and/or sorption may be pH-dependent." Moreover OECD (2002) does not require that the pH of the water is measured. It is of course possible that the pH of the water layer is not equal to that of the sediment. So there is even no guarantee that the pH of the water in the water-sediment study is known (although this will usually be measured).

Also the oxygen concentration in the water may influence the degradation rate in the water phase. OECD (2002) offers two options for conducting such a study: aerobic or anaerobic. The difference is the composition of the headspace of the water-sediment system (normal air or nitrogen gas). In view of the Dutch oxygen monitoring data in Appendix A, we consider only the aerobic studies meaningful. For the aerobic systems, air is led through the system so most of the water layer is expected to be aerobic.

For water-sediment studies (OECD guideline 308), OECD (2002) recommends using freshly sampled sediment and water but if storage is necessary, sediment and water should be stored together, water-logged (6-10 cm water layer), in the dark, at 4 °C for a maximum of 4 weeks. The period of acclimation should normally last between one week and two weeks and should not exceed four weeks. For studies with aerobic fresh surface water (OECD Guideline 309), OECD (2004a) recommends starting the study within 1 day after collection of the surface water but allows for four weeks of storage. So it is very well possible that an OECD-309 study starts 1 day after collection of the water whereas an OECD-308 study starts five weeks after collection of the water and the sediment. Thus the microbial activity in such a OECD-309 study may be closer to the microbial activity in the water in the field than the microbial activity in the water of an OECD-308 study. So there is no reasonable certainty that the *DegT50* in the water layer of a water-sediment study is closer to the reality in the field than the *DegT50* in an OECD-309 study.

FOCUS (2006) described guidance on how to derive degradation rates in water and in sediment from these water-sediment studies. However, experience with this guidance has shown that current water-sediment study designs are frequently insufficient for required kinetic applications (Beigel *et al.*, 2010). The guidance by FOCUS (2006) ensures that a conservative *DegT50* for the water phase is derived from these studies. This is done by recommending to perform two PEC calculations: in the first calculation it is assumed that the *DegT50* in water equals the system half-life and that the *DegT50* in sediment is 1000 d whereas in the second calculation it is assumed that the *DegT50* in water is 1000 d and that the *DegT50* in sediment equals the system half-life. Thereafter the highest PEC of the two is taken. The consequence of this procedure is that often the *DegT50* in water derived from these studies is longer than that derived from the studies in the lower steps. The obtained *DegT50* may be even longer than the hydrolysis rate at the pH of the water in the water-sediment study which is of course an irrelevant result in the context of the stepped approach of Figure 1. This may be prevented by using the *DegT50* derived from the hydrolysis rate studies (Section 2.2) as a maximum value for the *DegT50* in water. So whilst the water-sediment study is considered a higher tier than e.g. the hydrolysis study, the impossibility to derive an accurate *DegT50* in water from the water-sediment study makes the water-sediment study less useful. However, there may be cases where appropriate *DegT50* values for the water phase can be estimated and there is of course also the option to use the system half-life to estimate the *DegT50* in water (as one of the two PEC calculations as described by FOCUS, 2006). In such cases the guidance in the following paragraph is proposed.

Proposed guidance

We recommend accepting a water-sediment study only if the pH in the water was between 7 and 9.5 in view of the pH measurements in Dutch surface water cited in Section 2.2. Only water-sediment studies carried out with an aerobic composition of the headspace are considered relevant. All *DegT50* values should be calculated back to 20 °C using the Arrhenius activation energy of 75 kJ/mol (average value of the range 50-100 kJ/mol as reported by Deneer *et al.*, 2010, for hydrolysis rates in water). Thereafter a geomean *DegT50* is calculated from these water-sediment studies and used in the exposure assessment.

2.6 Photolysis studies with fresh surface water

Photolysis studies with fresh surface water performed in the laboratory are expected to give faster photolysis rates than studies with buffered pure water (under the same light regime) because indirect photolysis may occur as well in such a system. At this moment there seems to be no guidance at OECD level for performing such studies.

With respect to the use of such studies, there is the same problem as with the photolysis studies in buffered water (2.4): we cannot extrapolate to the light conditions in Dutch surface water because we have insufficient readily available information on these conditions. Moreover indirect photolysis depends not only on the light conditions. The mechanism of this process is that light is absorbed by another molecule (a photosensitizer, e.g. dissolved organic matter), generating a transient molecule (e.g. oxygen- or hydroxyl-radicals; Zepp, 1992) which transfers the energy to the molecule of the plant protection product, causing the latter to break down. The photosensitizer may vary between plant protection products and between locations (which may have different dissolved organic matter material). Therefore extrapolation of indirect photodegradation rates from one type of surface water to another is difficult. Moreover, different plant protection products may have different photosensitizers, so information gained on indirect photolysis for a certain plant protection product cannot be easily extrapolated to other plant protection products. So we recommend not using the results of these studies for estimating degradation rates in Dutch surface water.

2.7 Studies in water-sediment systems in light

With these studies, there is the same problem as with the photolysis studies: we cannot extrapolate to the light conditions in Dutch surface water because we have insufficient readily available information on these conditions. All the problems mentioned above for the water-sediment study in the dark apply also to these studies. So we recommend not using the results of these studies for estimating degradation rates in Dutch surface water.

2.8 Laboratory studies with algae and possibly macrophytes in light

It is practically impossible to perform studies with macrophytes but without algae. So we have either studies with algae or studies with both algae and macrophytes. At this moment not enough experience with these studies has been gained to be able to use them in the Dutch exposure assessment.

2.9 Studies in outdoor surface water systems with algae and macrophytes

Considerations

Plant protection products may degrade in surface water by (photo)chemical and microbial processes. Aquatic plants (macrophytes) may influence these processes and so also the degradation rate of these substances. The density of macrophytes in Dutch surface water varies strongly in time: surface water systems are cleaned in winter and the density of the plants increases strongly between about 1 March and 15 May. The maximal dry mass of plant material per area of surface water varies strongly from species to species (Brock, 1988). We are not aware of reliable information on the frequency distribution of the dry mass of plant material per area of surface water as a function of time for the different Dutch surface water systems. We cannot exclude that the *DegT50* in the water varies strongly with the type of plant. We have no information readily available on the distribution of plant biomass over the different types of plants in Dutch surface waters.

Also algae may increase the degradation rate in surface water (see e.g. O'Kelley & Deason, 1976; Caceres *et al.*, 2008). One may expect that algae occur in all Dutch surface waters and thus they will also occur in outdoor studies.

Outdoor studies with plants include also photolysis. As discussed before, there is not enough certainty to extrapolate photolysis rates measured in clear water to the full range of Dutch surface water. On the other hand it seems not defensible to ignore outdoor studies if these demonstrate a rapid degradation under a range of relevant conditions for low densities of the water plants. This may seem inconsistent with our recommendation not to use photolysis studies in the laboratory in both buffered pure water and fresh surface water. However, we consider these outdoor studies more valuable because they are much closer to the reality than the laboratory studies (where e.g. artificial light is used).

It is generally assumed that the rate coefficient for direct and indirect photodegradation is proportional to the product of the molar absorption coefficient and the light intensity at a certain wavelength (e.g. OECD, 2008). Usually meteorological stations provide only daily global radiation. As an approximation we consider it defensible to assume that the rate coefficient for photodegradation is proportional to daily global radiation:

$$k = k_{ref} \left(\frac{G}{G_{ref}} \right) \quad (1)$$

where k is the rate coefficient for photodegradation (d^{-1}), k_{ref} is the k at a reference global radiation G_{ref} (J cm^{-2}) and G is the daily global radiation (J cm^{-2}). So it is assumed that the daily UVB radiation is proportional to the daily global radiation (see Section 2.4). Eqn 1 gives for the $DegT50$:

$$DegT50 = DegT50_{ref} \left(\frac{G_{ref}}{G} \right) \quad (2)$$

where $DegT50_{ref}$ is the $DegT50$ at the reference daily global radiation. Daily global radiation varies in the Netherlands typically from 200 J cm^{-2} in winter to 2000 J cm^{-2} in summer (Velds, 1992). Therefore we propose to use a G_{ref} of 1000 J cm^{-2} . This reference value of the global radiation is an arbitrary value needed to standardise $DegT50$ values obtained from different outdoor experiments (e.g. a $DegT50$ of 10 d at $G = 1000 \text{ J cm}^{-2}$ is identical to a $DegT50$ of 5 d at $G = 2000 \text{ J cm}^{-2}$, so if different $DegT50$ values have to be averaged, they have to be calculated back first to the same reference G value). The G value that should be used to characterise the photodegradation rate in the scenario is a different matter. This has to be based on the radiation conditions to be expected under the scenario conditions. The $DegT50$ for the scenario ($DegT50_{sc}$) can be estimated with

$$DegT50_{sc} = DegT50_{ref} \left(\frac{G_{ref}}{G_{sc}} \right) \quad (3)$$

where G_{sc} is the daily global radiation of the scenario.

Proposed guidance

From photolysis studies with buffered pure water and/or with fresh surface water, it has to be assessed whether the substance may be degraded through direct or indirect photolysis or whether this is considered unlikely.

If it is unlikely that the substance degrades through direct or indirect photolysis only $DegT50$ values should be considered that are derived from outdoor studies with 1) different plant species, or 2) different plant communities or 3) at least two studies with algae. Only relatively low densities of the water plants and algae are considered acceptable in these studies. The $DegT50$ of each study has to be calculated back to 20°C using an Arrhenius activation energy of 75 kJ/mol (ie the average value of the range $50\text{-}100 \text{ kJ/mol}$ as reported by Deneer *et al.*, 2010, for hydrolysis rates in water). The geomean $DegT50$ should be calculated from all $DegT50$ values at 20°C derived from these outdoor studies. The $DegT50$ for the scenario should not be corrected using Eqn 3 because there is no direct relationship between the light intensity and the degradation rate for substances that are not liable to photodegradation.

If the substance degrades mainly through direct or indirect photolysis, expert judgement will be required to assess whether such an outdoor study is relevant; the main factor to be considered in such an assessment is the comparison between the light conditions in the study and those in a 90th percentile case in Dutch surface water (after correcting for the difference in daily global radiation). In this comparison also some judgement on the depth of penetration of the UVB light should be included (see discussion in third paragraph of Section 2.4). If several studies are available that are considered relevant, then the geomean $DegT50_{ref}$ should be taken.

In case of indications of a pH dependency of the $DegT50$, not the geomean should be used but the estimation of the $DegT50$ should be based on expert judgement.

For substances that reach the surface water also via drainpipes, we propose $G_{sc} = 800 \text{ J cm}^{-2}$, i.e. the annual median at weather station 'de Bilt' (Velds, 1992) because leaching from drainpipes may occur during the whole year. For substances that do not reach the surface water via drainpipes due to strong sorption (like example substance I_p ; Tiktak *et al.*, 2012), we recommend using the monthly median global radiation at weather station 'de Bilt' in the month of application (as given in Table 4.5 of Velds, 1992). In case there are repeated applications in several months, we recommend to use the average of the monthly medians.

2.10 Overview of possibilities for assessing the *DegT50* for Dutch surface water

In view of the foregoing not all steps shown in Figure 1 can be used to derive *DegT50* values for surface water in the Dutch exposure assessment. Figure 3 gives an overview of the possibilities of the use of the different steps. The hydrolysis half-life at the pH with the slowest hydrolysis rate within the pH range from 7 to 9.5 will always be available to be used for estimating the *DegT50* in Dutch surface water. Studies with aerobic fresh surface water in the dark will usually not be available in present dossiers but they have become an Annex-II data requirement at EU level as per January 2014³.

The water-sediment studies in the dark are available. Outdoor fate studies will usually not be available stand-alone in the dossier but they are likely to be available as the fate part of mesocosm studies for substances that require a higher-tier effect assessment.

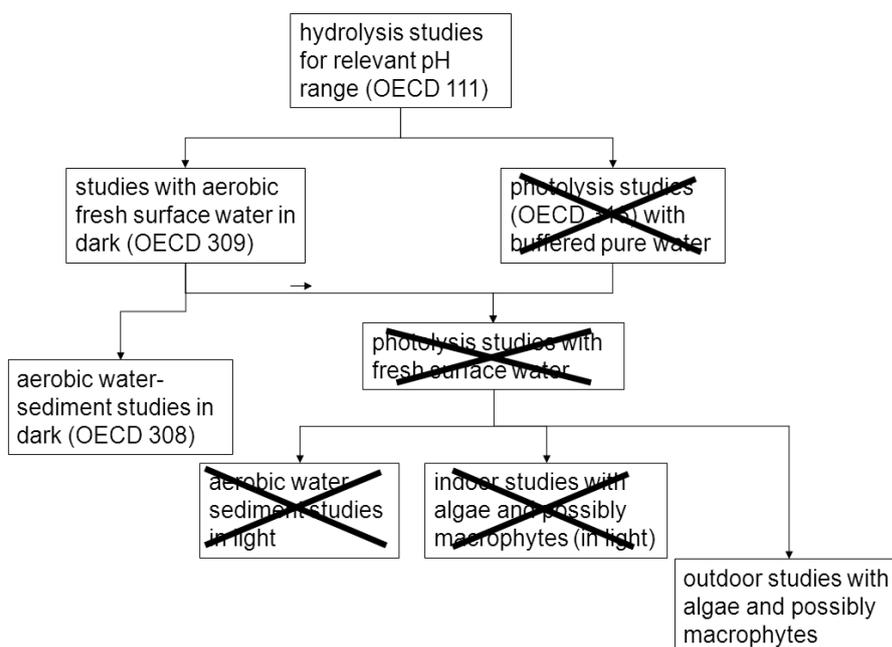


Figure 3. Stepped approach for estimating *DegT50* values for surface water to be used in the Dutch surface water scenarios. The steps with the crosses cannot be used based on the proposed guidance.

³See Commission Regulation (EU) No 283/2013 of 1 March 2013 setting out the data requirements for active substances, in accordance with Regulation (EC) No 1107/2009 of the European Parliament and of the Council concerning the placing of plant protection products on the market.

2.11 Considerations on the effect of coverage with duckweed

Coverage of Dutch surface water systems with duckweed in spring and summer may reduce the light intensity in the water considerably. This coverage is a scenario property which is relevant for substances that are susceptible to degradation induced by light. E.g. in outdoor Dutch small ponds, a dissipation half-life of about 60 d was observed for imidacloprid for a pond completely covered with duckweed whereas the dissipation half-life in a similar pond under the same conditions but without any duckweed (so a transparent surface) was about 6 days (G. Arts, personal communication, 2012; see also http://www.pfmodels.org/downloads/EMW6_13_Jos.Boesten_Tiered_approach_for_estimating_the_DegT50.pdf). So it does not make sense to use a *DegT50* based on photolysis for surface water systems completely covered with duckweed. So it is useful to assess the likelihood of coverage of Dutch ditches with duckweed. Data on this coverage are available in the Limnodatabase Neerlandica for the period 1981-2006. These are based on recordings on abundance/coverage in the Tansley or Braun-Blanquet scale considering the non-rooting floating plants *Azolla*, *Lemna*, *Spirodella* and *Wolffia* (see for more details Peeters *et al.*, 2013). The database contained 5741 measurements but the width of the ditch was available for only 1553 coverage measurements. From these 1553 measurements we selected those for ditches with widths equal to or less than 6 m because ditches that are wider than 6 m are not relevant for the exposure assessment. Also ditches with widths less than 1 m were excluded because the databased even reported measurements for ditches with widths of 5 cm which seem not relevant (only 24 ditches had widths less than 1 m). This resulted in 1079 remaining measurements.

The Dutch exposure assessment methodology for aquatic organisms has as target a 90th percentile case (Tiktak *et al.*, 2012). This 90th percentile is likely to occur in a small ditch because small ditches are shallower than wide ditches and shallow ditches lead to a higher concentration in surface water resulting from spray drift deposition. Therefore the 1079 measurements were split into the ditches with widths of 1-3 m (502 measurements) and those with widths of 3-6 m (577 measurements).

Figure 4 shows that the coverage of the 1-3 m ditches increases during the year starting from an almost negligible coverage in May to about 30% of the ditches being covered for more than 75% in September. Figure 5 shows that the 3-6 m ditches had lower coverages than the 1-3 m ditches and that the percentage ditches with only 0-25% coverage increased again in September. The reason for this is not clear.

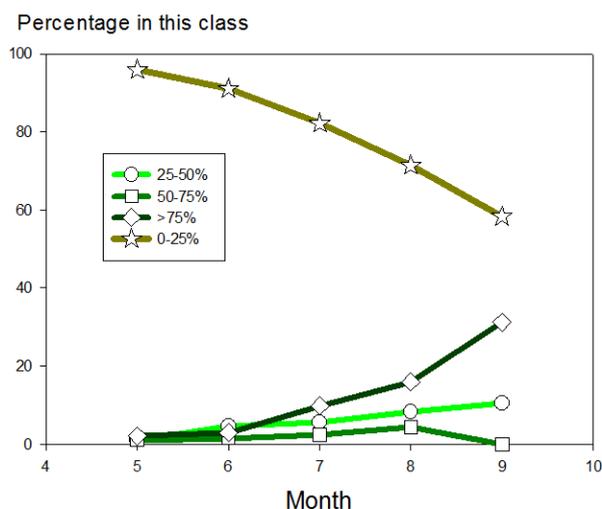


Figure 4. Four classes of percentages of duckweed coverage (0-25%, 25-50%, 50-75%, >75%) in Dutch ditches as a function of month in the year. Months 5 to 9 are May to September. For each month the classes sum up to 100%. The data are 577 measurements for ditches with widths between 1 and 3 m.

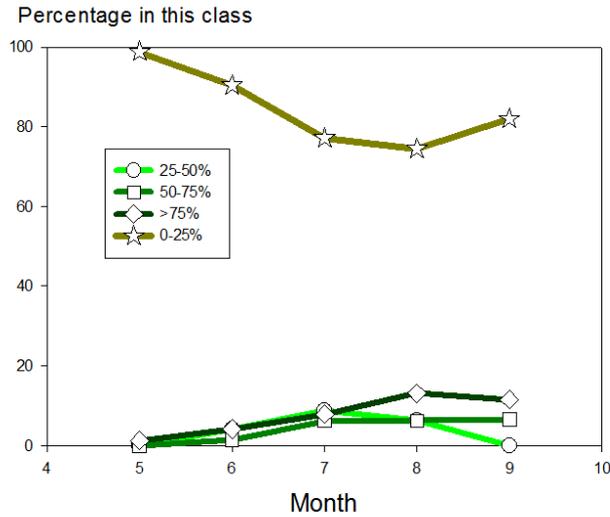


Figure 5. Four classes of percentages of duckweed coverage (0-25%, 25-50%, 50-75%, >75%) in Dutch ditches as a function of month in the year. Months 5 to 9 are May to September. For each month the classes sum up to 100%. The data are 502 measurements for ditches with widths between 3 and 6 m.

Tiktak *et al.* (2012) developed an exposure assessment methodology for aquatic organisms aiming at a 90th percentile case considering variation of exposure concentrations in space and time. The 90th percentile case in this methodology is based on spatial variation of properties of the soils and the ditches and on temporal variation of weather conditions. So temporal or spatial variations in substance properties such as the degradation rate in surface water were not considered in the methodology. The usual procedure is then to use a median value of such substance properties (EFSA, 2012). This approach will work well only when spatial and temporal variations in substance properties have no dominant effect on the spatial and temporal variation in exposure concentrations. If these substance properties have a dominant effect, the correct procedure would be to derive the 90th percentile case based on spatially distributed modelling considering this variation in substance properties. With respect to the effect of coverage by duckweed this would mean simulations considering the probability of coverage with a *DegT50* that is function of coverage ranging from a *DegT50* based on photolysis rates for 0% coverage to a *DegT50* from a study in the dark for 100% coverage (and including the effect of temporal variations in solar radiation on the photolysis rates). This function could as a first approximation be based on the following equation for the rate coefficient, k :

$$k = \delta k_{dark} + (1 - \delta) k_{light} \quad (4)$$

where δ is the fraction of the surface covered with duckweed (-), k_{dark} is the degradation rate coefficient under dark conditions (d^{-1}) and k_{light} is the degradation rate coefficient under light conditions (d^{-1}). The basis of Eqn 4 is that it divides the surface water into two compartments with each their own rate coefficient and that the overall degradation rate is the sum of the degradation rates in the two compartments. It can be derived from Eqn 4 that:

$$DegT50 = \frac{DegT50_{light} DegT50_{dark}}{\delta DegT50_{light} + (1 - \delta) DegT50_{dark}} \quad (5)$$

where $DegT50_{dark}$ is the degradation half-life under dark conditions (d) and $DegT50_{light}$ is the degradation half-life under light conditions (d^{-1}).

This approach is as yet impossible because no such spatially distributed modelling tool exists for Dutch surface water (see EFSA, 2012, for a similar discussion on exposure of soil organisms). In the absence of simulations with such a tool, it is somewhat speculative to assess the effect of the coverage of duckweed as shown in Figures 4 and 5 on the acceptability on using a photolysis-based *DegT50* in the Dutch exposure assessment for aquatic organisms. However, based on expert judgement we consider it justifiable to do so as long as at least 90% of the ditches has a coverage less than 25%. Figures 4 and 5 show that this is the case in the months May and June but not thereafter.

Besides its effect on the photodegradation rate in the surface water, coverage of the surface with duckweed has also another influence on the exposure concentrations: the spray drift deposition will not completely enter the ditch directly but part of it will be retained on the surface of the duckweed where it may photodegrade, volatilise or be taken up by the duckweed before it is washed off from the duckweed by rainfall or by waves in the water. This retention on the surface of the duckweed will lead to a reduction of concentrations in surface water which may compensate to some extent the effect of the decreased photolysis rate or even overrule this effect. So for a realistic inclusion of the effect of coverage by duckweed in the exposure assessment it would be necessary to develop a 'duckweed module' in TOXSWA that considers these processes together with guidance on how to assess the substance parameters describing the processes on the duckweed surface. This is now at least one bridge too far.

In view of these considerations, we recommend for the time being not to include the effect of coverage of duckweed in the Dutch exposure assessment of aquatic organisms. However, the data in Figures 4 and 5 show that coverage with duckweed may have a significant effect on the loading of the surface water resulting from spray drift for applications in July-September and on photolysis rates in the surface water in this same period. The photolysis rates in this period may have a distinct effect on the persistence of a considerable fraction of the pesticides in Dutch surface water. This is likely to have also a distinct effect on the outcome of the chronic risk assessment as proposed by Brock *et al.* (2011) and EFSA (2013). Therefore we recommend for the longer term (i) to develop and test such a duckweed module in TOXSWA and also (ii) to develop a spatially distributed modelling tool that allows inclusion of substance-specific aspects in the assessment of the 90th percentile exposure case.

3 Estimating the *DegT50* for sediments in Dutch surface water

The *DegT50* in sediment is set to infinity as a first step. For the next step, it is proposed to measure the *DegT50* in a number of sediments. No OECD guidance has been developed so far for conducting studies on the degradation rate in sediments. These sediments should have properties (pH, texture, organic matter, redox potential) that are in the range of those found in Dutch surface water sediments. The substance should be mixed through the sediment which should be kept water-saturated but with a water layer not thicker than about 5 mm. All *DegT50* values should be calculated back to 20°C using the Arrhenius activation energy of 65 kJ/mol found by EFSA (2007) for degradation in soil. Thereafter a geomean *DegT50* is calculated and used in the exposure assessment. As with the *DegT50* for surface water, we assume that each *DegT50* value is estimated sufficiently accurately from the experimental data.

4 Assessment of uncertainty in PEC resulting from limited number of *DegT50* values in dossiers

In the previous sections, it is recommended to calculate geomeans of sample populations of *DegT50* values (i) in aerobic fresh surface water in the dark, (ii) in the water layer of aerobic water-sediment systems in the dark, (iii) in outdoor surface water systems and (iv) sediments. For small numbers of *DegT50* values the uncertainty in this geomean may be large. If risk managers are interested in this uncertainty, we recommend performing PEC calculations with selected percentiles of the distribution of the geomean (e.g. the 90th percentile *DegT50*). EFSA (2012) suggested a similar procedure for assessing the uncertainty in the geomean *DegT50* and K_{om} for the exposure assessment for soil organisms.

We propose to assume for such calculations that all these types of *DegT50s* are lognormally distributed with a coefficient of variation (CV) of 0.5. This CV is recommended by EFSA (2010) for the *DegT50* for degradation in top soil. It would be of course better to base this CV on measurements of the *DegT50* in the aforementioned systems but we are not aware of datasets of such measurements that are suitable for estimating this CV.

5 Recommendations for future activities with respect to the assessment of the degradation rate in Dutch surface water and sediments

Probably we can do more with the photolysis studies if we can obtain reliable information on the light conditions in Dutch surface water. We recommend exploring whether such information is available. Moreover we recommend (i) exploring the possibilities of describing the direct photolysis rate in the TOXSWA model as a function of the light intensity based on the literature review by Deneer *et al.* (2010), and (ii) implementing the description of the light intensity in the water layer of the TOXSWA model on the basis of readily available meteorological information (e.g. hourly values of meteorological data) using the concepts proposed by Jacobs *et al.* (2010). Furthermore we recommend to develop and test a TOXSWA duckweed module and to develop a spatially distributed modelling tool that allows inclusion of substance-specific aspects in the assessment of the 90th percentile exposure case.

Deneer *et al.* (2010) showed that daily pH fluctuations in ditches with macrophytes in summer are typically 1-2 pH units in spring and summer. It is not clear to what extent these daily fluctuations may influence the overall hydrolysis rate in Dutch surface water. Therefore we recommend (i) implementing the description of the hydrolysis rate as a function of pH in the TOXSWA model as proposed by Deneer *et al.* (2010) and (ii) exploring the possibilities of describing the daily course of pH in Dutch surface water in this model.

In view of the limited experience with using outdoor systems for estimating *DegT50* in surface water, we recommend (i) making an inventory of all such outdoor studies available in regulatory dossiers and in open literature, (ii) selecting a few substances for which a large number of studies is available, (iii) estimating the *DegT50* for all studies of these selected substances, (iv) analysing all information and providing more detailed guidance for the use of such studies based on this, (v) providing recommendations for better designing studies.

We recommend developing a protocol for an indoor test study with fresh surface water containing algae at a normal concentration and at average light intensities in spring for the area of arable land of northern Europe. The background is that algae are expected to be present in all surface waters and that rates measured in northern Europa are considered conservative estimates of rates in the regulatory zone centre. So a degradation rate coefficient derived from such a study could be seen as a realistic minimum value for exposure assessments in the Netherlands and at EU level, at least for the central and southern zone.

We recommend further studies on the effect of temperature on the hydrolysis rate in water and the degradation rate in surface water (as measured using the OECD-309 guideline) because the underpinning of the Arrhenius activation energy of 75 kJ/mol for these systems is weak.

We recommend further studies on the variability of the *DegT50* (i) in aerobic fresh surface water in the dark, (ii) in the water layer of aerobic water-sediment systems in the dark, (iii) in outdoor surface water systems and (iv) sediments to underpin the CV of 0.5 to be used for such *DegT50* values in the assessments of the uncertainty of the PEC resulting from the uncertainty in these *DegT50* values.

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Appendix A Available data on pH, oxygen and suspended matter in surface water in the Netherlands

J. Klein (Deltares) & W.H.J. Beltman (Alterra)

A.1 Description of databases

In the Netherlands there are different databases with data of surface waters. When looking to ditches, however, not many data can be found because ditches are not regularly measured. Only in the National Program for Monitoring the Effectiveness of the Minerals Policy (LMM) ditches along fields are measured, together with drains and the upper groundwater. However, when looking to data of the pH, oxygen and suspended matter, only the pH is measured in these ditches and oxygen and suspended matter not. Besides this, this information is not totally free to use. This is, the data can be used, but the locations are secret.

The different water boards in the Netherlands usually measure the water quality in wider waters courses (A-water courses) than ditches.

The Ministry of Infrastructure & Environment, compartment Water Service, manages a big database, called the “bulkdatabse”, including the data of the water boards and the public waterways. In this database it should be possible to make a selection on ditches. However, this only results for four of the 26 water boards in the Netherlands in data of ditches. This is because it is uncertain if the labels connected to the database are filled in correctly by the water boards.

Table A-1 gives the statistics of the data between 2005 and 2009 of measurement locations with the label ‘ditch’.

Table A-1. Statistics of oxygen concentrations, pH and concentrations of suspended matter as reported for 16 measurement locations with label ‘ditch’ in the “bulkdatabse” between 2005 and 2009. The number of measurements is the total number of sampling times at these 16 locations.

	O₂ concentration (mg/L)	pH (-)	Suspended matter (mg/L)
Number of measurements	527	555	272
Median	8.2	7.4	5.0
Average	8.0	7.4	9.4
Minimum	0.0	6.3	1.0
Maximum	19.1	10.1	320.0

However, looking to the whole of the Netherlands is more representative than looking to some data of ditches, when it is not even sure that this is data of ditches and is only present in a small part of the Netherlands. Therefore the whole bulkdatabse is considered in the remainder of this document.

When looking to the whole database, only 2009 has been taken into account, because of the very high numbers of data. In 2009 at ca. 3500 measurement locations the pH and/or the oxygen concentration is measured. In total this results in 37304 measurements of oxygen and 42756 measurements of the pH in 2009. Suspended matter is only measured at ca 1520 locations, resulting in 11160 measurements in 2009.

To verify the data of the bulkdatabase, also data of the Limnodatabase Neerlandica (STOWA 2010; personal communication E.T.H.M. Peeters, WUR) is considered. This is a national database with data about the ecological quality of the surface water, measured by the regional water managers. In this database they also distinguish ditches. This results in 2436 ditches which are measured on the pH, resulting in 41624 measurements of the pH in the period between 1980 and 2005. Suspended matter is measured at 861 locations, with in total 12394 measurements in the period 1980-2005.

A.2 Oxygen concentrations from the bulkdatabase

In Table A-2 some statistics of the oxygen concentrations in surface water in the Netherlands are presented.

Table A-2. Statistics of oxygen concentrations in surface water in the Netherlands based on 37304 measurements on 3453 locations in 2009 taken from the bulkdatabase.

	O₂ (mg/L)
Median	8.4
Average	8.3
Minimum	0.0
Maximum	119.0

Figure A-1 represents the frequency distribution of the oxygen concentration with data of the bulkdatabase in 2009. The maximum of 119 mg/L in Table A-2 is considered not realistic because this is above the concentration of water saturated with oxygen but Figure A-1 shows that such high values were very exceptional.

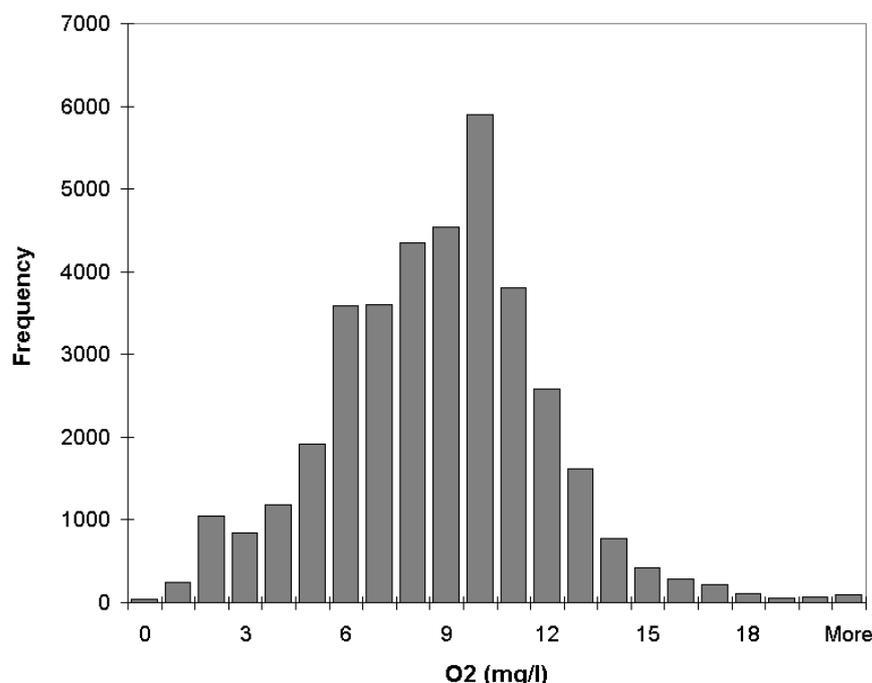


Figure A-1: Frequency distribution of the oxygen concentration (mg/L) in water courses in the Netherlands in 2009, based on data from the bulkdatabase. Same data as reported in Table A-2.

A.3 pH from bulkdatabase and Limnodatabase Neerlandica

In the Table A-3 some statistics of the pH in surface water in the Netherlands are presented.

Table A-3. Statistics of the pH in surface water in the Netherlands based on 42756 measurements on 3524 location in 2009 from the bulkdatabase.

	pH (-)
Median	7.7
Average	7.7
Minimum	2.4
Maximum	11.0

Figure A-2 shows the frequency distribution of the pH with data of the bulkdatabase in 2009.

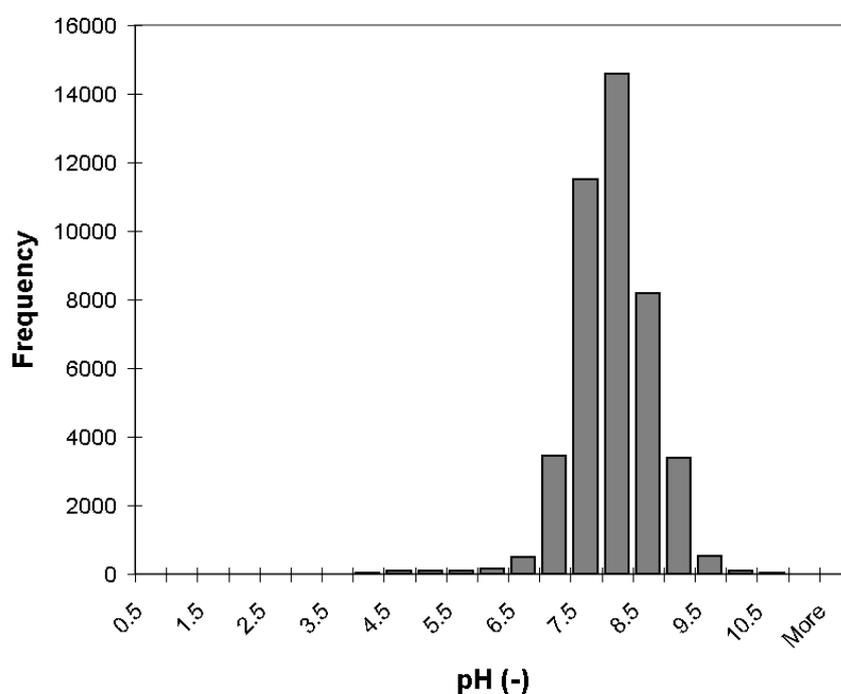


Figure A-2. Frequency distribution of the pH (-) in water courses in the Netherlands in 2009, based on data from the bulkdatabase (same data as reported in Table A-3).

To give some idea of the values, Table A-4 gives the frequency distribution.

Table A-4. Frequency distribution of the pH in surface water in the Netherlands based on 42756 measurements on 3524 location in 2009 from the bulkdatabase.

pH range	Frequency
0.5-1	0
1-1.5	0
1.5-2	0
2-2.5	0
2.5-3	1
3-3.5	0
3.5-4	10
4-4.5	24
4.5-5	82
5-5.5	107
5.5-6	81
6-6.5	160
6.5-7	499
7-7.5	3446
7.5-8	11501
8-8.5	14583
8.5-9	8199
9-9.5	3400
9.5-10	508
10-10.5	82
10.5-11	25
11-11.5	5

Figure A-3 is based on data from the Limnodatabase Neerlandica in the period between 1980 and 2005.

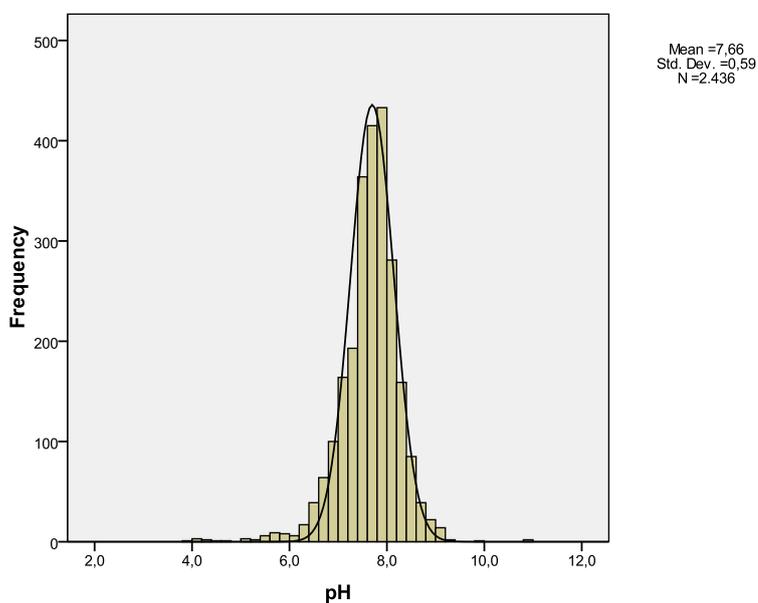


Figure A-3. Frequency distribution of the pH (-) in ditches in the Netherlands in the period 1980-2005, based on data from the Limnodatabase Neerlandica.

A.4 Suspended matter from bulkdatabase and Limnodatabase Neerlandica

Table A-5 gives some statistics of suspended matter concentrations in surface water in the Netherlands.

Table A-5. Statistics of concentrations of suspended matter based on 11160 measurements on 1523 locations in 2009 from the bulkdatabase.

Suspended matter (mg/L)	
Median	10.0
Average	17.5
Minimum	0.0
Maximum	1070.0

Figure A-4 shows the frequency distribution of the suspended matter concentrations with data of the bulkdatabase in 2009.

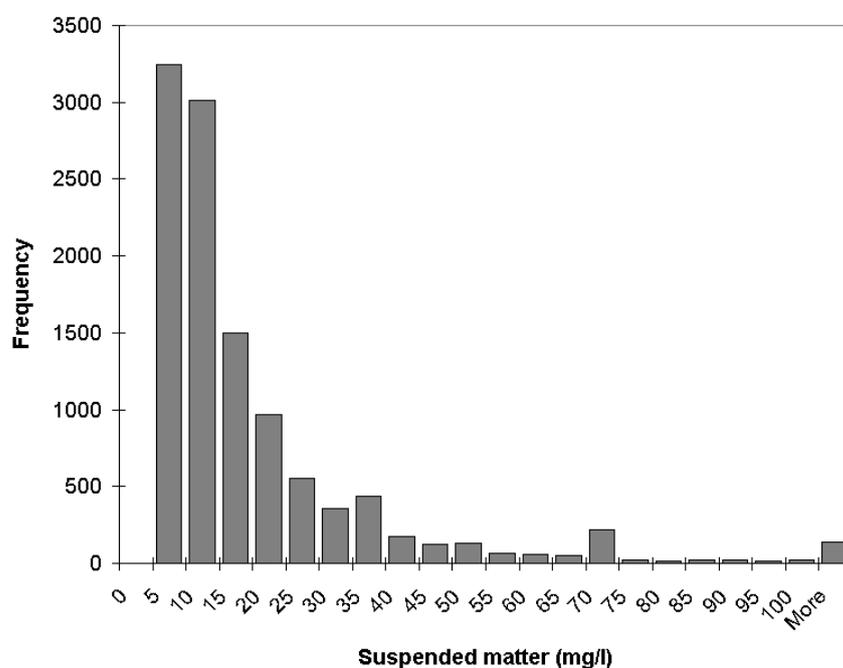


Figure A-4. Frequency distribution of the suspended matter concentration (mg/L) in water courses in the Netherlands in 2009, based on data from the bulkdatabase.

Figure A-5 is based on data from the Limnodatabase Neerlandica in the period between 1980 and 2005. The median of this database is 11 mg/L.

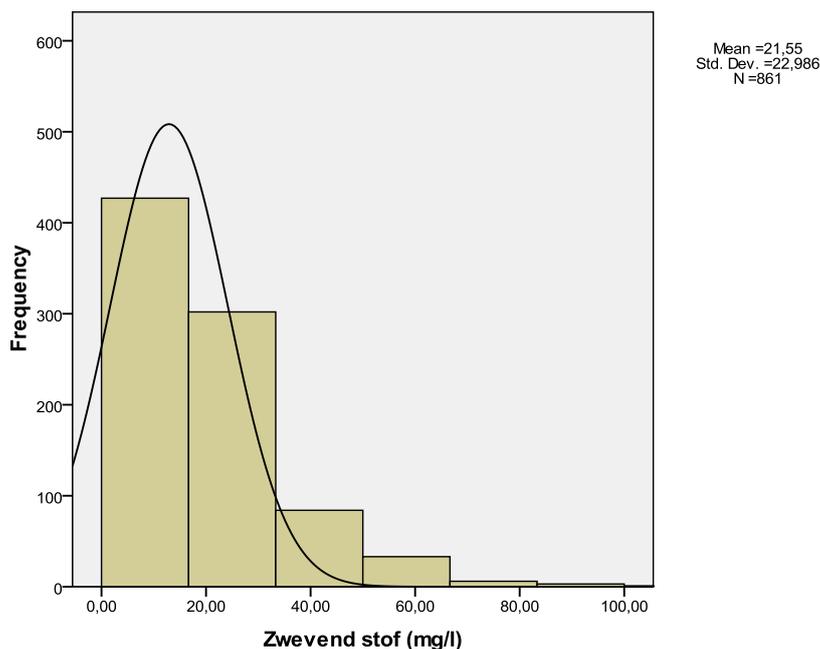


Figure A-5. Frequency distribution of the suspended matter concentration (mg/L) in ditches in the Netherlands in the period 1980-2005 from the Limnodatabase Neerlandica.

A.5 Conclusion

It can be concluded that the pH, the oxygen concentration and the suspended matter concentration are very variable in space and time. For example they are dependent on the amount of vegetation, the soil type and the time of the day. From the data presented above, it can be concluded that in more than 99% of the cases oxygen concentrations exceeded 0.5 mg/L indicating that the water in nearly all of the Dutch agricultural ditches is aerobic.

The most frequent pH is between 6.5 and 10 and the suspended matter concentration is most of the times between 0 and 40 mg/L.

A remark has to be placed by the presented data of the bulkdatabase when using this data as representative for ditches in the Netherlands. This is data of all available water courses in the Netherlands. In these data there will be ditches, but the presented data gives an overview of the statistics of the pH, the oxygen concentration and the suspended matter concentration in all water courses in the Netherlands.

Appendix B Abbreviations

CV	Coefficient of Variation
EFSA	European Food Safety Authority
OECD	Organisation for Economic Co-operation and Development
TOXSWA	TOXic substances in Surface WAter
TWA	Time-Weighted Average

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Natuur & Milieu

P.O. Box 47

NL-6700 AA Wageningen

T +31 (0) 317 48 54 71

E info.wnm@wur.nl

www.wageningenUR.nl/

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