*European Commission*

**Renewal Assessment Report of the Inclusion of the Active Substance in Annex I of the Regulation (EC) 1107/2009**

# Oxamyl

## Volume 3 (CA)
## ANNEX B.8
## Environmental fate and behaviour and environmental exposure assessment

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## *B.8 ENVIRONMENTAL FATE AND BEHAVIOUR*

This document provides an overview of the studies that were submitted and accepted for Annex I inclusion of Oxamyl under Council Directive 91/414/EEC.

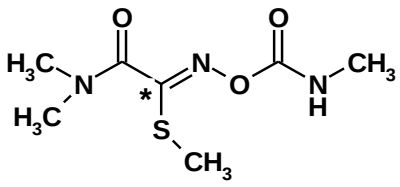
In addition, new unpublished data and published data are summarised and evaluated here. Data are presented to meet the data requirements of Regulation (EU) No 283/2013 under Regulation (EU) No 1107/2009.

Information is provided in the pages that follow with respect to the fate and behaviour in soil, water, and air of oxamyl, the active substance of a chemical nematicide/insecticide intended for use in potatoes. It is proposed that the plant protection product be applied once during a growing season (at the time of planting or drilling), at a maximum rate of 5.5 kg a.s./ha. The studies concerning the fate and behaviour of oxamyl in the environment were conducted using one labeled form of oxamyl. The central imine carbon ([1-$^{14}$C]) radiolabeled form of oxamyl was used to evaluate the fate of the active substance in the environment. The $^{14}$C-radiolabel was placed in the most stable position of oxamyl as indicated below in

**Figure 1. In certain studies the [1-$^{14}$C]oxamyl was amended with $^{13}$C-labeled oxamyl to create a distinct isotopic pattern that would aid in metabolite identification by mass spectrometry. This stable isotope was also labeled on the central imine carbon ([1-$^{13}$C]oxamyl) and was given the name IN-R4B16. The $^{13}$C test material is depicted in**

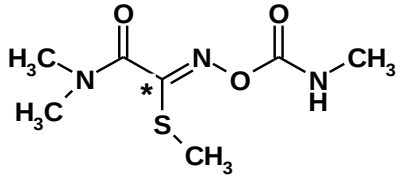
Figure 2.

**Figure 1 Positions of radiolabeling in [1-$^{14}$C]oxamyl**




* Denotes [1-$^{14}$C]oxamyl

**Figure 2 Positions of radiolabeling in [1-$^{13}$C]oxamyl (IN-R4B16)**




* Denotes [1-$^{13}$C]oxamyl

Unless specifically indicated, all reports in this section are submitted to address mandatory data requirements for the approval of active substance.

### B.8.1 Fate and behaviour in soil

#### B.8.1.1 Route of degradation in soil

##### B.8.1.1.1 Aerobic degradation

**B.8.1.1.1/01**

| | | |
|---|---|---|
| **Reference:** -- | **Report:** | Smyser, B.P., Mattson, S.L. (2000); Route of degradation of oxamyl in aerobic soil<br>**DuPont Report No.:** DuPont-2958<br>**Guidelines:** SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), U.S. EPA 162-1 (1982) |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 417
   Purity: Radiochemical purity - >95%

**Materials and Methods:**

The aerobic metabolism of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410, batch 417, specific activity 18.65 µCi/mg, radiochemical purity 97.4%) was studied in a field-fresh loam soil, from Nijmegen, The Netherlands. Soil samples were used within three months of collection from the field, and soil biomass determinations (made at the time of treatment and the end of the study) indicated the presence of a viable microbial population. Details of the soil characteristics are given in Table 1.

**Table 1 Characteristics of Nijmegen loam soil used to investigate the aerobic soil metabolism of oxamyl**

| Characteristic | Soil |
|---|---|
| Soil designation | Nijmegen |
| Origin | Nijmegen, The Netherlands |
| pH | 7.0 |
| % sand (2000-50 µm) | 45.2 |
| % silt (<50-2 µm) | 40.8 |
| % clay (<2 µm) | 14.0 |
| USDA texture | loam |
| Organic matter (%) | 2.4 |
| Organic carbon (%) | 1.4 |
| Cation exchange capacity (meq/100 g) | 10.1 |
| Moisture-holding capacity at 0 bar (%) | 33.3 |
| Initial microbial biomass (mg microbial C/100 g soil) | 18.71 |
| Final microbial biomass (mg microbial C/100 g soil) | 13.45 |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

Test soil was received fresh from an agricultural field and stored for less than 3 months at 5°C before use in the study and was sieved (2 mm) before use .

Prior to incubation under study conditions, the soil was adjusted to 40% of its 0-bar moisture content by the addition of purified water, resulting in a soil moisture content of 13.3% (g water/100 g dry soil).

Following a pre-incubation period of seven days, samples of the moist soil (equivalent to 50 g dry soil) were treated with ~100 µg [1-14C]-oxamyl. This treatment rate of 2 mg a.s./kg dry soil corresponds to an application rate of 3 kg a.s./ha (based on a soil incorporation depth of 10 cm and a default value of 1.5 g/cm3 for bulk dry soil density), which is 54.5% of the maximum recommended use rate for oxamyl (5.5 kg a.s./ha).

Sufficient samples were prepared so that replicates could be taken for analysis at each sampling timepoint. The treated soil samples were incubated for up to 59 days under aerobic conditions in the dark at 20 °C, with the moisture content being maintained at 40% of the 0- bar moisture-holding capacity throughout the incubation period (by the addition of purified water as necessary).

Humidified air was drawn over the soil samples by vacuum during the incubation period. In order to trap volatile products, evolved as a result of the application of the radiolabelled test substance, the effluent air from each treated sample was passed through an ethylene glycol trap and two caustic traps (both containing 1N $K_{OH}$ with phenolphthalein indicator). The purpose of the caustic traps was to collect acidic volatile components, such as carbon dioxide, while the ethylene glycol trap was in place to capture other volatile components.

Duplicate treated soil samples were removed for analysis at intervals of 0, 1, 2, 3, 7, 10, 14, 21, 31 and 59 days after test substance application. The volatile traps were collected and replenished at the same timepoints also. The collected soil samples were typically extracted three times with an acetonitrile/water mixture (50:50 v/v; 100 mL, 50 mL, 50 mL), followed by a final extraction with acetonitrile only (50 mL). The extracts were separated from the soil by centrifugation, decanted off and combined for each sample, in preparation for radioactivity quantification by liquid scintillation counting (LSC). Qualitative and quantitative determination of the distribution of [1- $^{14}$C]-oxamyl and its degradates in the soil extracts was undertaken using reversed phase high performance liquid chromatography (HPLC) with radiochemical detection, fraction collection and liquid scintillation analysis.

Radioactivity remaining in the soil samples after the extractions was determined by LSC, following combustion. In addition, the radioactivity remaining in the day-31 soil samples (both replicates) after the extractions described above was fractionated into that associated with the humic acid, fulvic acid and humin fractions of the organic phase of the soil by performing a further extraction with 0.5N NaOH and acidifying the extract to pH1 using concentrated HCl. The 0.5N NaOH extract (which contained the fulvic acid and humic acid extracts) was separated from the residue (humin) by centrifugation. The radioactivity associated with the humin fraction was determined by LSC, following air-drying and combustion of the residue. The humic acid fraction was precipitated by the acidification of the 0.5N NaOH extract and was separated from the fulvic acid supernatant by centrifugation. The humic acid precipitate was then redissolved with 0.5N NaOH, and LSC was used to quantify the radioactivity in the resulting solution and also that in the fulvic acid supernatant from the previous step.

Volatile radioactivity, collected in the trapping solutions, was quantified using LSC. The radioactivity in the 1N $K_{OH}$ traps was characterised as $^{14}CO_2$ by precipitation of the barium salt of [ $^{14}$C]-carbonate. Aliquots from the first 1N $K_{OH}$ trap from both day-31 replicates were mixed sequentially with BaCl2 and Na2CO3, forming a white precipitate, which was removed by centrifugation. The absence of $^{14}CO_2$ in the supernatants was confirmed using LSC.

**Findings:**

The limits of detection (LOD) for extract analysis, HPLC analysis and combustion analysis were respectively 0.20% applied radioactivity (0.004 mg oxamyl equivalents/kg soil), 0.39% applied radioactivity (0.0078 mg oxamyl equivalents/kg soil) and 0.03% applied radioactivity (0.0007 mg oxamyl equivalents/kg soil).

The levels (expressed as a percentage of applied radioactivity) of parent compound and degradates recovered at each sampling interval in the study, together with mass balances, are shown in Table 2.

Total recoveries of applied radioactivity (mass balance values) ranged from 100.3% (day 14) to 130.3% (day 59). In each case, the mass balance was calculated as the sum of volatile radioactivity in the $K_{OH}$ traps (14CO2), volatile radioactivity in the ethylene glycol trap, radioactivity extracted from the soil and unextracted radioactivity remaining in the soil.

There was very extensive mineralisation of the test substance during the study, as indicated by the increase in 14CO2 throughout the incubation period, which reached a level of 74.3% AR (applied radioactivity) by day 31 and a level of 108.5% AR by day 59. Concomitant with this was an increase in unextracted soil radioactivity, which reached a plateau towards the end of the study (19.7% AR on day 31, 19.8% AR on day 59). [An increase in unextractable radioactivity is expected when a microorganism utilises an exogenous compound for its growth and maintenance.] Volatile radioactivity other than 14CO2 was minimal, with a maximum level of 0.19% AR being found in the ethylene glycol trap by day 59.

**Table 2 Mass balance and distribution of applied radioactivity in aerobic Nijmegen loam soil after application of 2 mg/kg of [1-$^{14}$C]-oxamyl**

| DAA | Oxamyl | IN-A2213 | IN-D2708 | $^{14}CO_2$ | Volatiles | Others | NER | Total |
|---|---|---|---|---|---|---|---|---|
| 0 | 102.4 | 4.8 | - | - | - | 0.7 | 0.7 | 108.6 |
| 1 | 85.4 | 15.0 | 2.9 | 1.1 | 0.00 | 0.3 | 1.7 | 107.1 |
| 2 | 93.2 | 14.5 | 6.0 | 1.9 | 0.00 | 0.65 | 2.3 | 118.6 |
| 3 | 73.4 | 11.7 | 11.7 | 2.8 | 0.00 | 0.45 | 3.8 | 103.9 |
| 7 | 56.8 | 13.4 | 19.8 | 7.3 | 0.01 | 0.75 | 6.4 | 104.4 |
| 10 | 42.1 | 13.1 | 25.9 | 12.6 | 0.01 | 0.30 | 9.3 | 103.2 |
| 14 | 28.9 | 7.8 | 29.6 | 21.0 | 0.04 | 0.45 | 12.1 | 100.3 |
| 21 | 17.8 | 3.7 | 28.8 | 37.3 | 0.04 | - | 15.9 | 103.6 |
| 31 | 6.4 | 1.9 | 16.5 | 74.3 | 0.18 | - | 19.7 | 118.9 |
| 59 | <1.9 | <1.9 | <1.9 | 108.5 | 0.19 | - | 19.8 | 130.3 |

DAA = days after application, NER = non-extractable radioactivity

The figures for each timepoint are an average from two replicates. Day-59 extracts were not analysed by HPLC, since the extractable radioactivity at this timepoint accounted for only 1.9% of the applied radioactivity.

Soil-extractable radioactivity (comprising parent oxamyl, degradates IN-A2213 and IN-D2708 and other extractable radioactivity) declined from 107.9% AR on day 0 to 1.9% AR by day 59. Oxamyl itself declined rapidly from an initial level of 102.4% AR, accounting for <50% AR by day 10 and accounting for <1.9% AR by day 59. The incubation period for the study did not extend beyond 59 days because the degradation of oxamyl was essentially complete by this stage. The very good recovery of oxamyl on day 0 was considered to demonstrate test substance extractability and stability.

IN-A2213 (oxamyl oxime – the hydrolysis product of oxamyl) was the main soil degradation product initially. It reached a peak value of 15.0% AR on day 1 and then declined, accounting for 1.9% AR by day 31. IN-D2708, which was detected on day 1 at a level of 2.9% AR, subsequently became the main soil degradation product, reaching a maximum value of 29.6% AR on day 14. Levels of IN-2708 then decreased, such that it accounted for <1.9% AR by the end of the incubation period. Apart from $^{14}$CO2, IN-A2213, IN-D2708 and unextractable soil residues, no other degradation products were detected at levels exceeding 1% AR. The identities of IN-A2213 and IN-D2708 were confirmed by matching the retention time in samples to that of authentic standards using two separate HPLC analytical methods. Selected soil extracts were also directly fortified with IN-D2708 to confirm the retention time match.

The results of the fractionation of the unextracted soil radioactivity from the day-31 replicates (average of 19.7% AR) to determine the distribution of radioactivity in the fulvic acid, humic acid and humin fractions of the soil are given in Table 3.

**Table 3 Distribution of unextracted soil radioactivity in soil organic matter at day 31**

| Soil fraction | % of applied radioactivity | % of non-extractable fraction |
|---|---|---|
| Fulvic acid | 6.6 | 33.4 |
| Humic acid | 6.1 | 31.2 |
| Humin | 2.2 | 11.4 |

Average of unextracted soil radioactivity in the day-31 replicates was 19.7% AR. The fulvic acid, humic acid and humin fractions from the post-extracted day-31 soil samples respectively contained 6.6%, 6.1% and 2.2% of applied radioactivity, showing that the soil- bound (unextracted) portion of radioactivity in these samples was associated with all three fractions of the soil organic matter.

**Conclusions:**

Following application at a nominal rate of 2 mg a.s./kg dry soil, oxamyl was degraded extensively at 20 °C, under aerobic conditions in the dark, in the Dutch loam soil (Nijmegen) used in this laboratory study. Degradation was rapid with oxamyl accounting for <50% AR by day 10 and <1.9% AR by the end of the incubation period (day 59). The ultimate, and principal, degradation product was carbon dioxide, which increased throughout the study, to reach a level of 74.3% AR by day 31 and 108.5% AR by day 59. Unextracted soil radioactivity also increased during the incubation (reaching a plateau of approximately 20% AR towards the end of the study), and was shown to be primarily associated with the fulvic and humic acid fractions of the soil in approximately equal proportions and to a lesser extent with the humin fraction (based on fractionation of post-extracted day-31 soil samples).

The main soil degradation products were IN-A2213 and IN-D2708, which were detected at maximum respective levels of 15.0% AR (day 1) and 29.6% AR (day 14). Both these substances had declined to <1.9% AR by the end of the study. No other metabolite was observed at >1% AR. Total recovery is more than 110% twice out of 10. The minimal deviations outlined do not compromise the scientific validity of this study.

Overall, the study shows that oxamyl was readily utilised by soil microorganisms to form carbon dioxide, via the intermediate degradation products IN-A2213 and IN-D2708. On the basis of this study, the primary degradation pathway for oxamyl in aerobic soil appears to be hydrolysis to form IN-A2213, which subsequently degrades to IN-D2708, with carbon dioxide and unextractable soil residues being the final products.

The aerobic degradation study DuPont-2958, originally submitted under EU Rev8 Point IIA 7.1.1.1.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), and U.S. EPA 162-1 (1982). A review of this study indicates that it fully meets the current guideline (OECD 307) and is relied upon.

**RMS comments and conclusion :**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl

**B.8.1.1.1/02**

| **Reference:** -- | **Report:** | Mattson, S.L., Smyser, B.P. (2000); Rate of degradation of oxamyl in three aerobic soils<br>**DuPont Report No.:** DuPont-2957<br>**Guidelines:** SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), U.S. EPA 162-1 (1982) |
|---|---|---|

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 417
   Purity: Radiochemical purity - >97%

**Materials and Methods:**

The aerobic metabolism of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410, batch 417, specific activity 18.65 µCi/mg, radiochemical purity >97%) was studied in three field-moist soils at 20 °C, two of which were from the USA and one from Germany. The soils used were 'Commerce' (silt loam, USA), 'Drummer #6' (silty clay loam, USA) and 'Gross Umstadt' (silt loam, Germany). In addition, the aerobic soil metabolism of [1- $^{14}$C]-oxamyl at 10 °C was investigated using Commerce soil.

Test soils were received fresh from agricultural fields and stored for less than 3 months at 5°C before use in the study and were sieved (2 mm) before use .

Soil samples were used within three months of collection from the field, and soil biomass was measured at the beginning of the study and towards the end of the incubation periods. Details of the soil characteristics are given in Table 4.

Prior to incubation under study conditions, purified water was added to the Commerce and Drummer #6 soils, with Commerce being adjusted to 40% of its 0-bar moisture content (resulting in 13.3 g water/100 g dry soil) and Drummer #6 being adjusted to 47% of its 0-bar moisture content (resulting in 23.2 g water/100 g dry soil). Gross Umstadt soil was used as received, with a moisture content of 40% of its 0-bar moisture-holding capacity (corresponding to 20 g water/100 g dry soil).

Following a pre-incubation period of 7-9 days, samples of the moist soils (equivalent to 50 g dry soil) were treated with ~100 µg [1-$^{14}$C]-oxamyl. This treatment rate of 2 mg a.s./kg dry soil corresponds to an application rate of 2.46 kg a.s./ha for Commerce soil, 1.86 kg a.s./ha for Gross Umstadt soil and 2.34 kg a.s./ha for Drummer #6 soil (based on a soil incorporation depth of 10 cm and respective reported bulk soil densities of 1.23 g/cm3, 0.93 g/cm3 and 1.17 g/cm3). When expressed in percentage terms as a proportion of the maximum recommended use rate for oxamyl (5.5 kg a.s./ha), these theoretical application rates are respectively 45%, 34% and 43%.

Sufficient samples were prepared so that replicates could be taken for analysis at each sampling timepoint. Treated samples of all three soils were incubated at 20 °C under aerobic conditions in the dark (for up to 123 days). A separate incubation was carried out at 10 °C using Commerce soil, with treated samples being incubated under aerobic conditions in the dark for up to 179 days. The moisture contents of all the incubated soils were maintained at 40-50% of 0-bar moisture-holding capacity throughout the incubation periods (by the addition of purified water as necessary).

**Table 4 Characteristics of Commerce, Gross Umstadt and Drummer #6 soils used to investigate the aerobic soil metabolism of oxamyl**

| Parameter | Commerce | Gross Umstadt | Drummer #6 |
|---|---|---|---|
| Origin | Greenville, MS, USA | Gross Umstadt, Germany | Rochelle, IL, USA |
| USDA texture | silt loam | silt loam | silty clay loam |
| % sand | 32.8 | 5.6 | 8.4 |
| % silt | 56.4 | 77.2 | 60.8 |
| % clay | 10.8 | 17.2 | 30.8 |
| pH | 7.0 | 7.8 | 4.8 |
| Organic matter (%) | 0.4 | 2.1 | 4.4 |
| Organic carbon (%) | 0.2 | 1.2 | 2.6 |
| Cation exchange capacity (meq/100 g) | 6.7 | 9.6 | 26.3 |
| Bulk density (g/cm$^3$) | 1.23 | 0.93 | 1.17 |
| 0-bar moisture (%) | 33.3 | 50.0 | 49.4 |
| Initial biomass (mg microbial C/100 g soil) | 5.31 | 30.73 | 12.3 |
| Final biomass (mg microbial C/100 g soil) | 4.76 | 29.7 | 7.33 |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

Humidified air was drawn over the soil samples by vacuum during the incubations. In order to trap volatile products, evolved as a result of the application of the radiolabelled test substance, the effluent air from each treated sample was passed through an ethylene glycol trap and two caustic traps (both containing 1N $K_{OH}$ with phenolphthalein indicator). The purpose of the caustic traps was to collect acidic volatile components, such as carbon dioxide, while the ethylene glycol trap was in place to capture other volatile components.

Duplicate treated soil samples were removed for analysis at each sampling timepoint. The sampling intervals (days after test substance application) for the 20 °C incubations were 0, 1, 2, 3, 7, 11, 14, 21, 32 and 60 (Commerce soil), 0, 1, 2, 3, 7, 10, 14, 21 and 31 (Gross Umstadt soil), and 0, 1, 2, 3, 7, 11, 14, 21, 32, 60, 90 and 123 (Drummer #6). In the case of the 10 °C incubation (Commerce soil), the sampling intervals were 0, 3, 7, 14, 21, 32, 46, 60, 90, 123 and 179 days after test substance application. The volatile traps for each sample train were also collected and replenished at the same timepoints as the soil samples were removed for analysis.

The collected soil samples were typically extracted four times with an acetonitrile/water mixture (50:50 v/v, 100 mL per extraction), with the extracts being separated from the soil by centrifugation and then decanted off, in preparation for radioactivity quantification by LSC. Qualitative and quantitative determination of the distribution of [1- $^{14}$C]-oxamyl and its degradates in the soil extracts was undertaken using reversed phase HPLC with radiochemical detection, fraction collection and liquid scintillation analysis.

Radioactivity remaining in the soil samples after the extractions was determined by LSC, following combustion. Volatile radioactivity, collected in the trapping solutions, was quantified using LSC. The radioactivity in the 1N $K_{OH}$ traps was characterised as $^{14}$CO2 by precipitation of the barium salt of [ $^{14}$C]-carbonate. Aliquots from the first 1N $K_{OH}$ trap from both replicates at selected timepoints for each soil (Commerce: 90-day, Gross Umstadt: 31- day and Drummer #6: 123-day) were mixed sequentially with BaCl2 and Na2CO3, forming a white precipitate, which was removed by centrifugation. The absence of $^{14}$CO2 in the supernatants was confirmed using LSC.

**Findings:**

The limits of detection for extract analysis, HPLC analysis and combustion analysis were respectively 0.30% AR (0.006 mg oxamyl equivalents/kg soil), 0.36% AR (0.0072 mg oxamyl equivalents/kg soil) and 0.02% AR (0.0004 mg oxamyl equivalents/kg soil).

The levels (expressed as a percentage of applied radioactivity) of parent compound and degradates recovered at each sampling interval in the study, together with mass balances, are shown in Table 5.

Total recoveries of applied radioactivity (mass balance values) from the different incubations ranged from 99.0-103.6% (Commerce, 20 °C), 100.3-114.0% (Gross Umstadt, 20 °C), 101.3-109.9% (Drummer #6, 20 °C) and 96.3-121.9% (Commerce, 10 °C). In each case, the mass balance was calculated as the sum of volatile radioactivity in the $K_{OH}$ traps ( $^{14}$CO2), volatile radioactivity in the ethylene glycol trap, radioactivity extracted from the soil and unextracted radioactivity remaining in the soil.

Carbon dioxide was the predominant degradation product in all incubations, with levels increasing over time in each case. Mineralisation was more extensive in the Commerce and Gross Umstadt soils than in Drummer #6 soil and, as expected, was faster in Commerce soil incubated at 20 °C than in Commerce soil incubated at 10 °C. Maximum detected levels of $^{14}$CO2 at the end of the incubations were 73.1% AR (Commerce, 20 °C – day 60), 76.1% AR (Gross Umstadt, 20 °C – day 31), 25.6% AR (Drummer #6, 20 °C – day 123) and 81.0% AR (Commerce, 10 °C – day 179). Accompanying the increase in $^{14}$CO2 levels during the incubations was an increase in unextracted soil radioactivity, which reached maximum values of 26.4% AR (Commerce, 20 °C – day 32), 26.4% AR (Gross Umstadt, 20 °C – day 21), 20.7% AR (Drummer #6, 20 °C – day 123) and 17.7% AR (Commerce, 10 °C – day 179). Volatile radioactivity other than $^{14}$CO2 was negligible, with maximum detected levels of 0.05% AR (Commerce, 20 °C), 0.01% AR (Gross Umstadt, 20 °C), 0.01% AR (Drummer #6, 20 °C) and 0.08% AR (Commerce, 10 °C) being found in the ethylene glycol trap at the end of the incubations.

Soil-extractable radioactivity comprised parent oxamyl, degradates IN-A2213 and IN-D2708 and other extractable radioactivity for the Commerce and Gross-Umstadt soils, and comprised oxamyl, IN-A2213 and other extractable radioactivity for Drummer #6 soil. Levels of soil- extractable radioactivity decreased from initial values of around 100% AR to final values of 3.7% AR (Commerce, 20 °C – day 60), 4.5% AR (Gross Umstadt, 20 °C – day 31), 59.8% AR (Drummer #6, 20 °C – day 123) and 23.1% AR (Commerce, 10 °C – day 179).

Parent oxamyl declined rapidly in the Commerce and Gross Umstadt soils incubated at 20 °C, such that it was not detected by day 32 in Commerce soil and only accounted for 0.2% AR in Gross Umstadt soil by the end of the incubation period (31 days). In both these incubations, oxamyl accounted for <50% AR by day 7. As expected, degradation of oxamyl in 10 °C Commerce soil was slower than in 20 °C Commerce soil. However, degradation was still quite fast in Commerce soil at 10 °C, with oxamyl accounting for <50% AR by day 21.

Degradation of oxamyl in Drummer #6 soil (incubated at 20 °C) was considerably slower, with oxamyl accounting for 51.2% AR by the end of the incubation period (123 days). This is consistent with the lower extent of mineralisation observed in this soil (25.6% AR maximum). It appears that Drummer #6 soil may have had a less viable microbial population during its incubation than the Commerce and Gross Umstadt soils, since the initial and final microbial biomass measurements for Drummer #6 soil were both <1% of the total soil organic carbon content (accounting respectively for 0.48% and 0.29% of total organic carbon content).

**Table 5 Distribution of applied radioactivity for 20 °C incubations (Commerce, Gross Umstadt and Drummer #6 soils) and 10 °C incubation (Commerce soil)**

| DAA | Oxamyl | IN-A2213 | IN-D2708 | $^{14}CO_2$ | Volatiles | Others | NER | Total |
|---|---|---|---|---|---|---|---|---|
| **Commerce – 20 °C** | | | | | | | | |
| 0 | 97.3 | 1.1 | - | - | - | 0.9 | 2.5 | 101.9 |
| 1 | 79.1 | 17.2 | 0.9 | 0.6 | 0.00 | 0.6 | 1.5 | 100.0 |
| 2 | 68.5 | 27.8 | 2.9 | 1.2 | 0.00 | 1.1 | 1.7 | 103.2 |
| 3 | 53.2 | 38.5 | 5.9 | 1.5 | 0.01 | 1.6 | 2.9 | 103.6 |
| 7 | 10.8 | 51.0 | 19.6 | 6.9 | 0.01 | 3.5 | 7.4 | 99.0 |
| 11 | 5.0 | 39.3 | 25.7 | 15.1 | 0.02 | 3.8 | 10.6 | 99.5 |
| 14 | 3.0 | 30.4 | 25.3 | 24.1 | 0.04 | 2.7 | 14.8 | 100.4 |
| 21 | 0.8 | 16.8 | 12.4 | 47.5 | 0.05 | 1.4 | 23.1 | 102.0 |
| 32 | nd | 3.4 | nd | 65.2 | 0.05 | 1.5 | 26.4 | 101.5 |
| 60 | nd | nd | nd | 73.1 | 0.05 | 0.4 | 24.1 | 100.9 |
| **Gross Umstadt – 20 °C** | | | | | | | | |
| 0 | 91.3 | 13.8 | - | - | - | 0.7 | 1.0 | 107.9 |
| 1 | 78.5 | 16.6 | 3.2 | 1.0 | 0.00 | 1.0 | 2.2 | 102.5 |
| 2 | 73.7 | 24.9 | 8.7 | 2.1 | 0.00 | 0.9 | 3.6 | 114.0 |
| 3 | 60.6 | 20.9 | 13.0 | 3.3 | 0.00 | 0.7 | 5.2 | 103.9 |
| 7 | 32.2 | 18.3 | 31.0 | 10.1 | 0.01 | 0.6 | 10.1 | 102.3 |
| 10 | 17.5 | 12.5 | 34.7 | 20.8 | 0.01 | 0.3 | 16.2 | 102.3 |
| 14 | 11.6 | 5.1 | 20.6 | 40.1 | 0.01 | 0.3 | 22.4 | 100.3 |
| 21 | 2.4 | 1.1 | 2.5 | 63.7 | 0.01 | nd | 26.4 | 100.9 |
| 31 | 0.2 | 0.2 | 0.3 | 76.1 | 0.01 | nd | 23.7 | 104.3 |
| **Drummer #6 – 20 °C** | | | | | | | | |
| 0 | 97.2 | - | - | - | - | 0.8 | 3.3 | 101.3 |
| 1 | 100.0 | 0.8 | - | 0.3 | 0.00 | 0.9 | 2.3 | 104.2 |
| 2 | 98.0 | 0.3 | - | 0.4 | 0.00 | 1.0 | 2.6 | 103.0 |
| 3 | 103.8 | 1.7 | - | 0.6 | 0.00 | 1.0 | 2.7 | 109.9 |
| 7 | 96.5 | 3.2 | - | 1.2 | 0.00 | 1.0 | 4.6 | 106.5 |
| 11 | 93.2 | 3.8 | - | 2.1 | 0.00 | 1.3 | 4.9 | 105.3 |
| 14 | 91.9 | 4.1 | - | 2.9 | 0.00 | 1.0 | 6.6 | 106.5 |
| 21 | 84.8 | 5.5 | - | 4.4 | 0.00 | 1.0 | 7.4 | 103.1 |
| 32 | 79.3 | 6.7 | - | 6.9 | 0.01 | 1.3 | 9.8 | 104.0 |
| 60 | 66.7 | 7.6 | - | 12.4 | 0.01 | 1.9 | 13.9 | 102.5 |
| 90 | 55.6 | 7.0 | - | 17.9 | 0.01 | 2.4 | 19.3 | 102.2 |
| 123 | 51.2 | 6.0 | - | 25.6 | 0.01 | 2.7 | 20.7 | 106.2 |
| **Commerce – 10 °C** | | | | | | | | |
| 0 | 95.1 | 1.8 | - | - | - | 1.0 | 2.7 | 100.6 |
| 3 | 94.6 | 6.9 | 0.4 | 0.6 | 0.00 | 0.8 | 1.3 | 104.5 |
| 7 | 80.6 | 15.8 | 2.6 | 1.4 | 0.00 | 0.8 | 2.4 | 103.6 |
| 14 | 55.6 | 28.1 | 7.8 | 3.7 | 0.00 | 2.0 | 4.4 | 101.5 |
| 21 | 39.1 | 33.6 | 14.1 | 6.0 | 0.01 | 2.0 | 4.9 | 99.6 |
| 32 | 26.8 | 33.8 | 20.7 | 11.1 | 0.01 | 2.7 | 6.6 | 101.0 |
| 46 | 13.8 | 30.9 | 27.4 | 12.2 | 0.01 | 2.1 | 10.6 | 97.0 |
| 60 | 5.7 | 19.5 | 37.6 | 19.3 | 0.01 | 1.6 | 12.6 | 96.3 |
| 90 | 0.4 | 9.5 | 39.5 | 35.2 | 0.02 | 1.1 | 15.3 | 101.0 |
| 123 | 0.3 | 4.1 | 33.5 | 52.1 | 0.04 | 0.5 | 15.4 | 109.6 |
| 179 | nd | 0.5 | 20.1 | 81.0 | 0.08 | 0.5 | 17.7 | 121.9 |

DAA = days after application, NER = non-extractable radioactivity, nd = not detected (limit of detection was 0.36% AR)
The figures for each timepoint are an average from two replicates.

OECD Test Guideline 307 ('Aerobic and Anaerobic Transformation in Soil' – adopted 24 April 2002) recommends that soils selected for study should have a microbial biomass of at least 1% of total organic carbon. [This soil quality criterion was agreed at an OECD workshop on the selection of soils and sediments, held in Belgirate, Italy in 1995.1] The strongly acidic nature of Drummer #6 soil (pH of 4.8) may have resulted in a stressed microbial population and may also have inhibited degradation of oxamyl by hydrolysis to IN- A2213, which is a base-catalysed reaction.

IN-A2213 was the initial soil degradation product of oxamyl in all cases. The time at which maximum levels of IN-A2213 were detected varied according to the rate of oxamyl degradation, with the time of maximum detect being nearer to the beginning of the incubation period for the faster oxamyl-degrading incubations (Commerce, 20 °C and Gross Umstadt, 20°C). Maximum detected levels of IN-A2213 were 51.0% AR (Commerce, 20 °C – day 7), 24.9% AR (Gross Umstadt, 20 °C – day 2), 7.6% AR (Drummer #6, 20 °C – day 60) and 33.8% AR (Commerce, 10 °C – day 32). By the end of the incubations, IN-A2213 had declined to final detected levels of 3.4% AR (Commerce, 20 °C – day 32), 0.2% AR (Gross Umstadt, 20 °C – day 31), 6.0% AR (Drummer #6, 20 °C – day 123) and 0.5% AR (Commerce, 10 °C – day 179). The lower levels of IN-A2213 observed in Drummer #6 soil (all <10% AR) may be due to a combination of slow formation (via oxamyl degradation) and relatively fast degradation of IN-A2213 to subsequent products in this case (including carbon dioxide and unextracted radioactivity).

In the Commerce and Gross Umstadt soils, IN-D2708 became a major soil degradation product as IN-A2213 declined, reaching maximum levels of 25.7% AR (Commerce, 20 °C – day 11), 34.7% AR (Gross Umstadt, 20 °C – day 10) and 39.5% AR (Commerce, 10 °C – day 90). Levels of IN-D2708 then decreased, such that by the end of the incubation periods it accounted for less than detectable levels in 20 °C Commerce soil, 0.3% AR (Gross Umstadt, 20 °C) and 20.1% AR (Commerce, 10 °C). The identities of IN-A2213 and IN-D2708 were confirmed by matching the retention time in samples to that of authentic standards using two separate HPLC analytical methods.

IN-D2708 was not seen in Drummer #6 soil. The slow rate of oxamyl degradation in this soil produced low levels of IN-A2213, which appears to be the precursor for IN-D2708 formation. Apart from $^{14}CO2$, IN-A2213 and IN-D2708, no other significant degradation products were identified. Other soil-extractable radioactivity accounted for maximum values of 3.8% AR (Commerce, 20 °C – day 11), 1.0% AR (Gross Umstadt, 20 °C – day 1), 2.7% AR (Drummer#6, 20 °C – day 123) and 2.7% AR (Commerce, 10 °C – day 32).

**Conclusions:**

Following application at a nominal rate of 2 mg a.s./kg dry soil, oxamyl was degraded extensively, under aerobic conditions in the dark, in Commerce (silt loam, USA) and Gross Umstadt (silt loam, Germany) soils incubated at 20 °C and in Commerce soil incubated at 10°C. Degradation was rapid in these incubations, with oxamyl accounting for <50% AR by day 7 in both 20 °C Commerce soil and Gross Umstadt soil and by day 21 in 10 °C Commerce soil. By the end of these incubations, oxamyl accounted for less than detectable levels in 20°C Commerce soil (day 32 onwards), 0.2% AR in Gross Umstadt soil (day 31) and less than detectable levels in 10 °C Commerce soil (day 179).

Degradation of oxamyl was less extensive in Drummer #6 soil (silty clay loam, USA), incubated at 20 °C, with oxamyl accounting for 51.2% AR by the end of this incubation (day 123). Mineralisation in Drummer #6 soil was also substantially lower than in the other incubations but was still significant (25.6% AR maximum). It appears that Drummer #6 soil may have had a stressed microbial population due to its strongly acidic nature (pH of 4.8) and that, therefore, it may not have been representative of a typical European agricultural soil. The acidic nature of Drummer #6 soil may also have inhibited degradation of oxamyl by hydrolysis to IN-A2213, which occurs faster under alkaline conditions.

The predominant degradation product in all cases was carbon dioxide, which increased throughout each incubation, reaching final levels of 73.1% AR (Commerce, 20 °C – day 60), 76.1% AR (Gross Umstadt, 20 °C – day 31), 25.6% AR (Drummer #6, 20 °C – day 123) and 81.0% AR (Commerce, 10 °C – day 179). Unextracted soil radioactivity also increased during the incubations, reaching maximum values of 26.4% AR (Commerce, 20 °C – day 32), 26.4% AR (Gross Umstadt, 20 °C – day 21), 20.7% AR (Drummer #6, 20 °C – day 123) and 17.7% AR (Commerce, 10 °C – day 179). Total recovery is more than 110% one out of 9 for Gross Umstadt soil, and one out of 11 for Commerce soil. The deviations outlined are minimal and do not compromise the scientific validity of this study.

The main degradation products extracted from soil were IN-A2213 and IN-D2708. IN-A2213 was detected in all soils, while IN-D2708 was observed in the Commerce and Gross Umstadt soils only. No other significant degradation products were identified apart from $^{14}CO2$, IN- A2213 and IN-D2708.

Maximum detected levels of IN-A2213 were 51.0% AR (Commerce, 20 °C – day 7), 24.9% AR (Gross Umstadt, 20 °C – day 2), 7.6% AR (Drummer #6, 20 °C – day 60) and 33.8% AR (Commerce, 10 °C – day 32). By the end of the incubations, IN-A2213 had declined to final detected levels of 3.4% AR (Commerce, 20 °C – day 32), 0.2% AR (Gross Umstadt, 20 °C – day 31), 6.0% AR (Drummer #6, 20 °C – day 123) and 0.5% AR (Commerce, 10 °C – day 179).

IN-D2708 became a major soil degradation product in the Commerce and Gross Umstadt soils as IN-A2213 declined, reaching maximum levels of 25.7% AR (Commerce, 20 °C – day 11), 34.7% AR (Gross Umstadt, 20 °C – day 10) and 39.5% AR (Commerce, 10 °C – day 90). By the end of these incubations, IN-D2708 had declined to less than detectable levels in 20 °C Commerce soil, 0.3% AR (Gross Umstadt, 20 °C) and 20.1% AR (Commerce, 10 °C). It was surmised that IN-D2708 was not seen in Drummer #6 soil due to the slow rate of oxamyl degradation in this soil, which resulted in lower levels of IN-A2213 (the precursor for IN- D2708 formation).

With regard to the effect of temperature on the incubations, although degradation of oxamyl in 10 °C Commerce soil was slower than in 20 °C Commerce soil (as expected), the route of degradation was the same in both cases. Overall, the study confirms the findings from Study 1 in this section (report no. DuPont-2958), indicating that the major degradation pathway for oxamyl during aerobic incubation in microbially viable agricultural topsoil is hydrolysis to form IN-A2213, which subsequently degrades to IN-D2708, with carbon dioxide and unextractable soil residues being the final products.

The aerobic degradation study DuPont-2957, originally submitted under EU Rev8 Point IIA 7.1.1.1.1 and conducted with test material [1- $^{14}$C]oxamyl, was conducted under guidelines SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), and U.S. EPA 162-1 (1982). A review of this study indicates that it partially meets the current guideline (OECD 307); deviations include the Drummer#6 soil having soil properties outside of those recommended in the guideline. In particular, the Drummer#6 soil had a pH of 4.8, which is outside the guideline recommended range of 5.5–8.0. Microbial measurements of this soil at the start and termination of the incubation demonstrated that the soil did not meet the additional criteria of a microbial biomass of at least 1% of the total organic carbon. It is believed that the extremely low pH of this Drummer#6 soil hindered its microbial health and rendered it a non-viable soil. However, reconduct is unlikely to yield a significantly different result. The two remaining soils used in this study provide an adequate and consistent picture of the route of oxamyl degradation in aerobic soils when considered with the additional route studies, DuPont-2958 and DuPont-39014. Therefore, this study is relied upon

**RMS comments and conclusion :**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl

**B.8.1.1.1/03**

| **Reference:** | **Report:** | Spare, W.C. (1991); Anaerobic soil metabolism of [1-$^{14}$C]oxamyl in Madera, California soil<br><br>**DuPont Report No.:** AMR 1851-90<br><br>**Guidelines:** U.S. EPA 162-2 (1982) |
|---|---|---|
| -- | | |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 311
   Purity: Radiochemical purity - >99%

**Materials and Methods:**

The metabolism and degradation of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410, batch 311, specific activity 78.2 µCi/mg, radiochemical purity >99%) were studied in a US sandy clay loam soil (from Madera, California) under aerobic conditions followed by anaerobic conditions. Only the initial aerobic portion of the study is described in this section. Details of the soil characteristics are given in Table 6. Soil microbial biomass measurements were not reported.

**Table 6 Characteristics of Madera sandy clay loam soil used to investigate the aerobic and anaerobic soil metabolism of oxamyl**

| Characteristic | Soil |
|---|---|
| Soil designation | Madera |
| Origin | Madera, California |
| % clay | 22.4 |
| % silt | 24.0 |
| % sand | 53.6 |
| USDA texture | sandy clay loam |
| pH | 7.7 |
| Organic matter (%) | 1.5 |
| Organic carbon (%) | 0.87 |
| Cation exchange capacity (meq/100 g) | 20.81 |
| Bulk density (g/cm$^3$) | 1.25 |
| Moisture-holding capacity at ⅓ bar (%) | 15.39 |
| Moisture-holding capacity at 15 bar (%) | 13.56 |

Prior to incubation under study conditions, the moisture content of the soil was adjusted to 50% of field capacity by the addition of purified water, resulting in a soil moisture content of 7.7 g water/100 g dry soil (assuming field capacity to be the moisture-holding capacity at ⅓ bar). During the aerobic incubation phase of the study, the soil moisture content was adjusted to, and maintained at, 70-75% of field capacity (by the addition of purified water as necessary).

Following a pre-incubation period of approximately one week, samples of the moist soil (equivalent to 50 g dry soil) were treated with ~475 µg [1- $^{14}$C]-oxamyl. This treatment rate of 9.5 mg a.s./kg dry soil corresponds to an application rate of 11.875 kg a.s./ha (based on a soil incorporation depth of 10 cm and the reported bulk soil density of 1.25 g/cm3), which is 216% of the maximum recommended use rate for oxamyl (5.5 kg a.s./ha).

The treated soil samples were incubated for up to 51 days under aerobic conditions in the dark at 25 ±1 °C. Humidified air was passed through the flasks containing the samples for one hour each working day, at a flow-rate of 20 mL/minute. In order to trap volatile products, evolved as a result of the application of the radiolabelled test substance, effluent air from each treated sample passed through a polyurethane foam plug and two caustic traps (both containing 2N $K_{OH}$). The purpose of the caustic traps was to collect acidic volatile components, such as carbon dioxide, while the polyurethane foam plug was in place to capture other volatile components.

Soil samples were removed for analysis at intervals of 0, 1, 3, 7, 10, 11, 21, 31 and 51 days after test substance application. The traps for volatiles were replaced at the same timepoints also. All collected soil samples were extracted three times with a methanol/water mixture (50:50 v/v, 100 mL per extraction) and once with methanol (100 mL), with sonication being used to aid the extraction process. The extracts were separated from the soil by centrifugation, decanted off and combined for each sample, in preparation for radioactivity quantification by LSC. Qualitative and quantitative determination of the distribution of [1- $^{14}$C]-oxamyl and its degradates in the soil extracts was undertaken using thin layer chromatography (TLC). In addition, HPLC was used as a confirmatory chromatographic method for selected representative soil extracts.

Radioactivity remaining in the soil samples after the extractions was determined by LSC, following combustion of air-dried samples. Volatile radioactivity, collected in the caustic trapping solutions and polyurethane foam plugs, was quantified using LSC; with the foam plugs being extracted by sonication with a methanol/water mixture (50:50 v/v, 10 mL) prior to analysis. The radioactivity in the 2N $K_{OH}$ traps was characterised as $^{14}CO2$ by precipitation of the barium salt of [ $^{14}C$]-carbonate. Aliquots from the 2N $K_{OH}$ traps were mixed with BaCl2, forming a white precipitate, which was removed by centrifugation. The absence of $^{14}CO2$ in the supernatants was confirmed using LSC.

**Findings:**

The levels (expressed as a percentage of applied radioactivity) of parent compound and degradates recovered at each sampling interval in the aerobic phase of the study, together with mass balances, are shown in Table 7.

**Table 7 Mass balance and distribution of applied radioactivity in aerobic Madera sandy clay loam soil after application of 9.5 mg/kg of [1-$^{14}C$]-oxamyl**

| Sampling time | % of applied radioactivity | | | | | | |
|---|---|---|---|---|---|---|---|
| | Soil extraction | | | $^{14}CO_2$ | Other volatiles | NER | Total |
| | Oxamyl | IN-A2213 | IN-D2708 | | | | |
| Day 0 | 101.1 | 1.7 | <0.1 | <0.1 | nd | 0.4 | 103.2 |
| Day 1 | 90.1 | 6.7 | <0.1 | <0.1 | nd | 1.0 | 97.8 |
| Day 3 | 74.3 | 13.0 | 4.4 | 0.1 | nd | 1.9 | 93.7 |
| Day 7 | 67.8 | 19.6 | 8.7 | 0.6 | nd | 2.9 | 99.6 |
| Day 10 | 51.0 | 24.3 | 15.0 | 0.9 | nd | 4.3 | 95.5 |
| Day 11 | 53.1 | 23.9 | 18.1 | 1.1 | nd | 5.3 | 101.5 |
| Day 21 | 27.6 | 22.3 | 20.3 | 8.3 | nd | 11.4 | 89.9 |
| Day 31 | 12.7 | 21.6 | 9.0 | 25.6 | nd | 18.9 | 87.8 |
| Day 51 | 3.7 | 8.6 | 5.0 | 45.3 | nd | 24.2 | 86.8 |

NER = non-extractable radioactivity, nd = not detected (<0.1% AR) Sampling time refers to days after treatment. The data presented are averages of two replicate samples for each timepoint, except day 3 and day 11, which were from a single sample.

Total recoveries of applied radioactivity (mass balance values) ranged from 86.8% (day 51) to 103.2% (day 0). In each case, the mass balance was calculated as the sum of volatile radioactivity in the KOH traps ( $^{14}CO2$), volatile radioactivity in the polyurethane foam plugs, radioactivity extracted from the soil and unextracted radioactivity remaining in the soil.

Extensive mineralisation of the test substance occurred under aerobic conditions, with $^{14}CO2$ increasing throughout the incubation period to become the predominant degradation product, such that it accounted for 45.3% AR by day 51. Unextracted soil radioactivity also increased throughout the incubation period, in parallel with carbon dioxide evolution, reaching a level of 24.2% AR by day 51. Volatile radioactivity other than $^{14}CO2$ was negligible, with the radioactivity extracted from the polyurethane foam plugs accounting for <0.1% AR in all cases.

Soil-extractable radioactivity (comprising parent oxamyl and degradates IN-A2213 and IN- D2708) declined from 102.8% AR on day 0 to 17.3% AR by day 51. Oxamyl declined rapidly, accounting for 53.1% AR by day 11 and 3.7% AR by the end of the aerobic incubation (day 51).

The hydrolysis product IN-A2213 was the initial soil degradation product. It reached a peak value of 24.3% AR on day 10 and then declined, accounting for 8.6% AR by day 51. IN- D2708 formed subsequently, being detected at a level of 4.4% AR on day 3, reaching a maximum value of 20.3% AR on day 21 and accounting for 5.0% AR by day 51. The identities of IN-A2213 and IN-D2708 were confirmed by matching their retention times in samples to those of authentic standards using TLC and HPLC analytical methods. Apart from $^{14}CO2$, IN-A2213, IN-D2708 and unextractable soil residues, no other degradation products were detected in excess of 0.1% AR.

**Conclusions:**

Following application at a nominal rate of 9.5 mg a.s./kg dry soil, oxamyl was degraded extensively at 25 ±1 °C, under aerobic conditions in the dark, in the US sandy clay loam soil (Madera, California) used in this laboratory study. Degradation was rapid with oxamyl accounting for 53.1% AR by day 11 and 3.7% AR by the end of the aerobic incubation (day 51). Carbon dioxide increased throughout the incubation period to become the predominant degradation product, accounting for 45.3% AR by day 51. Evolution of carbon dioxide was paralleled by an increase in unextracted soil radioactivity, which reached a level of 24.2% AR by day 51.

The degradation products IN-A2213 and IN-D2708 were extracted from soil, being detected at maximum respective levels of 24.3% AR (day 10) and 20.3% AR (day 21). These substances had declined to levels of 8.6% AR and 5.0% AR respectively by day 51. No other metabolites in excess of 0.1% AR were detected.

The study shows that oxamyl degraded readily in aerobic topsoil to form IN-A2213, IN- D2708 and unextractable soil residues, with carbon dioxide as the resulting major degradation product. This is in agreement with the reported findings from Study 1 and Study 2 in this section (report nos. DuPont-2958 and DuPont-2957 respectively), confirming that the major degradation pathway for oxamyl in aerobic soil is hydrolysis to form IN-A2213, which subsequently degrades to IN-D2708, with carbon dioxide and unextractable soil residues being the final products.

The aerobic part of degradation study AMR 1851-90, originally submitted under EU Rev8 Point IIA 7.1.1.1.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline U.S. EPA 162-2 (1982). A review of this study indicates that the aerobic soil experiment performed in this study meets the current guideline (OECD 307). The only minor deviation is a mass balance slightly below the guideline recommended 90-100%AR at the last 2 sampling points. This deviation does not impact the outcome of the study since the degradation pathway of oxamyl was well established before the final timepoints. Losses are attributed to slight ineffieciencies in trapping the large amounts of $^{14}C0_2$ generated during the extensive mineralization of oxamyl. Therefore reconduct is unlikely to yield a significantly different results, and, this study is relied upon.

**RMS comments and conclusion :**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl

**B.8.1.1.1/04**

| | | |
|---|---|---|
| **Reference:** **CA 7.1.1.1/01** | **Report:** | Clark, B. (2015); Aerobic rate of degradation of [$^{14}$C]-DPX-D1410 (oxamyl) in four acidic soils<br>**DuPont Report No.:** DuPont-39014<br>**Guidelines:** OECD 307, OPPTS 835.4100, SETAC (1995)<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** 80581<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

The rate of degradation of oxamyl was examined in four acidic soils in the laboratory under aerobic conditions. The test soils were treated with radiolabeled oxamyl at a concentration of 5.0 μg a.s./g soil. Samples were

incubated in darkness at 20 ± 2°C with a soil moisture of 100% of maximum water holding capacity (0.1 bar moisture, pF2).

Oxamyl degraded at 20°C in all test soils with $DT_{50}$ values ranging from 0.6 to 9.7 days and $DT_{90}$ values ranging from 2.0 to 79.5 days.

The major transformation products detected were IN-A2213 and IN-D2708, accounting for a maximum of 13.5 and 78.0 AR%, respectively, after 91 days of incubation.

Material balance ranged from 93.3 to 96.4% of the applied radioactivity for $^{14}$C-oxamyl. Non-extractable $^{14}$C-residues increased from 1% of the applied amount at Day 0 to 22.0% of the applied at the end of the study. At study termination, evolved $^{14}CO_2$ was 63.1% of the applied amount.

The relative rate of degradation of oxamyl by the four soils was related to the microbial activity, pH, and organic matter content of the soils.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabeled test material: $^{14}$C-oxamyl
   Lot/Batch #: 1841000
   Radiochemical purity: 99.7%
   Specific activity: 75.6 µCi/mg
   Description: Solid
   Stability of test compound: Not determined
2. Stable labeled test material: $^{13}$C-oxamyl
   Lot/Batch #: MBBB0508V
   Chemical purity: 98%
   Description: Solid
   Stability of test compound: Not determined

4. Soils

Four soils were chosen to represent conditions of use; Speyer loamy sand (Hanhofen, Germany), Tama light clay (Stark County, Illinois), LRA-D sandy loam (Derbyshire, England), and Goch sandy loam (Northrhine-Westphalia, Germany). Prior to use, each test soil was homogenized and passed through a 2-mm mesh sieve. Soils were then stored at approximately 4°C in the dark in closed bags when not in use. Pre-incubation was performed to acclimate soil samples to the test temperature and achieve aerobic conditions prior to study initiation. Samples were pre-incubated for 9 days for the Definitive Test sample series before the test substance was applied.

**Table 8 Characteristics of test soils**

| Characteristic | Soil | | | |
|---|---|---|---|---|
| **Soil name or designation** | **Speyer** | **Tama** | **LRA-D** | **Goch** |
| Origin location | Hanhofen, Germany | Stark County, Illinois | Derbyshire, England | Northrhine-Westphalia, Germany |
| pH (1:2 soil:0.01M $CaCl_2$) | 6.1 | 6.7 | 5.4 | 5.7 |
| % Sand (2000-50 µm) | 87 | 31 | 79 | 70 |
| % Silt (<50-2 µm) | 8 | 36 | 14 | 21 |
| % Clay (<2 µm) | 5 | 33 | 7 | 9 |
| Texture[a] | Loamy Sand | Light Clay | Sandy Loam | Sandy Loam |
| % Organic matter | 2.7 | 5.4 | 5.8 | 3.5 |
| Cation exchange capacity | 6.8 | 20.9 | 10.5 | 8.8 |
| Maximum water-holding capacity (%) | 45.4 | 86.5 | 57.1 | 54.2 |
| Microbial biomass (µg C $g^{-1}$ soil) | | | | |
| Initial: | 425.4 | 723.1 | 613.9 | 553.3 |
| Final: | 328.4 | 448.8 | 426.7 | 264.6 |

[a] International soil classification system

B. STUDY DESIGN

1. Experimental conditions

Portions of sieved soils (50 g dry-soil equivalent) were adjusted to 100% of their respective 1/10 bar water holding capacities (pF2). Solutions of the radiolabeled test substance in acetonitrile were prepared and applied to soil samples in separate 250-mL Nalgene bottles. The organic solvent was allowed to evaporate, after which the soil was mixed to ensure homogeneity. Water lost to evaporation was replaced, and soils were incubated in the dark at 20 ± 2°C under aerobic conditions for up to 91 days in closed systems to trap evolved carbon dioxide and volatile organic compounds.

Soil samples were taken and analysed for oxamyl and degradates. Additional samples were prepared and incubated for determination of biomass.

2. Sampling

Microbial biomass was determined at zero time and Day 91 (the last sampling point). Soil samples were taken for analysis at zero time and 1, 4, 7, 14, 28, 45, 60, and 91 days after application.

3. Description of analytical procedures

The KOH traps for collecting $^{14}CO_2$ produced by the systems were collected at each sampling day after Day 0 and replaced with fresh material at each sampling event.

Soil samples were typically extracted four times with acetonitrile:0.1 M formic acid (aq) (1:1 v/v), centrifuged, and decanted. The extracts were analysed in triplicate for total radioactivity by LSC analysis. Soil extracts were combined, concentrated with rotary evaporator, mixed thoroughly, and filtered, then analysed using reverse phase HPLC (Phenomenex Aqua C18, 250 mm × 4.6 mm × 5µm id) eluted with a gradient of pH3 formic acid (aq) and acetonitrile. The effluent was passed through a UV detector (254 nm) to detect reference standards and a radioactivity detector to determine the quantities of radiolabeled degradation products present. The limit of detection (LOD) for oxamyl in KOH traps, organic extracts, soil combustions, and HPLC analyses (organic) were 15, 12, 17, and 100 dpm of applied $^{14}C$, respectively.

Soil samples were combusted, and unextracted $^{14}C$ levels were measured using LSC.

## II. RESULTS AND DISCUSSION

A. DATA

**Table 9 Distribution of radiolabeled components in aerobic soils after application of [1-$^{14}$C]oxamyl (% applied radioactivity)**

| Soil (20°C) | Sampling interval (days) | Oxamyl | IN-A2213 | IN-D2708 | $^{14}CO_2$ | Organic volatiles | Others[a] | Bound residue[b] | Total |
|---|---|---|---|---|---|---|---|---|---|
| Speyer | 0 | 97.7 | 0.0 | 0.0 | NA | NA | 1.9 | 0.3 | 99.8 |
| | 1 | 84.8 | 3.7 | 3.9 | 0.9 | NA | 0.0 | 1.0 | 94.3 |
| | 4 | 67.0 | 9.1 | 11.3 | 2.2 | NA | 0.0 | 1.9 | 91.4 |
| | 7 | 47.1 | 7.0 | 29.5 | 4.8 | NA | 0.0 | 3.3 | 91.7 |
| | 14 | 24.9 | 4.9 | 46.3 | 10.2 | NA | 0.0 | 5.6 | 91.9 |
| | 28 | 10.7 | 2.2 | 55.5 | 19.8 | NA | 0.2 | 6.8 | 95.2 |
| | 45 | 3.4 | 0.9 | 49.4 | 26.9 | NA | 0.2 | 8.7 | 89.5[c] |
| | 60 | 2.1 | 1.0 | 46.8 | 33.3 | NA | 0.0 | 9.0 | 92.2 |
| | 91 | 1.7 | 0.2 | 36.9 | 48.4 | NA | 0.2 | 14.1 | 101.4 |
| Tama | 0 | 92.5 | 0.0 | 0.0 | N/A | NA | 0.8 | 0.7 | 94.0 |
| | 1 | 86.4 | 5.2 | 3.4 | 0.9 | NA | 0.0 | 1.9 | 97.8 |
| | 4 | 63.5 | 13.5 | 11.3 | 2.3 | NA | 0.7 | 6.8 | 98.1 |
| | 7 | 47.7 | 9.0 | 20.2 | 5.2 | NA | 0.6 | 10.8 | 93.4 |
| | 14 | 35.5 | 5.0 | 25.2 | 16.4 | NA | 0.5 | 14.6 | 97.2 |
| | 28 | 15.6 | 2.7 | 15.6 | 34.5 | NA | 2.1 | 17.2 | 87.6 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 45 | 10.8 | 1.9 | 0.0 | 53.2 | NA | 1.3 | 18.2 | 85.5[c] |
| | 60 | 6.9 | 1.3 | 0.0 | 63.1 | NA | 1.1 | 20.1 | 92.5 |
| LRA-D | 0 | 95.1 | 0.0 | 0.0 | N/A | NA | 2.0 | 0.5 | 97.5 |
| | 1 | 85.3 | 2.4 | 3.5 | 0.7 | NA | 0.3 | 1.5 | 93.7 |
| | 4 | 69.6 | 4.7 | 10.6 | 2.7 | NA | 0.0 | 4.1 | 91.7 |
| | 7 | 57.5 | 5.8 | 17.2 | 6.5 | NA | 0.4 | 6.5 | 93.9 |
| | 14 | 37.7 | 5.7 | 25.9 | 15.2 | NA | 0.0 | 11.2 | 95.7 |
| | 28 | 19.7 | 3.4 | 27.4 | 27.3 | NA | 0.5 | 14.5 | 92.7 |
| | 45 | 12.5 | 1.9 | 25.9 | 35.7 | NA | 0.6 | 14.8 | 91.5 |
| | 60 | 12.5 | 1.8 | 25.3 | 40.6 | NA | 0.0 | 14.8 | 95.1 |
| | 91 | 8.4 | 0.7 | 18.4 | 46.3 | NA | 0.2 | 22.0 | 95.9 |
| Goch | 0 | 99.3 | 0.0 | 0.0 | N/A | NA | 0.9 | 0.3 | 100.5 |
| | 1 | 93.1 | 1.7 | 0.0 | 0.6 | NA | 0.7 | 1.0 | 97.0 |
| | 4 | 2.8 | 5.0 | 76.3 | 1.5 | NA | 0.0 | 9.5 | 95.1 |
| | 7 | 0.4 | 3.7 | 78.0 | 2.5 | NA | 0.0 | 9.8 | 94.3 |
| | 15 | 0.2 | 1.6 | 75.2 | 7.0 | NA | 0.0 | 11.4 | 95.3 |

[a] Sum of all unknown products

[b] Unextractable Residue in soil

NA = Not Applicable/Not analysed

[c] Time point had low recovery but was ≥90% at the next scheduled sampling.

C. MASS BALANCE

Material balance for the [1-$^{14}$C]oxamyl ranged from 93.3% to 96.4%.

D. BOUND AND EXTRACTABLE RESIDUES

The percentage of radioactivity in the extractable fraction decreased from Day 0 to Day 91 for all four soils. The level of bound residue increased steadily throughout the course of the study with all four soils. Extractability values for [1-$^{14}$C]oxamyl ranged from 99.6% AR (Day 0) to 39.0% AR (Day 91) for the Speyer soil, 93.3% AR (Day 0) to 9.3% AR (Day 60) for the Tama soil, 97.1% AR (Day 0) to 27.6% AR (Day 91) for the LRA-D soil, and 100.2% AR (Day 0) to 76.9% AR (Day 15) for the Goch soil.

Bound residue values for [1-$^{14}$C]oxamyl ranged from 0.3% AR (Day 0) to 14.1% AR (Day 91) for the Speyer soil, 0.7% AR (Day 0) to 20.1% AR (Day 60) for the Tama soil, 0.5% AR (Day 0) to 22.0% AR (Day 91) for the LRA-D soil, and 0.3% AR (Day 0) to 11.4% AR (Day 15) for the Goch soil.

E. VOLATILISATION

Volatile radioactivity, identified as $^{14}CO_2$ represented 63.1% of applied radioactivity at Day 60 for [1-$^{14}$C]oxamyl. The ultimate degradation product was carbon dioxide, showing that the compound was available for mineralisation by micro-organisms.

F. TRANSFORMATION OF PARENT COMPOUND

Levels of oxamyl in the soil declined continuously over a period of 91 days incubation. The $DT_{50}$ and $DT_{90}$ values for oxamyl ranged from 1.6 to 12.1 days and 5.3 to 40.2 days, respectively as shown in Table 10.

**Table 10 Summary of degradation parameters as persistence triggers for oxamyl**

| Study | Soil/Condition | $r^2$ | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|---|
| DuPont-39014 | Goch (Sandy Loam) 20°C | 0.9261 | 1.6 | 5.3 | SFO |
| | LRA-D (Sandy Loam) 20°C | 0.9824 | 12.1 | 40.2 | SFO |
| | Speyer (Loamy Sand) 20°C | 0.9863 | 7.2 | 24.0 | SFO |
| | Tama (Light Clay) 20°C | 0.9810 | 9.8 | 32.4 | SFO |

[a] Due a rapid degradation, enough data points were not available to produce robust kinetic fits.

The kinetics presented in this report are superseded by the FOCUS kinetic results derived in the modelling position paper DuPont-41859 EU (summarised in Point 0 of this document).

Two major degradation products were formed in soil. IN-A2213 accounted for 0.2% of applied radioactivity for [1-$^{14}$C]oxamyl after 91 days incubation. IN-D2708 accounted for 36.9% of applied radioactivity for [1-$^{14}$C]oxamyl after 91 days incubation.

**III. CONCLUSION**

Oxamyl degraded at 20°C in all test soils with $DT_{50}$ values ranging from 1.6 to 12.1 days and $DT_{90}$ values ranging from 5.3 to 40.2 days. The relative rate of degradation of oxamyl by the four soils was related to the microbial activity and organic matter content of the soils.

The results demonstrate that oxamyl is rapidly transformed in acidic soils over time and would extensively degrade to $CO_2$ and non-extractable residues.

(Clark, B., 2014)

The aerobic degradation study DuPont-39014, submitted for the first time in this submission and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (April 2002), and U.S. EPA OPPTS 835.4100 (Oct. 2008). A review of this study indicates that it fully meets the current guideline (OECD 307) and is relied upon.

**RMS comments and conclusion**

The study was conducted in accordance with OECD 307 and SETAC (1995) guidelines. The study was conducted according to Good Laboratory Practice. The study is considered acceptable.
The results demonstrate that oxamyl is rapidly transformed in acidic soils over time and would extensively degrade to $CO_2$ and non-extractable residues.

#### B.8.1.1.2 Anaerobic degradation

**B.8.1.1.2/01**

| | | |
|---|---|---|
| **Reference:**<br>-- | **Report:** | Spare, W.C. (1991); Anaerobic soil metabolism of [1-$^{14}$C]oxamyl in Madera, California soil<br><br>**DuPont Report No.:** AMR 1851-90<br><br>**Guidelines:** U.S. EPA 162-2 (1982) |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 311
   Purity: Radiochemical purity - >99%

**Materials and Methods:**

The metabolism and degradation of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410, batch 311, specific activity 78.2 µCi/mg, radiochemical purity >99%) were studied in a US sandy clay loam soil (from Madera, California) under aerobic conditions followed by anaerobic conditions. The initial aerobic portion of the study has been described in section B.8.1.1.1.

Details of the soil characteristics have already been given in Table 1. Soil microbial biomass measurements were not reported.

Thirty-eight vessels were originally prepared, each containing 50 g of soil (dry weight equivalent) dosed with ~475 µg [1- $^{14}$C]-oxamyl (following a pre-incubation period of approximately one week). This treatment rate of 9.5 mg a.s./kg dry soil corresponds to an application rate of 11.875 kg a.s./ha (based on a soil incorporation depth of 10 cm and the reported bulk soil density of 1.25 g/cm3), which is 216% of the maximum recommended use rate for oxamyl (5.5 kg a.s./ha).

All the test vessels were initially incubated under aerobic conditions in the dark at 25 ±1 °C, with the soil moisture content being maintained at 70-75% of field capacity. After 11 days aerobic incubation (corresponding approximately to one half-life for oxamyl under these aerobic incubation conditions), 20 flasks were converted to anaerobic conditions by adding deionised water (to a depth of about 1 cm) and purging nitrogen through the test vessels into the traps for volatiles. These flasks were then incubated under anaerobic conditions (nitrogen atmosphere) in the dark at 25 ±1 °C for up to a further 60 days. The remaining 18 flasks were left to incubate under aerobic conditions for up to a further 40 days (resulting in a maximum aerobic incubation period of 51 days).

Anaerobic vessels were removed for sampling at intervals of 1, 3, 7, 10, 20, 32, 45 and 60 days after conversion to anaerobic conditions. Traps for volatiles (2N $K_{OH}$ for carbon dioxide and polyurethane foam plugs for other volatile components) were replaced at the same time points also.

The flooded soil samples from the anaerobic phase of the study were extracted three times with a methanol/water mixture (50:50 v/v, 100 mL per extraction) and once with methanol (100 mL), with sonication being used to aid the extraction process. [This procedure constitutes a point of departure from SETAC guidance, which recommends that the overlying flood water be first removed from the soil solids, so that these components can be analysed separately.]

The extracts (containing radioactivity from the soil solids and flood water) were separated from the soil by centrifugation, decanted off and combined for each sample, in preparation for radioactivity quantification by LSC. Qualitative and quantitative determination of the distribution of [1- $^{14}$C]-oxamyl and its degradates in the extracts was undertaken using TLC. In addition, HPLC was used as a confirmatory chromatographic method for selected representative extracts.

Radioactivity remaining in the soil samples after the extractions was determined by LSC, following combustion of air-dried samples. Volatile radioactivity, collected in the 2N $K_{OH}$ trapping solutions and polyurethane foam plugs, was quantified using LSC; with the foam plugs being extracted by sonication with a methanol/water mixture (50:50 v/v, 10 mL) prior to analysis. The radioactivity in the 2N $K_{OH}$ traps was characterised as $^{14}CO2$ by precipitation of the barium salt of [ $^{14}$C]-carbonate. Aliquots from the 2N $K_{OH}$ traps were mixed with BaCl2, forming a white precipitate, which was removed by centrifugation. The absence of $^{14}CO2$ in the supernatants was confirmed using LSC.

**Findings:**

The levels (expressed as a percentage of applied radioactivity) of parent compound and degradates recovered at each sampling interval in the anaerobic phase of the study, together with mass balances, are shown in Table 11.

The total recovery of applied radioactivity (mass balance value) immediately prior to conversion to anaerobic conditions was 101.5% and during the first 32 days of anaerobic incubation varied from 92.7% (day 10) to 97.6% (day 1). Mass balance values for the two subsequently sampled timepoints in the anaerobic phase were significantly lower (72.9% for day 45 and 61.5% for day 60). In each case, the mass balance was calculated as the sum of volatile radioactivity in the $K_{OH}$ traps ( $^{14}CO2$), volatile radioactivity in the polyurethane foam plugs, radioactivity extracted from the soil and flood water and unextracted radioactivity remaining in the soil.

Some mineralisation occurred but was less than that observed in the aerobic portion of the study, where carbon dioxide accounted for 45.3% AR by 51 days (see Table 7). Evolution of carbon dioxide was more gradual under anaerobic conditions, with levels increasing over time to 12.0% AR by day 60 (from a value of 1.1% AR immediately prior to conversion to anaerobic conditions). Unextracted soil radioactivity also increased during the anaerobic incubation period, reaching a level of 18.4% AR by day 60 (compared with a value of 24.2% AR after 51 days aerobic incubation – see Table 7). Detected volatile radioactivity other than $^{14}CO2$ was negligible, with the radioactivity extracted from the polyurethane foam plugs accounting for <0.1% AR in all cases. However, it was speculated that the decline in mass balance towards the end of the anaerobic phase might have

been due to formation of a volatile component that was not captured in the trapping system or was lost during sample handling.

**Table 11 Mass balance and distribution of applied radioactivity in Madera sandy clay loam soil treated with [1-$^{14}$C]-oxamyl (9.5 mg a.s./kg) and incubated under anaerobic conditions**

| Sampling time | % of applied radioactivity | | | | | | |
|---|---|---|---|---|---|---|---|
| | Soil and flood water extraction | | | $^{14}CO_2$ | Other volatiles | NER | Total |
| | Oxamyl | IN-A2213 | IN-D2708 | | | | |
| Day 0 | 53.1 | 23.9 | 18.1 | 1.1 | nd | 5.3 | 101.5 |
| Day 1 | 56.4 | 24.7 | 12.5 | 1.2 | nd | 2.8 | 97.6 |
| Day 3 | 38.8 | 43.1 | 10.2 | 1.2 | nd | 3.2 | 96.5 |
| Day 7 | 29.3 | 48.5 | 12.7 | 1.4 | nd | 3.0 | 94.9 |
| Day 10 | 16.0 | 55.8 | 15.9 | 1.5 | nd | 3.5 | 92.7 |
| Day 20 | <0.1 | 69.5 | 18.5 | 2.0 | nd | 4.2 | 94.2 |
| Day 32 | <0.1 | 57.3 | 23.1 | 5.4 | nd | 8.9 | 94.7 |
| Day 45 | <0.1 | 40.2 | 9.0 | 9.4 | nd | 14.3 | 72.9 |
| Day 60 | <0.1 | 22.4 | 8.7 | 12.0 | nd | 18.4 | 61.5 |

NER = non-extractable radioactivity, nd = not detected (<0.1% AR)

Sampling time refers to days after conversion to anaerobic conditions. All samples were incubated aerobically for 11 days initially. Therefore, day-0 figures are the same as the day- 11 figures for the aerobic part of the study (see Table 7). The data presented are averages of two replicate samples for each timepoint (except day 0, which was from a single sample). Figures for extracted radioactivity (oxamyl, IN-A2213 and IN-D2708) refer to combined radioactivity from soil solids and overlying flood water, since these components were not separated prior to analysis.

Extractable radioactivity from the soil solids and overlying flood water (comprising parent oxamyl and degradates IN-A2213 and IN-D2708) accounted for 95.1% AR immediately prior to conversion to anaerobic conditions. By the end of the anaerobic phase (day 60), it accounted for 31.1% AR.

Oxamyl declined rapidly under anaerobic conditions, such that its level by day 10 (16.0% AR) was less than one-third of the level at the start of the anaerobic phase (53.1% AR), and by day 20 it accounted for <0.1% AR. The decline was faster than that observed in the aerobic portion of the study, where it took 10-11 days for oxamyl to decline to approximately half its initial level (see Table 7), despite the fact that mineralisation was less under anaerobic conditions than under aerobic conditions.

The major degradation products under anaerobic conditions were IN-A2213 and IN-D2708. Immediately prior to conversion to anaerobic conditions, IN-A2213 and IN-D2708 accounted for 23.9% AR and 18.1% AR respectively. Following the onset of anaerobic conditions, IN- A2213 increased to a maximum level of 69.5% AR on day 20 and then declined to 22.4% AR by the end of the anaerobic phase (day 60). The level of IN-D2708 decreased at first, reaching a value of 10.2% AR after 10 days anaerobic incubation, before increasing to a peak value of 23.1% AR on day 32 and then declining to 8.7% AR by day 60. [The maximum levels of IN-A2213 and IN-D2708 detected in the aerobic portion of the study were respectively 24.3% AR and 20.3% AR (see Table 7).]

The identities of IN-A2213 and IN-D2708 were confirmed by matching their retention times in samples to those of authentic standards using TLC and HPLC analytical methods. Apart from $^{14}$CO2, IN-A2213, IN-D2708 and unextractable soil residues, no other degradation products were detected in excess of 0.1% AR.

**Conclusions:**

Oxamyl was degraded extensively under anaerobic conditions in the dark, at 25 ±1 °C, following application at a nominal rate of 9.5 mg a.s./kg dry soil to a US sandy clay loam soil (Madera, California) and aerobic incubation in the dark for 11 days at 25 ±1 °C. Degradation was rapid, with the level of oxamyl on day 10

(16.0% AR) being less than one-third of the level at the start of the anaerobic phase (53.1% AR) and its level by day 20 being <0.1% AR. The decline was faster than that observed in the aerobic portion of the study, where it took 10- 11 days for oxamyl to decline to approximately half its initial level. However, mineralisation was less under anaerobic conditions than under aerobic conditions, with carbon dioxide increasing gradually throughout the anaerobic incubation period to reach a level of 12.0% AR by day 60 (as compared to a level of 45.3% AR by day 51 of the aerobic portion of the study). Evolution of carbon dioxide was paralleled by an increase in unextracted soil radioactivity, which reached a level of 18.4% AR by day 60 of the anaerobic phase (compared to a level of 24.2% AR after 51 days aerobic incubation).

IN-A2213 and IN-D2708 were the major degradation products under anaerobic conditions. The peak value for IN-D2708 under anaerobic conditions (23.1% AR, day 32) was similar to its aerobic peak value (20.3%, day 21), whereas the anaerobic peak value for IN-A2213 (69.5% AR, day 20) was significantly higher than its aerobic peak value (24.3% AR, day 10). These findings suggest that, compared to aerobic incubation, anaerobic incubation increased the rate of oxamyl degradation to IN-A2213 but slowed the rate of degradation of IN-A2213 to IN-D2708. By the end of the anaerobic phase (day 60), IN-A2213 and IN-D2708 had declined to respective levels of 22.4% AR and 8.7% AR. No other metabolites in excess of 0.1% AR were detected.

Overall, the results indicate that degradation of oxamyl in topsoil follows the same pattern under anaerobic and aerobic conditions, with the primary degradation pathway being hydrolysis to form IN-A2213, which subsequently degrades to IN-D2708, with carbon dioxide and unextractable soil residues being the final products.

The anaerobic degradation study AMR 1851-90, originally submitted under EU Rev8 Point IIA 7.1.1.1.2.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline U.S. EPA 162-2 (1982). A review of this study indicates that it partially meets the current guideline (OECD 307); deviations include a mass balance below 90% AR at the last two sampling points. However, reconduct is unlikely to yield a significantly different result because the route of degradation of oxamyl was already well demonstrated by the previous sampling points with compliant mass balance. At the last two sampling points, where the mass balance was low, oxamyl had already degraded to less than 1% AR and the degradation pattern was well established. This study was repeated with abbreviated sampling in Supplement 1 to this report (AMR 1851-90, Supplement No. 1, summarised below). In this supplement, a more elaborate volatile trapping system was used in attempts to capture any organic volatiles that may have not been trapped in the original study. No additional organic volatile metabolites were observed in the supplemental study. The results of this repeat experiment, therefore, demonstrate that the low mass balance in the final sampling points of the original study were not due to a failure to trap small organic volatiles but instead were likely due to insufficient trapping of the large amounts of $CO_2$ formed as a result of degradation. Thus, all possible metabolites of oxamyl formed under anaerobic conditions were identified in this original study, and the route of degradation is well defined. Therefore, this study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl

**B.8.1.1.2/02**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.1.1.2/01** | **Report:** | Spare, W.C. (1992); Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil, supplemental report<br><br>**DuPont Report No.:** AMR 1851-90, Supplement No. 1<br><br>**Guidelines:** U.S. EPA 162-2 (1982) **Deviations:** None<br><br>**Testing Facility:** Agrisearch, Inc, Frederick, Maryland, USA<br><br>**Testing Facility Report No.:** 1712 |

| | | |
|---|---|---|
| | | **GLP:** Yes<br><br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

The anaerobic biotransformation of radiolabeled oxamyl was studied in one soil system under aerobic conditions for 11 days, and then the soil was flooded to promote anaerobic conditions. The test soils were treated with radiolabeled oxamyl at a concentration of 10.7 μg a.s./g dry weight soil. Samples were incubated in darkness at 25 ± 1°C with a soil moisture content at 70–75% of field capacity.

The objective of this supplemental work was to confirm the pattern of metabolism seen in the original incubation of [1-$^{14}$C]oxamyl in Madera, California, soil under aerobic and anaerobic conditions. The number of sampling points was chosen to account for the low material balance originally observed at 45 and 60 days anaerobic incubation.

In the subject experiment, after 11 days of aerobic soil incubation, 74% of the applied dose was identified as oxamyl; the remainder comprised the two major transformation products, identified as oxime (13%) and IN-D2708 (8%) together with $^{14}$CO2 (3%) and bound residues (3%). The remaining test soil flasks were flooded with water and flushed with nitrogen for continued incubation under anaerobic conditions. After 60 days of incubation under anaerobic conditions (total of 71 days from dosing), 1% of the applied radioactivity was identified as oxamyl; the remainder was identified as oxime (3%), IN-D2708 (20%), $^{14}CO_2$ (21%), and bound residues (25%).

The half-life of oxamyl in soil under aerobic conditions was calculated, using least squares regression analysis, to be 22 days ($r^2$ = 0.977) using all data generated through 11 days (aerobic conditions). The half-life of oxamyl in soil under anaerobic conditions was 9 days ($r^2$=0.786).

The radioactivity material balance was 105% and 101% of applied at 0 and 11 days (1 half-life) of the aerobic phase and 64% and 70% for the anaerobic samplings at 45 and 60 days. The percentage of radioactivity in the extractable fraction decreased with time in the soil with a concomitant increase in the level of bound residue. Extractability values for the soil were 105% and 95% for days 0 and 11 (aerobic phase) and 23% and 4% of the applied for days 45 and 60 (anaerobic phase). The bound residues accounted for 0 and 3% of the applied on day 0 and day 11 and 25% of the applied after 45 and 60 days of anaerobic incubation.

The results demonstrate that oxamyl is transformed in soil over time and would extensively degrade to a number of metabolites, including oxime and IN-D2708, and non-extractable residues and be mineralized to $CO_2$.

In summary, this supplemental incubation gave results that were similar to those of the original study. Oxamyl had almost completely disappeared by 45 days, accounting for less than 1% of the applied radioactivity in the anaerobic samples. The non-volatile products were the same as those observed in the original study. The amount of bound radioactivity and the amount of $^{14}CO_2$ were somewhat higher than in the original study. An extensive volatile trapping regime was established to attempt to recover any possible volatiles produced during incubation. The only volatile product was $^{14}CO_2$. No radiocarbon was recovered above 0.1% in the acetone/dry ice "cold traps" or in the $CO_2$ traps which followed the FID combustor. Therefore, the low recoveries were not caused by loss of an organic volatile.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabeled test material: [1-$^{14}$C]oxamyl
   Radiolabeled position: 1-Oxamyl
   Lot/batch #: E62727-86
   Radiochemical purity: 98%
   Specific activity: 74.8 μCi/mg (33078 DPM/μg)
   Stability of test compound: The test substance was shown to be stable

2. Soil

One soil was chosen to represent conditions of use: a sandy clay loam soil from Madera, California, USA. Prior to the initiation of the supplemental incubation, the test soil was screened through a 2 mm wire mesh sieve and stored at 4°C. The moisture content of the sieved soil was determined by drying approximately 10 g of the soil to a constant weight in duplicate. Approximately two weeks before dosing the incubation, 50 g of test soil (dry weight equivalent) was added to each test vessel, and the soil was brought to 50% field capacity with deionized water. The vessels were stoppered with polyurethane plugs, weighed, and incubated at 25 ± 1°C in the dark.

**Table 12 Characteristics of test soils**

| Characteristic | Soil |
|---|---|
| Soil name or designation | Madera, CA |
| Origin location | Madera, CA, USA |
| % Sand | 52.8 |
| % Silt | 32.0 |
| % Clay | 15.2 |
| Texture | Sandy Loam |
| % Organic matter | 1.78 |
| pH | 7.8 |
| Cation exchange capacity (meq/100g) | 19.34 |
| Moisture holding capacity (%)<br>at 1/3 bar<br>at 15 bar | <br>26.7<br>11.1 |
| Bulk density (lb/ft$^3$) | 77.00 (1233 kg/m$^3$) |

B. STUDY DESIGN

1. Experimental conditions

The test system consisted of 12 Erlenmeyer flasks (250 mL) containing soil (50 g each, dry weight equivalent) fitted with glass inlet and outlet fittings and connected in series with glass tubing. Six flasks were connected in series for the aerobic phase of the study and six flasks were connected for the anaerobic phase. ·An aliquot of the dose solution was applied to each test soil sample to give a final concentration of approximately 10.7 ppm, which was equivalent to a field use rate of approximately 18 lb a.s./A (20 kg a.s./ha). The flasks were weighed periodically and deionized water was added when necessary to maintain the soil moisture content at 70 to 75% field capacity.

The outlet to each series of flasks was sealed until weekly flushing with air for aerobic or nitrogen for anaerobic incubation. An elaborate series of trapping solutions and conditions was established in an attempt to provide better total radiocarbon recovery during this supplemental incubation. At approximately weekly intervals, each series of flasks was flushed with compressed breathing air (aerobic incubation) or compressed nitrogen (anaerobic incubation) for one hour at approximately 40 mL/minute. The purge gas passed through the series of flasks through duplicate 1N KOH traps into a "cold trap" flask immersed in dry ice/acetone and then into a flame ionization detector (FID) to combust any volatile radiocarbon present. The exhaust from the flame was swept through duplicate traps containing $CO_2$ trapping cocktail.

All of the flasks were incubated aerobically in the dark in an environmental chamber at 25 ± 1°C for 11 days, which was the originally observed aerobic half-life of [1-$^{14}$C]oxamyl. The flasks were purged with humidified air one hour once per week at a flow rate of approximately 40 mL/min. After 11 days, six flasks (one series of six flasks) were converted to anaerobic conditions by adding deionized water (to a depth of about 1 cm) and purging nitrogen through the flasks and into traps to collect volatiles. The flasks were incubated under anaerobic conditions at 25°C in the dark for up to 60 days.

2. Sampling

During the aerobic phase, samples were taken at day 0 and 11 days (the aerobic half-life). Following conversion to anaerobic conditions (at the aerobic half-life, day 11), samples were taken at 45 and 60 days of incubation under anaerobic conditions. Duplicate flasks were taken at each time point. All samples were extracted and analysed immediately at each sampling.

3. Description of analytical procedures

The traps for volatiles were replaced with fresh solutions prior to each weekly collection. Aliquots of 1N KOH trap solutions were analysed for total radioactivity by LSC; the presence of $^{14}CO_2$ was verified by barium chloride ($BaC1_2$) precipitation. Following each trapping episode, the "cold trap" was rinsed 2–3 times with methanol, and the collected methanol was assayed by LSC. The $CO_2$ trapping scintillation fluid from the traps following FID combustion was counted directly by LSC.

All samples (except 60 day) were extracted twice with methanol/deionized water 1:1, (v:v) and once with methanol, centrifuged, and the supernatants decanted and filtered (glass wool). The combined extracts were then assayed by LSC to determine the total radioactivity. Prior to extracting the anaerobic day 60 samples, the flood water was decanted from each flask for LSC analysis and TLC. The soil remaining after decantation of the water was extracted as described above. After extraction, soil samples were combusted wet to determine total un-extracted radioactivity.

Soil extracts and 60 day water samples were qualitatively and quantitatively analysed by one dimensional TLC for $^{14}$C-labeled oxamyl and $^{14}$C-labeled degradates on silica gel plates using chloroform/ethanol 9:1 (v:v) as the mobile phase. The TLC plates were observed under UV light (254 nm), and the radiolabeled components quantitated using a radio-analytical imaging system; co-chromatography with known reference standards was used for identification. For additional confirmation, select soil extracts and 60 day water samples were analysed by reverse phase HPLC (PRP-1 column, 7 × 300 mm) eluted with a gradient of acetonitrile-deionised water (0:100 to 20:80 to 0:100, v/v). The elution of radioactivity was monitored by a radiochemical detector equipped with a calcium fluoride flow cell. The radioactive compounds were identified by comparing their retention times with those of co-chromatographed reference standards that were monitored by UV detection at 220 nm. The post-extraction soil samples were combusted and $^{14}$C levels were measured by LSC.

## II. RESULTS AND DISCUSSION

### A. DATA

**Table 13 Distribution of radiolabeled components in aerobic and anaerobic soils after application of $^{14}$C-oxamyl (% applied radioactivity, mean of duplicate replicates)**

| Soil (25°C) | Sampling interval (days) | Oxamyl | $^{14}CO_2$ | Organic volatiles | IN-D2708 (DMOA) | IN-A2213 (Oxime) | Bound residue[a] | Total |
|---|---|---|---|---|---|---|---|---|
| Madera | 0 | 105 (11.2)[b] | <0.1 (<0.1) | <0.1 (<0.1) | <0.1 (<0.1) | <0.1 (<0.1) | <0.1 (<0.1) | 105 (11.2) |
| | 11 | 74 (8.0) | 3 (0.3) | <0.1 (<0.1) | 8 (0.8) | 13 (1.4) | 3 (0.3) | 101 (10.8) |
| | 45[c] | 0.4 (<0.1) | 16 (1.7) | <0.1 (<0.1) | 20 (2.1) | 3 (0.3) | 25 (2.7) | 64 (6.9) |
| | 60[c,d] | 1.4 (0.1) | 21 (2.2) | <0.1 (<0.1) | 20 (2.1) | 3 (0.3) | 25 (2.7) | 70 (7.4) |

[a] Unextractable $^{14}$C-residues in soil.

[b] Values shown in parentheses are concentrations reported as parts per million (ppm).

[c] Days after establishment of anaerobic conditions by flooding the soils with water and purging with nitrogen.

[d] Includes aqueous layer, which accounted for 20% of the applied.

### C. MASS BALANCE

The radioactivity material balance was 105% and 101% of applied at 0 and 11 days (1 half-life) of the aerobic phase and 64% and 70% for the anaerobic samplings at 45 and 60 days.

### D. BOUND AND EXTRACTABLE RESIDUES

The percentage of radioactivity in the extractable fraction decreased with time in the soil. The level of bound residue increased steadily throughout the course of the study. Extractability values for the soil were 105% and 95% for days 0 and 11 (aerobic phase) and 23% and 4% of applied for Days 45 and 60 (anaerobic phase). The bound residue values ranged from 0 and 3% of applied (Days 0 and 11) to 25% of the applied (Days 45 and 60) for the soil.

E. VOLATILISATION

Confirmation of $^{14}CO_2$ in the KOH was performed for the soil. A barium chloride test performed on this system confirmed that the activity trapped was due to the presence of $^{14}CO_2$. At study termination, evolved $^{14}CO_2$ was 21% of the applied amount in the soil. Volatile organics accounted for <0.1% of the applied radioactivity in all samples of the aerobic and anaerobic incubation.

F. TRANSFORMATION OF PARENT COMPOUND

The half-life of oxamyl was calculated, using least squares regression analysis, to be 22 days with a correlation coefficient ($r^2$) of 0.977 using all data generated through 11 days. The major transformation products under aerobic conditions were identified as oxime, IN-D2708 ("DMOA"), and $CO_2$. After 60 days, 1% of the applied radioactivity was identified as oxamyl. The half-life and correlation coefficient ($r^2$) of oxamyl, calculated using least squares regression analysis were 9 days and 0.786, respectively. The major degradation products under anaerobic conditions were oxime, IN-D2708, and $CO_2$.

**III. CONCLUSION**

This supplemental incubation, performed to clarify the cause of the low recoveries at 45 and 60 days in the original study, gave results that were similar to those of the original study. Oxamyl had almost completely disappeared by 45 days, accounting for less than 1% of the applied radioactivity in the anaerobic samples. The non-volatile products were the same as those observed in the original study. The amount of bound radioactivity and the amount of $^{14}CO_2$ were somewhat higher than in the original study. An extensive volatile trapping regime was established to attempt to recover any possible volatiles produced during incubation. The only volatile product was $^{14}CO_2$. No radiocarbon was recovered above 0.1% in the acetone/dry ice "cold traps" or in the $CO_2$ traps that followed the FID combustor. Therefore, the low recoveries in the original study were not caused by loss of an organic volatile metabolite.

(Spare, W.C., 1992)

The anaerobic degradation study AMR 1851-90 supplement No. 1, submitted for the first time in this submission and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline U.S. EPA 162-2 (1982). A review of this study indicates that it partially meets the current guideline (OECD 307); deviations include a mass balance below 90% AR at the last two sampling points. However, reconduct is unlikely to yield a significantly different result because the route of degradation of oxamyl demonstrated in this abbreviated study is in line with that seen in AMR 1851-90. The purpose of this supplement was to utilize extensive trapping methods to specifically monitor for any small volatile metabolites (besides $C0_2$) that may be forming. This study adequately demonstrated that losses in material balance in the original study were in fact only $^{14}C0_2$ and that no additional volatile metabolites are formed. When this supplement is considered with the original study, AMR 1851-90, the anaerobic degradation pathway of oxamyl is fully captured and understood.

**RMS comments and conclusion**

The study was conducted partially in accordance with OECD 307 ( deviations include a mass balance below 90% AR at the last two sampling points) and USEPA 162-2 (1982) guidelines. The study was conducted according to Good Laboratory Practice. The study is considered reasonable acceptable.
The results demonstrate that losses in material balance in the original study were in fact only $C0_2$ and that no additional volatile metabolites are formed. The anaerobic degradation pathway of oxamyl is fully captured and understood.

#### B.8.1.1.3 Soil photolysis

**B.8.1.1.3/01**

| | | |
|---|---|---|
| **Reference:** **CA 7.1.1.3/01** | **Report:** | Habeeb, S.B. (2011); Photodegradation of $^{14}$C-oxamyl on soil<br>**DuPont Report No.:** DuPont-31501<br>**Guidelines:** OECD January (2002), SETAC Europe (1995), OPPTS 835.2410 (2008)<br>**Deviations:** None |

| | | |
|---|---|---|
| | | **Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** 66555<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

The photodegradation of oxamyl on non-sterile silty clay loam soil (USDA textural class, pH 6.7, organic matter content of 4.1% by Walkley Black method, common name Tama) from Stark County, Illinois, U.S.A., was investigated. Thin layers of soil (2 mm) were treated with oxamyl to obtain a concentration of 5.308 µg a.s./g dry weight soil. The temperature of the irradiated soil samples was maintained at approximately 21 ± 2°C with continuous irradiation for up to 15 days under simulated natural sunlight produced by a Xenon arc lamp. A set of non-irradiated controls were incubated at approximately 20 ± 2°C and kept in the dark in the environmental chamber.

Recovery of radioactivity ranged from 87.6% AR to 102.9% AR in all samples.

In the irradiated oxamyl samples, the degradation products were IN-D2708, IN-N0079, and IN-A2213, which reached average maximum concentrations of 44.7% (Day 15), 8.7% (Day 5), and 3.6% AR (Day 3), respectively.

In the non-irradiated oxamyl samples, the degradation products were IN-D2708 and IN-A2213, which reached average maximum concentrations of 6.7% (Day 11) and 8.0% AR (Day 3), respectively.

The $DT_{50}$ and $DT_{90}$ values using first order kinetics of oxamyl were 4.6 and 15.2 days in irradiated samples and 24.2 and 80.5 days in non-irradiated samples, respectively. The rate constant and $r^2$ values for the irradiated soil photolysis system were 0.1462 $day^{-1}$ and 0.9903 respectively. The rate constant and $r^2$ values for the non-irradiated soil photolysis system were 0.0286 $day^{-1}$ and 0.9498 respectively

**Kinetics information**

The kinetic analysis of this study has been updated to meet current FOCUS guidelines. This analysis supersedes the values presented in this report, and can be found in the modelling position paper DuPont-41859 EU (summarised in Point 0 of this document). The updated persistence endpoints for oxamyl and IN-N0079 are presented in the following tables.

**Table 14 Summary of degradation parameters as persistence triggers for oxamyl degradation in light (soil photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-31501 | Tama 20°C | 4.6 | 15.2 | SFO |

**Table 15 Summary of degradation parameters as persistence triggers for IN-N0079 (derived from oxamyl soil photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-31501 | Tama 20°C | 2 | 6.5 | SFO-SFO |

The results of this study indicate that oxamyl readily undergoes photolysis on soil surfaces. In the presence of light, oxamyl is converted to IN-N0079, which is the further degraded to IN-D2708 and ultimately mineralized to $CO_2$.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabelled test items: [1-$^{14}$C]oxamyl
   Lot/Batch #: 3560-135
   Radiochemical purity: 100%
   Specific activity: 23.8 μCi/mg
   Storage conditions: ≤-10°C
   Stability of test compound: The test substance was shown to be stable

2. Soil

The soil was a light clay (International textural class) from Stark County, Illinois, U.S.A. (common name Tama), with a pH of 6.7 (1:1 soil:water ratio) and organic matter content of 4.1% (Walkley Black method). This test soil was a representative agricultural soil where oxamyl is used.

B. STUDY DESIGN

1. Experimental conditions

The test vessels were clear, borosilicate glass scintillation vials. The vessels were placed into the water-jacketed beaker, covered with a quartz plate, and the plate was sealed. There were two test systems, one for each test condition (irradiated or dark control). The test equipment for irradiation consisted of stainless steel tanks with a quartz glass lid and inlet and outlet points designed for the collection of volatiles. The inlet and outlet points were kept sealed except for sampling. The test systems were maintained below the irradiation source at a distance to achieve a light intensity of 8.83 W/m$^2$. The tank was placed in the photolysis chamber under the Xenon arc light.

The test system to contain the non-irradiated test vessels was similar, except that the chamber was covered with nontransparent glass and sealed with aluminum foil to maintain dark conditions. The test systems were kept in the photolysis chamber under the Xenon arc light.

Eighteen samples in individual vials were prepared for irradiation; 16 samples in individual vials were prepared for the non-irradiated (dark) controls; and two samples in individual vials were prepared to serve as Day 0 samples. Individual sample vials composed of soil amended with [1-$^{14}$C]oxamyl. Thus, there were sufficient samples to accommodate 7 sampling points and have reserve samples to use as needed. Aliquots of soil (*ca* 1.162-g dry weight equivalents) were prepared and added to the sample vials. Each comprised a *ca 2* mm thin layer of soil with a relatively smooth surface. Air exiting the test apparatus flowed through an ethylene glycol trap and a 1 N KOH trap for collection of any volatile metabolites and $^{14}CO_2$, respectively. A flow rate that sustained a gentle stream of bubbles through the entire trapping series was maintained.

2. Description of analytical procedures for the test item

Individual irradiated and dark treatment samples were analysed immediately following test item application (zero time) and after the following time periods of irradiation or incubation in dark: 1, 3, 5, 7, 11, and 15 days. Day 0 served the irradiated and dark control systems. The soil samples were extracted at three times with 50:50 (v:v) mixture of acetonitrile and water. The samples were shaken, centrifuged, and the supernatants combined and radioassayed by LSC. A sub-sample of the combined soil extract was concentrated and analysed by HPLC after they were radioassayed by LSC to verify process recovery.

Since the extractability was less than 90% AR in Day 1 and Day 3 (replicate 1) and from Day 5 (both replicates) in the irradiated soils and dark control soils dosed with [1-$^{14}$C]oxamyl, the remaining non-extractable residue was subjected to humic/fulvic fractionation. The humic and fulvic acid fractions were radioassayed using LSC, and the remaining pellet associated with the humin was radioassayed by combustion followed by LSC analysis.

Metabolites were identified in the initial soil extractions by HPLC by retention time comparison and LC-MS/MS analysis comparison with authentic standards. A kinetic analysis was performed on the data generated by HPLC.

The detection limit of $^{14}$C for the subsamples (e.g., traps, organic extracts, combustions) and HPLC analyses were <1.0% of applied radiocarbon for each process.

The limit of quantitation (LOQ) of $^{14}C$ for the subsamples and HPLC analyses for [1-$^{14}C$]oxamyl were 0.51, 0.40, 2.5, 0.21, and 0.18% for the KOH trap, ethylene glycol trap, organic extracts, soil combustions, and HPLC analysis, respectively.

## II. RESULTS AND DISCUSSION

A. DATA

**Table 16 Mass balance of radioactivity in soil samples treated with [1-$^{14}C$]oxamyl expressed as a percentage of applied radioactivity (% AR), for irradiated samples**

| Component | | Replicate | Irradiation period following application (days) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | **0** | **1** | **3** | **5** | **7** | **11** | **15** |
| Volatiles | $CO_2$ | 1 | N/A | 0.55 | 1.24 | 3.83 | 8.06 | 13.1 | 17.5 |
| | | 2 | N/A | 0.55 | 1.24 | 3.83 | 8.06 | 13.1 | 17.5 |
| | | Mean | N/A | 0.55 | 1.24 | 3.83 | 8.06 | 13.1 | 17.5 |
| | Organic volatiles | 1 | N/A | 0.26 | 0.40 | 0.60 | 0.69 | 0.74 | 0.74 |
| | | 2 | N/A | 0.26 | 0.40 | 0.60 | 0.69 | 0.74 | 0.74 |
| | | Mean | N/A | 0.26 | 0.40 | 0.60 | 0.69 | 0.74 | 0.74 |
| | Total | 1 | N/A | 0.81 | 1.64 | 4.42 | 8.76 | 13.8 | 18.2 |
| | | 2 | N/A | 0.81 | 1.64 | 4.42 | 8.76 | 13.8 | 18.2 |
| | | Mean | N/A | 0.81 | 1.64 | 4.42 | 8.76 | 13.8 | 18.2 |
| Soil extract | | 1 | 96.9 | 89.5 | 89.3 | 84.1 | 76.4 | 71.3 | 59.3 |
| | | 2 | 99.3 | 95.0 | 91.4 | 84.0 | 78.5 | 71.6 | 57.6 |
| | | Mean | 98.1 | 92.3 | 90.3 | 84.0 | 77.4 | 71.5 | 58.4 |
| Fulvic acid | | 1 | NA | 1.57 | 4.34 | 5.16 | 6.30 | 7.35 | 8.56 |
| | | 2 | NA | NA | NA | 5.41 | 6.10 | 7.32 | 7.73 |
| | | Mean | NA | 1.57 | 4.34 | 5.29 | 6.20 | 7.33 | 8.15 |
| Humic acid | | 1 | NA | 0.15 | 0.55 | 0.25 | 0.21 | 0.12 | 0.18 |
| | | 2 | NA | NA | NA | 0.86 | 0.06 | 0.15 | 0.00 |
| | | Mean | NA | 0.15 | 0.55 | 0.55 | 0.14 | 0.14 | 0.09 |
| Non-extractables | | 1 | 0.34 | 0.67 | 1.11 | 1.56 | 1.93 | 3.62 | 4.19 |
| | | 2 | 0.34 | 2.53 | 5.28 | 1.60 | 1.85 | 3.29 | 4.04 |
| | | Mean | 0.34 | 1.60 | 3.19 | 1.58 | 1.89 | 3.45 | 4.11 |
| Mass balance | | 1 | 97.2 | 92.7 | 96.9 | 95.5 | 93.6 | 96.2 | 90.4 |
| | | 2 | 99.7 | 98.3 | 98.3 | 96.3 | 95.2 | 96.2 | 87.6 |
| | | Mean | 98.5 | 95.5 | 97.6 | 95.9 | 94.4 | 96.2 | 89.0 |
| **Overall mass balance mean (all sampling intervals)** | | | | | | | | | **95.3** |
| **Overall mass balance standard deviation (all sampling intervals)** | | | | | | | | | **2.9** |

Values are not rounded during calculations.

**Table 17 Mass balance of radioactivity in soil samples treated with [1-$^{14}$C]oxamyl expressed as a percentage of applied radioactivity (% AR), for non-irradiated samples**

| Component | | Replicate | Dark period following application (days) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | 0 | 1 | 3 | 5 | 7 | 11 | 15 |
| Volatiles | $CO_2$ | 1 | N/A | 0.07 | 0.80 | 1.93 | 3.31 | 5.17 | 7.43 |
| | | 2 | N/A | 0.07 | 0.80 | 1.93 | 3.31 | 5.17 | 7.43 |
| | | Mean | N/A | 0.07 | 0.80 | 1.93 | 3.31 | 5.17 | 7.43 |
| | Organic volatiles | 1 | N/A | 0.43 | 0.72 | 0.93 | 1.10 | 1.19 | 1.23 |
| | | 2 | N/A | 0.43 | 0.72 | 0.93 | 1.10 | 1.19 | 1.23 |
| | | Mean | N/A | 0.43 | 0.72 | 0.93 | 1.10 | 1.19 | 1.23 |
| | Total | 1 | N/A | 0.50 | 1.52 | 2.85 | 4.41 | 6.36 | 8.65 |
| | | 2 | N/A | 0.50 | 1.52 | 2.85 | 4.41 | 6.36 | 8.65 |
| | | Mean | N/A | 0.50 | 1.52 | 2.85 | 4.41 | 6.36 | 8.65 |
| Soil extract | | 1 | 96.9 | 97.9 | 94.9 | 88.8 | 81.0 | 82.2 | 75.1 |
| | | 2 | 99.3 | 101.2 | 93.8 | 86.4 | 86.9 | 84.6 | 74.5 |
| | | Mean | 98.1 | 99.6 | 94.3 | 87.6 | 84.0 | 83.4 | 74.8 |
| Fulvic acid | | 1 | NA | NA | NA | 2.84 | 3.26 | 5.39 | 6.88 |
| | | 2 | NA | NA | NA | 2.73 | 3.34 | 5.00 | 6.77 |
| | | Mean | NA | NA | NA | 2.79 | 3.30 | 5.19 | 6.82 |
| Humic acid | | 1 | NA | NA | NA | 0.31 | 0.00 | 0.15 | 0.12 |
| | | 2 | NA | NA | NA | 0.06 | 0.09 | 0.09 | 0.21 |
| | | Mean | NA | NA | NA | 0.18 | 0.05 | 0.12 | 0.17 |
| Non-extractables | | 1 | 0.34 | 1.00 | 3.50 | 1.23 | 2.00 | 3.64 | 4.48 |
| | | 2 | 0.34 | 1.15 | 2.99 | 1.24 | 1.88 | 3.05 | 4.53 |
| | | Mean | 0.34 | 1.08 | 3.25 | 1.23 | 1.94 | 3.35 | 4.51 |
| Mass balance | | 1 | 97.2 | 99.4 | 99.9 | 96.0 | 90.7 | 97.7 | 95.3 |
| | | 2 | 99.7 | 102.9 | 98.3 | 93.3 | 96.6 | 99.1 | 94.7 |
| | | Mean | 98.5 | 101.1 | 99.1 | 94.7 | 93.7 | 98.4 | 95.0 |
| **Overall mass balance mean (all sampling intervals)** | | | | | | | | | **97.2** |
| **Overall mass balance standard deviation (all sampling intervals)** | | | | | | | | | **2.8** |

Values are not rounded during calculations.

**Table 18 Phototransformation of [1-$^{14}$C]oxamyl, expressed as a percentage of applied radioactivity (% AR), for irradiated samples**

| Compound | Replicate | Sampling times (days) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 0 | 1 | 3 | 5 | 7 | 11 | 15 |
| Oxamyl | 1 | 95.4 | 73.4 | 55.6 | 40.3 | 32.5 | 20.6 | 10.6 |
| | 2 | 98.2 | 80.6 | 67.2 | 45.7 | 30.6 | 22.8 | 8.7 |
| | Mean | 96.8 | 77.0 | 61.4 | 43.0 | 31.5 | 21.7 | 9.7 |
| IN-D2708 | 1 | 1.5 | 13.1 | 25.3 | 32.3 | 40.9 | 45.4 | 44.4 |
| | 2 | 1.2 | 9.7 | 16.7 | 26.2 | 42.9 | 42.9 | 45.0 |
| | Mean | 1.3 | 11.4 | 21.0 | 29.2 | 41.9 | 44.1 | 44.7 |
| IN-N0079 | 1 | 0.0 | 3.0 | 4.6 | 7.1 | 0.9 | 4.3 | 0.7 |
| | 2 | 0.0 | 2.8 | 3.0 | 10.2 | 2.7 | 3.6 | 2.2 |
| | Mean | 0.0 | 2.9 | 3.8 | 8.7 | 1.8 | 3.9 | 1.5 |
| IN-A2213 | 1 | 0.0 | 0.0 | 2.8 | 3.7 | 2.1 | 1.1 | 1.4 |
| | 2 | 0.0 | 1.9 | 4.5 | 0.9 | 2.2 | 1.0 | 0.7 |
| | Mean | 0.0 | 1.0 | 3.6 | 2.3 | 2.2 | 1.0 | 1.0 |

**Table 19 Phototransformation of [1-$^{14}$C]oxamyl, expressed as a percentage of applied radioactivity (% AR), for non-irradiated samples**

| Compound | Replicate | Sampling times (days) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **11** | **15** |
| Oxamyl | 1 | 95.4 | 93.3 | 84.7 | 78.8 | 71.2 | 69.3 | 65.0 |
| | 2 | 98.2 | 97.7 | 85.2 | 77.6 | 76.4 | 71.2 | 60.8 |
| | Mean | 96.8 | 95.5 | 85.0 | 78.2 | 73.8 | 70.3 | 62.9 |
| IN-D2708 | 1 | 1.5 | 2.1 | 1.5 | 3.5 | 2.7 | 6.8 | 3.2 |
| | 2 | 1.2 | 1.5 | 1.3 | 3.4 | 4.1 | 6.5 | 3.9 |
| | Mean | 1.3 | 1.8 | 1.4 | 3.5 | 3.4 | 6.7 | 3.5 |
| IN-N0079 | 1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | 2 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | Mean | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| IN-A2213 | 1 | 0.0 | 2.5 | 8.7 | 6.5 | 7.1 | 6.1 | 4.6 |
| | 2 | 0.0 | 2.0 | 7.2 | 5.4 | 6.3 | 6.9 | 7.9 |
| | Mean | 0.0 | 2.3 | 8.0 | 6.0 | 6.7 | 6.5 | 6.2 |

B. MASS BALANCE

The material balance for the irradiated test systems remained relatively constant with individual values ranging from 87.6% AR to 99.7% AR (mean of 95.3% AR) over the incubation period. There was therefore no indication of loss of volatile components during the incubation period.

The material balance for the non-irradiated test systems remained relatively constant with individual values ranging from 90.7% AR to 102.9% AR (mean of 97.2% AR) over the incubation period. There was therefore no indication of loss of volatile components during the incubation period.

C. VOLATILISATION

Recovery from $CO_2$-related radioactivity ranged from 0% AR to 7.43% AR in all samples.

D. TRANSFORMATION OF PARENT COMPOUND

In the irradiated system with [1-$^{14}$C]oxamyl treated soil, the parent test substance accounted for 96.8% AR on Day 0 and 9.7% AR on Day 15. In the [1-$^{14}$C]oxamyl system, transformation products were IN-D2708, IN-N0079, and IN-A2213; reaching maximum mean concentrations of 44.7% AR (Day 15), 8.7% AR (Day 5), and 3.6% AR (Day 3), respectively.

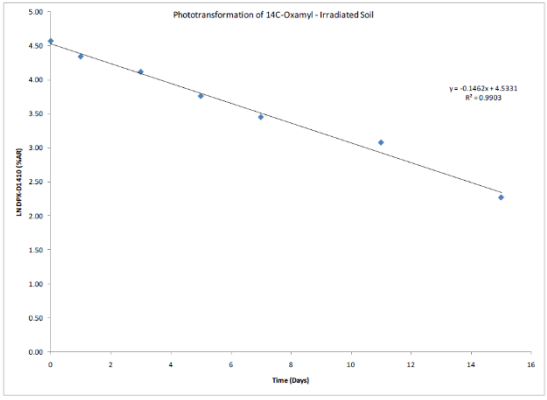
In the non-irradiated system with [1-$^{14}$C]oxamyl treated soil, the parent test substance accounted for 96.8% AR on Day 0 and 62.9% AR on Day 15. The major transformation products were IN-D2708 and IN-A2213; reaching maximum concentrations of 6.7% AR (Day 11), and 8.0% AR (Day 3), respectively.

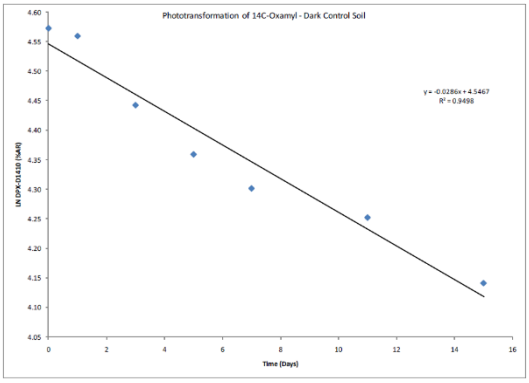
Phototransformation results are presented in Table 18 and Table 19.

E. KINETIC ANALYSIS OF DATA

The % AR from both irradiated and non-irradiated samples were plotted to derive rate constant and $r^2$ values.

**Figure 3 Kinetic model of oxamyl degradation in the irradiated and not irradiated soil photolysis study**




The kinetic analysis of this study has been updated to meet current FOCUS guidelines. This analysis supersedes the values presented in this report, and can be found in the modelling position paper DuPont-41859 EU (summarised in Point 0 of this document). The updated persistence endpoints for oxamyl and IN-N0079 are presented in the following tables.

**Table 20 Summary of degradation parameters as persistence triggers for oxamyl degradation in light (soil photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-31501 | Tama 20°C | 4.6 | 15.2 | SFO |

**Table 21 Summary of degradation parameters as persistence triggers for IN-N0079 (derived from oxamyl soil photolysis study)**

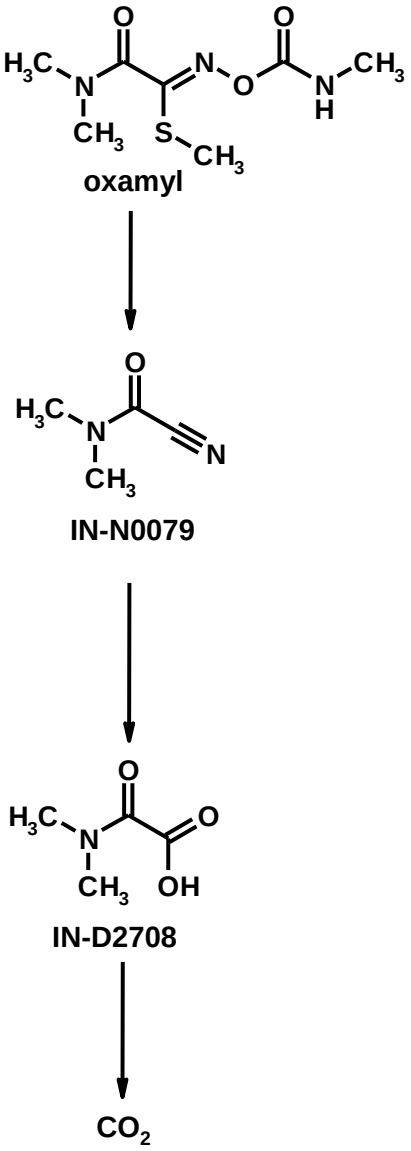
| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-31501 | Tama 20°C | 2 | 6.5 | SFO-SFO |

## III. CONCLUSION

The $DT_{50}$ and $DT_{90}$ values for oxamyl, determined using first order kinetics, were 4.6 and 15.2 days, respectively, in the irradiated samples and 24.2 and 80.5 days, respectively, in the non-irradiated samples. This indicates that oxamyl readily undergoes photolysis on soil surfaces. In the presence of light, oxamyl is converted to IN-N0079, which is the further degraded to IN-D2708 and ultimately mineralized to $CO_2$. The photodegradation pathway for oxamyl on soil is presented below.

(Habib, S., 2011)

**Figure 4 Proposed degradation pathway for the soil photolysis of oxamyl**




The aerobic degradation study DuPont-39014, submitted for the first time in this submission and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines OECD phototransformation of chemicals on soil draft guideline (January 2002), SETAC Europe (1995), and OPPTS 835.2410 (2008). A review of this study indicates that it fully meets the current guideline (OPPTS 835.2410 and OECD draft) and is relied upon.

**RMS comments and conclusion**

This study is considered as sufficient to support the data requirement for route of degradation in soil via photolysis. The study is considered acceptable.
The $DT_{50}$ and $DT_{90}$ values for oxamyl, determined using first order kinetics, were 4.6 and 15.2 days, respectively, in the irradiated samples.
In the irradiated system, up to three transformation products were IN-D2708, IN-N0079, and IN-A2213; reaching maximum mean concentrations of 44.7% AR (Day 15), 8.7% AR (Day 5), and 3.6% AR (Day 3), respectively. In the presence of light, oxamyl is converted to IN-N0079, which is the further degraded to IN-D2708 and ultimately mineralized to $CO_2$.

#### B.8.1.1.4 Overall assessment: Route of degradation

On the basis of the described soil metabolism studies, the primary route of degradation for oxamyl is *via* microbial decomposition and hydrolysis to form IN-A2213, which is further microbially degraded to IN-D2708 and then extensive mineralization to $CO_2$ and bound residue. In the presence of light, oxamyl can also undergo a photocatalysed, iron-mediated conversion to IN-N0079 as an additional minor degradation route. Degradation *via* this alternate, light-mediated, route does also proceed to formation of IN-D2708, which can then undergo microbial mineralization. Therefore, the proposed degradation pathway of oxamyl in soil is as shown in Figure 3. The maximum occurrences of the major soil metabolites in the laboratory degradation studies are listed in Table 22.

**Figure 3 Pathway of oxamyl degradation in soil**

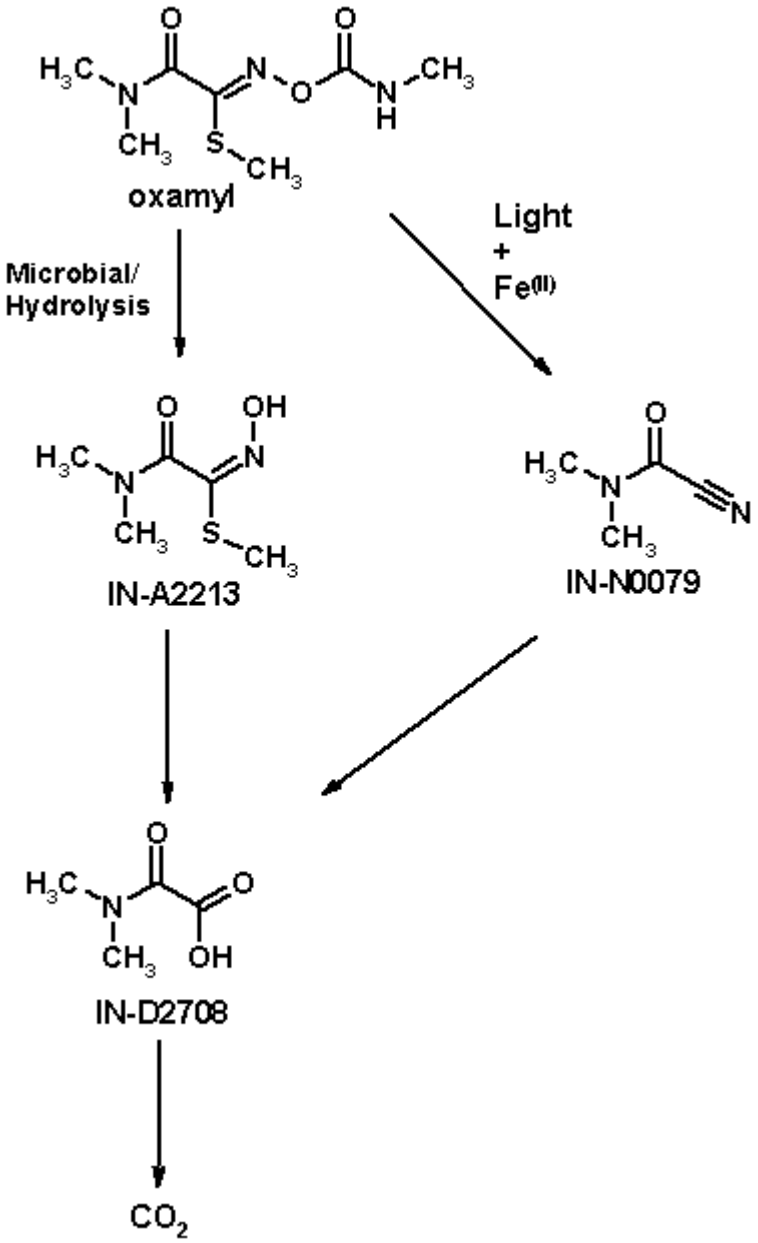


**Table 22 Maximum occurrences of oxamyl metabolites in laboratory soils**

| Soil | IN-A2213 | IN-D2708 | IN-N0079 |
|---|---|---|---|
| **Aerobic laboratory degradation studies, occurrences in % AR** | | | |
| Nijmegen | 15.0 | 29.6 | n.d.[a] |
| Commerce (20°C) | 51.0 | 25.7 | n.d. |
| Commerce (10°C) | 33.8 | 39.5 | n.d. |
| Gross Umstadt | 24.9 | 34.7 | n.d. |
| Drummer | 7.6 | nd | n.d. |
| Madera | 24.3 | 20.3 | n.d. |
| Goch | 5.0 | 78.0 | n.d. |
| Speyer | 7.0 | 55.5 | n.d. |
| LRA-D | 5.8 | 27.4 | n.d. |
| Tama | 13.5 | 25.2 | n.d. |
| **Soil photolysis, occurrences in % AR** | | | |
| Tama | 4.5 | 45 | 10.2 |
| **Overall maximum** | **51.0** | **78.0** | **10.2** |

[a] n.d.= not detected

### B.8.1.2 Rate of degradation in soil

#### B.8.1.2.1 Laboratory studies

RMS comments and conclusion

zRMS precise that all kinetic evaluations performed in studies B.8.1.2.1.1/01 to 04 are superseded by the FOCUS kinetic results derived in study summarised in Points B.8.1.2.1.1/05 and B.8.1.2.1.2/03

*B.8.1.2.1.1 Aerobic degradation of the active substance*

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.1.2.1.1/01**

| | | |
|---|---|---|
| **Reference:** -- | **Report:** | Mattson, S.L., Smyser, B.P. (2000); Rate of degradation of oxamyl in three aerobic soils<br>**DuPont Report No.:** DuPont-2957<br>**Guidelines:** SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), U.S. EPA 162-1 (1982) |

**Summary:**

Oxamyl degraded with a $DT_{50}$ ($DT_{90}$) of 3.0 (9.9), 16.4 (54.4), 4.1 (13.6) and 112.2 (372.6) days in the Commerce 20 °C, Commerce 10 °C, Gross Umstadt and Drummer soils, respectively. There were two significant degradation products - Oxamyl oxime (IN-A2213) and N,N-dimethyloxamic acid (DMOA, IN-D2708). Oxamyl oxime, had a $DT_{50}$ ($DT_{90}$) of 5.9 (19.7), 21.5 (71.3), 1.7 (5.7) and 17.5 (58.2) days in the Commerce 20 °C, Commerce 10 °C, Gross Umstadt and Drummer soils, respectively. DMOA, degraded with a $DT_{50}$ ($DT_{90}$) of 3.6 (12.1), 65.9 (219.1), and 3.4 (11.2) days in the Commerce 20 °C, Commerce 10 °C, and Gross Umstadt, respectively. DMOA was not seen in the Drummer soil. At the last sampling interval, $CO_2$ represented approximately 73.1, 81.0, 76.1, and 25.6% of applied radioactivity in the Commerce 20 °C, Commerce 10 °C, Gross Umstadt and Drummer soils, respectively.

**Materials and Methods:**

The rate of degradation of [1-$^{14}$C]-oxamyl ([1-$^{14}$C]-DPX-D1410) in aerobic soil was studied in three field-moist soils at 20 °C, two of which were from the USA and one from Germany. The soils used were 'Commerce' (silt

loam, USA), 'Drummer #6' (silty clay loam, USA) and 'Gross Umstadt' (silt loam, Germany). In addition, the aerobic soil metabolism of [1-$^{14}$C]-oxamyl at 10 °C was investigated using Commerce soil.
Soil samples were used within three months of collection from the field, and soil biomass was measured at the beginning of the study and towards the end of the incubation periods. Details of the soil characteristics are given in Table 23.
Prior to incubation under study conditions, purified water was added to the Commerce and Drummer #6 soils, with Commerce being adjusted to 40% of its 0-bar moisture content (resulting in 13.3 g water/100 g dry soil) and Drummer #6 being adjusted to 47% of its 0-bar moisture content (resulting in 23.2 g water/100 g dry soil). Gross Umstadt soil was used as received, with a moisture content of 40% of its 0-bar moisture-holding capacity (corresponding to 20 g water/100 g dry soil).
Following a pre-incubation period of 7-9 days, samples of the moist soils (equivalent to 50 g dry soil) were treated with ~100 µg [1-$^{14}$C]-oxamyl. This treatment rate of 2 mg a.s./kg dry soil corresponds to an application rate of 2.46 kg a.s./ha for Commerce soil, 1.86 kg a.s./ha for Gross Umstadt soil and 2.34 kg a.s./ha for Drummer #6 soil (based on a soil incorporation depth of 10 cm and respective reported bulk soil densities of 1.23 g/cm3, 0.93 g/cm3 and 1.17 g/cm3). When expressed in percentage terms as a proportion of the maximum recommended use rate for oxamyl (5.5 kg a.s./ha), these theoretical application rates are respectively 45%, 34% and 43%.
Sufficient samples were prepared so that replicates could be taken for analysis at each sampling timepoint. Treated samples of all three soils were incubated at 20 °C under aerobic conditions in the dark (for up to 123 days). A separate incubation was carried out at 10 °C using Commerce soil, with treated samples being incubated under aerobic conditions in the dark for up to 179 days. The moisture contents of all the incubated soils were maintained at 40-50% of 0-bar moisture-holding capacity throughout the incubation periods (by the addition of purified water as necessary).

**Table 23 Characteristics of Commerce, Gross Umstadt and Drummer #6 soils used to investigate the aerobic soil metabolism of oxamyl**

| Parameter | Commerce | Gross Umstadt | Drummer #6 |
|---|---|---|---|
| Origin | Greenville, MS, USA | Gross Umstadt, Germany | Rochelle, IL, USA |
| USDA texture | silt loam | silt loam | silty clay loam |
| % sand | 32.8 | 5.6 | 8.4 |
| % silt | 56.4 | 77.2 | 60.8 |
| % clay | 10.8 | 17.2 | 30.8 |
| pH | 7.0 | 7.8 | 4.8 |
| Organic matter (%) | 0.4 | 2.1 | 4.4 |
| Organic carbon (%) | 0.2 | 1.2 | 2.6 |
| Cation exchange capacity (meq/100 g) | 6.7 | 9.6 | 26.3 |
| Bulk density (g/cm$^3$) | 1.23 | 0.93 | 1.17 |
| 0-bar moisture (%) | 33.3 | 50.0 | 49.4 |
| Initial biomass (mg microbial C/100 g soil) | 5.31 | 30.73 | 12.3 |
| Final biomass (mg microbial C/100 g soil) | 4.76 | 29.7 | 7.33 |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

Duplicate treated soil samples were removed for analysis at each sampling timepoint. The sampling intervals (days after test substance application) for the 20 °C incubations were 0, 1, 2, 3, 7, 11, 14, 21, 32 and 60 (Commerce soil), 0, 1, 2, 3, 7, 10, 14, 21 and 31 (Gross Umstadt soil), and 0, 1, 2, 3, 7, 11, 14, 21, 32, 60, 90 and 123 (Drummer #6). In the case of the 10 °C incubation (Commerce soil), the sampling intervals were 0, 3, 7, 14, 21, 32, 46, 60, 90, 123 and 179 days after test substance application.

The collected soil samples were typically extracted four times with an acetonitrile/water mixture (50:50 v/v, 100 mL per extraction), with the extracts being separated from the soil by centrifugation and then decanted off, in preparation for radioactivity quantification by LSC. Qualitative and quantitative determination of the distribution of [1-$^{14}$C]-oxamyl and its degradates in the soil extracts was undertaken using reversed phase HPLC with radiochemical detection, fraction collection and liquid scintillation analysis.

The limits of detection for extract analysis, HPLC analysis and combustion analysis were respectively 0.30% AR (0.006 mg oxamyl equivalents/kg soil), 0.36% AR (0.0072 mg oxamyl equivalents/kg soil) and 0.02% AR (0.0004 mg oxamyl equivalents/kg soil).

**Findings:**

The levels (expressed as a percentage of applied radioactivity) of parent compound and degradates recovered at each sampling interval in the study, together with mass balances, are shown in Table 24.
Total recoveries of applied radioactivity (mass balance values) from the different incubations ranged from 99.0-103.6% (Commerce, 20 °C), 100.3-114.0% (Gross Umstadt, 20 °C), 101.3-109.9% (Drummer #6, 20 °C) and 96.3-121.9% (Commerce, 10 °C). In each case, the mass balance was calculated as the sum of volatile radioactivity in the KOH traps ( $^{14}CO2$), volatile radioactivity in the ethylene glycol trap, radioactivity extracted from the soil and unextracted radioactivity remaining in the soil.

Soil-extractable radioactivity comprised parent oxamyl, degradates IN-A2213 and IN-D2708 and other extractable radioactivity for the Commerce and Gross-Umstadt soils, and comprised oxamyl, IN-A2213 and other extractable radioactivity for Drummer #6 soil. Levels of soil- extractable radioactivity decreased from initial values of around 100% AR to final values of 3.7% AR (Commerce, 20 °C – day 60), 4.5% AR (Gross Umstadt, 20 °C – day 31), 59.8% AR (Drummer #6, 20 °C – day 123) and 23.1% AR (Commerce, 10 °C – day 179).

Parent oxamyl declined rapidly in the Commerce and Gross Umstadt soils incubated at 20 °C, such that it was not detected by day 32 in Commerce soil and only accounted for 0.2% AR in Gross Umstadt soil by the end of the incubation period (31 days). In both these incubations, oxamyl accounted for <50% AR by day 7. As expected, degradation of oxamyl in 10 °C Commerce soil was slower than in 20 °C Commerce soil. However, degradation was still quite fast in Commerce soil at 10 °C, with oxamyl accounting for <50% AR by day 21.

Degradation of oxamyl in Drummer #6 soil (incubated at 20 °C) was considerably slower, with oxamyl accounting for 51.2% AR by the end of the incubation period (123 days). This is consistent with the lower extent of mineralisation observed in this soil (25.6% AR maximum). It appears that Drummer #6 soil may have had a less viable microbial population during its incubation than the Commerce and Gross Umstadt soils, since the initial and final microbial biomass measurements for Drummer #6 soil were both <1% of the total soil organic carbon content (accounting respectively for 0.48% and 0.29% of total organic carbon content).

**Table 24 Distribution of applied radioactivity for 20 °C incubations (Commerce, Gross Umstadt and Drummer #6 soils) and 10 °C incubation (Commerce soil)**

| DAA | Oxamyl | IN-A2213 | IN-D2708 | $^{14}CO_2$ | Volatiles | Others | NER | Total |
|---|---|---|---|---|---|---|---|---|
| **Commerce – 20 °C** | | | | | | | | |
| 0 | 97.3 | 1.1 | - | - | - | 0.9 | 2.5 | 101.9 |
| 1 | 79.1 | 17.2 | 0.9 | 0.6 | 0.00 | 0.6 | 1.5 | 100.0 |
| 2 | 68.5 | 27.8 | 2.9 | 1.2 | 0.00 | 1.1 | 1.7 | 103.2 |
| 3 | 53.2 | 38.5 | 5.9 | 1.5 | 0.01 | 1.6 | 2.9 | 103.6 |
| 7 | 10.8 | 51.0 | 19.6 | 6.9 | 0.01 | 3.5 | 7.4 | 99.0 |
| 11 | 5.0 | 39.3 | 25.7 | 15.1 | 0.02 | 3.8 | 10.6 | 99.5 |
| 14 | 3.0 | 30.4 | 25.3 | 24.1 | 0.04 | 2.7 | 14.8 | 100.4 |
| 21 | 0.8 | 16.8 | 12.4 | 47.5 | 0.05 | 1.4 | 23.1 | 102.0 |
| 32 | nd | 3.4 | nd | 65.2 | 0.05 | 1.5 | 26.4 | 101.5 |
| 60 | nd | nd | nd | 73.1 | 0.05 | 0.4 | 24.1 | 100.9 |
| **Gross Umstadt – 20 °C** | | | | | | | | |
| 0 | 91.3 | 13.8 | - | - | - | 0.7 | 1.0 | 107.9 |
| 1 | 78.5 | 16.6 | 3.2 | 1.0 | 0.00 | 1.0 | 2.2 | 102.5 |
| 2 | 73.7 | 24.9 | 8.7 | 2.1 | 0.00 | 0.9 | 3.6 | 114.0 |
| 3 | 60.6 | 20.9 | 13.0 | 3.3 | 0.00 | 0.7 | 5.2 | 103.9 |
| 7 | 32.2 | 18.3 | 31.0 | 10.1 | 0.01 | 0.6 | 10.1 | 102.3 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 10 | 17.5 | 12.5 | 34.7 | 20.8 | 0.01 | 0.3 | 16.2 | 102.3 |
| 14 | 11.6 | 5.1 | 20.6 | 40.1 | 0.01 | 0.3 | 22.4 | 100.3 |
| 21 | 2.4 | 1.1 | 2.5 | 63.7 | 0.01 | nd | 26.4 | 100.9 |
| 31 | 0.2 | 0.2 | 0.3 | 76.1 | 0.01 | nd | 23.7 | 104.3 |
| **Drummer #6 – 20 °C** | | | | | | | | |
| 0 | 97.2 | - | - | - | - | 0.8 | 3.3 | 101.3 |
| 1 | 100.0 | 0.8 | - | 0.3 | 0.00 | 0.9 | 2.3 | 104.2 |
| 2 | 98.0 | 0.3 | - | 0.4 | 0.00 | 1.0 | 2.6 | 103.0 |
| 3 | 103.8 | 1.7 | - | 0.6 | 0.00 | 1.0 | 2.7 | 109.9 |
| 7 | 96.5 | 3.2 | - | 1.2 | 0.00 | 1.0 | 4.6 | 106.5 |
| 11 | 93.2 | 3.8 | - | 2.1 | 0.00 | 1.3 | 4.9 | 105.3 |
| 14 | 91.9 | 4.1 | - | 2.9 | 0.00 | 1.0 | 6.6 | 106.5 |
| 21 | 84.8 | 5.5 | - | 4.4 | 0.00 | 1.0 | 7.4 | 103.1 |
| 32 | 79.3 | 6.7 | - | 6.9 | 0.01 | 1.3 | 9.8 | 104.0 |
| 60 | 66.7 | 7.6 | - | 12.4 | 0.01 | 1.9 | 13.9 | 102.5 |
| 90 | 55.6 | 7.0 | - | 17.9 | 0.01 | 2.4 | 19.3 | 102.2 |
| 123 | 51.2 | 6.0 | - | 25.6 | 0.01 | 2.7 | 20.7 | 106.2 |
| **Commerce – 10 °C** | | | | | | | | |
| 0 | 95.1 | 1.8 | - | - | - | 1.0 | 2.7 | 100.6 |
| 3 | 94.6 | 6.9 | 0.4 | 0.6 | 0.00 | 0.8 | 1.3 | 104.5 |
| 7 | 80.6 | 15.8 | 2.6 | 1.4 | 0.00 | 0.8 | 2.4 | 103.6 |
| 14 | 55.6 | 28.1 | 7.8 | 3.7 | 0.00 | 2.0 | 4.4 | 101.5 |
| 21 | 39.1 | 33.6 | 14.1 | 6.0 | 0.01 | 2.0 | 4.9 | 99.6 |
| 32 | 26.8 | 33.8 | 20.7 | 11.1 | 0.01 | 2.7 | 6.6 | 101.0 |
| 46 | 13.8 | 30.9 | 27.4 | 12.2 | 0.01 | 2.1 | 10.6 | 97.0 |
| 60 | 5.7 | 19.5 | 37.6 | 19.3 | 0.01 | 1.6 | 12.6 | 96.3 |
| 90 | 0.4 | 9.5 | 39.5 | 35.2 | 0.02 | 1.1 | 15.3 | 101.0 |
| 123 | 0.3 | 4.1 | 33.5 | 52.1 | 0.04 | 0.5 | 15.4 | 109.6 |
| 179 | nd | 0.5 | 20.1 | 81.0 | 0.08 | 0.5 | 17.7 | 121.9 |

DAA = days after application, NER = non-extractable radioactivity, nd = not detected (limit of detection was 0.36% AR)
The figures for each timepoint are an average from two replicates.

OECD Test Guideline 307 ('Aerobic and Anaerobic Transformation in Soil' – adopted 24 April 2002) recommends that soils selected for study should have a microbial biomass of at least 1% of total organic carbon. [This soil quality criterion was agreed at an OECD workshop on the selection of soils and sediments, held in Belgirate, Italy in 1995.1] The strongly acidic nature of Drummer #6 soil (pH of 4.8) may have resulted in a stressed microbial population and may also have inhibited degradation of oxamyl by hydrolysis to IN- A2213, which is a base-catalysed reaction.

IN-A2213 was the initial soil degradation product of oxamyl in all cases. The time at which maximum levels of IN-A2213 were detected varied according to the rate of oxamyl degradation, with the time of maximum detect being nearer to the beginning of the incubation period for the faster oxamyl-degrading incubations (Commerce, 20 °C and Gross Umstadt, 20°C). Maximum detected levels of IN-A2213 were 51.0% AR (Commerce, 20 °C – day 7), 24.9% AR (Gross Umstadt, 20 °C – day 2), 7.6% AR (Drummer #6, 20 °C – day 60) and 33.8% AR (Commerce, 10 °C – day 32). By the end of the incubations, IN-A2213 had declined to final detected levels of 3.4% AR (Commerce, 20 °C – day 32), 0.2% AR (Gross Umstadt, 20 °C – day 31), 6.0% AR (Drummer #6, 20 °C – day 123) and 0.5% AR (Commerce, 10 °C – day 179). The lower levels of IN-A2213 observed in Drummer #6 soil (all <10% AR) may be due to a combination of slow formation (via oxamyl degradation) and relatively fast degradation of IN-A2213 to subsequent products in this case (including carbon dioxide and unextracted radioactivity).

In the Commerce and Gross Umstadt soils, IN-D2708 became a major soil degradation product as IN-A2213 declined, reaching maximum levels of 25.7% AR (Commerce, 20 °C – day 11), 34.7% AR (Gross Umstadt, 20 °C – day 10) and 39.5% AR (Commerce, 10 °C – day 90). Levels of IN-D2708 then decreased, such that by the end of the incubation periods it accounted for less than detectable levels in 20 °C Commerce soil, 0.3% AR (Gross Umstadt, 20 °C) and 20.1% AR (Commerce, 10 °C). The identities of IN-A2213 and IN-D2708 were

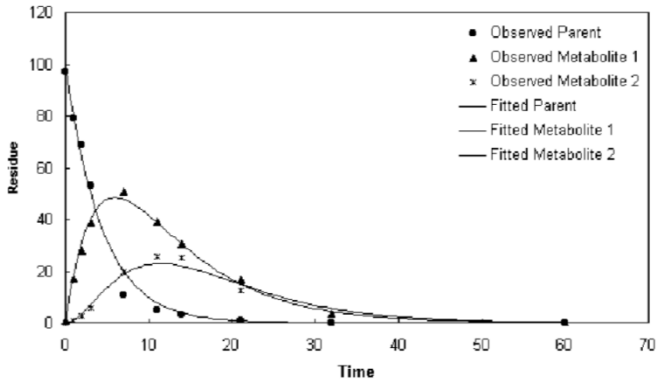
confirmed by matching the retention time in samples to that of authentic standards using two separate HPLC analytical methods.

IN-D2708 was not seen in Drummer #6 soil. The slow rate of oxamyl degradation in this soil produced low levels of IN-A2213, which appears to be the precursor for IN-D2708 formation. Apart from $^{14}CO2$, IN-A2213 and IN-D2708, no other significant degradation products were identified. Other soil-extractable radioactivity accounted for maximum values of 3.8% AR (Commerce, 20 °C – day 11), 1.0% AR (Gross Umstadt, 20 °C – day 1), 2.7% AR (Drummer #6, 20 °C – day 123) and 2.7% AR (Commerce, 10 °C – day 32).

Degradation rates for oxamyl, oxamyl oxime (IN-A2213) and DMOA (IN-D2708) were determined from the reported data using a nonlinear regression of conventional first-order kinetic equations. The software used for this fitting procedure was ModelManager, Version 1.1 (Cherwell Scientific Limited, 1999).
Table 24 contain values for oxamyl, oxamyl oxime, and DMOA (average of the two replicates, as %AR) remaining at each sampling point. The average %AR of each was plotted as a function of time and the best fit determined in ModelManager are depicted in Figure 5 .

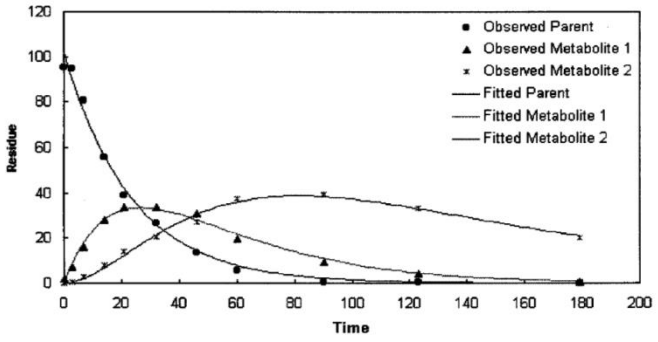
**Figure 5 Soil degradation kinetics of oxamyl, oxamyl-oxime and DMOA**

**A. Commerce Soil at 20°C**



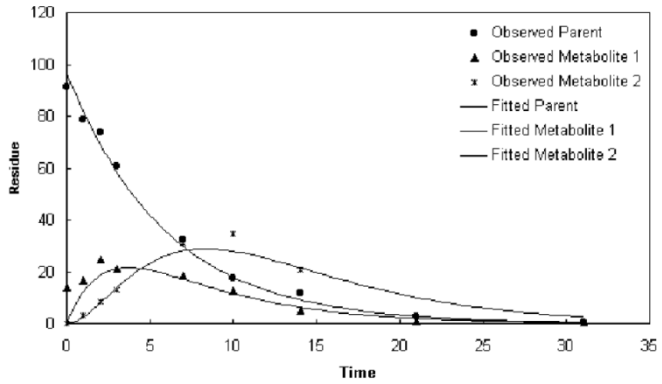

Parent = Oxamyl (DPX-D1410)
Metabolite 1 = Oxamyl oxime (IN-A2213)
Metabolite 2 = DMOA (IN-D2708)

**B. Commerce Soil at 10°C**



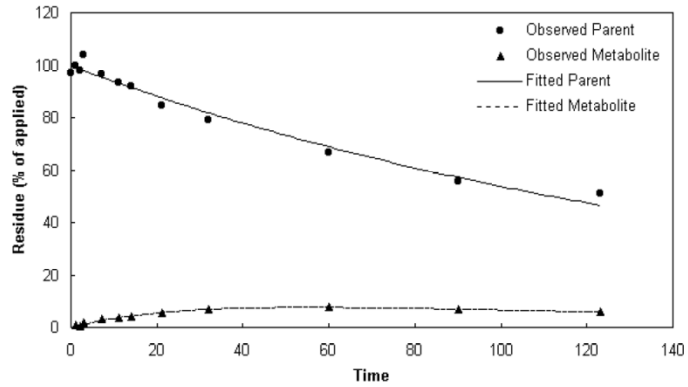

Parent = Oxamyl (DPX-D1410)
Metabolite 1 = Oxamyl oxime (IN-A2213)
Metabolite 2 = DMOA (IN-D2708)

**C. Gross Umstadt Soil**




Parent = Oxamyl (DPX-D1410)
Metabolite 1 = Oxamyl oxime (IN-A2213)
Metabolite 2 = DMOA (IN-D2708)

**D. Drummer Soil**




Parent = Oxamyl (DPX-D1410)
Metabolite 1 = Oxamyl oxime (IN-A2213)

A $DT_{50}$ (half-life) and $DT_{90}$ for oxamyl, oxamyl oxime, and DMOA in all soils (except Drummer) were determined. DMOA was not present in the Drummer soil.
These values are as follows:

| **Oxamyl** | **Soil** | **$DT_{50}$ (days)** | **$DT_{90}$ (days)** |
|---|---|---|---|
| | Commerce 20 °C | 3.0 | 9.9 |
| | Commerce 10 °C | 16.4 | 54.4 |
| | Gross Umstadt 20 °C | 4.1 | 13.6 |
| | Drummer#6 20 °C | 112.2 | 372.6 |
| **Oxime** | **Soil** | **$DT_{50}$ (days)** | **$DT_{90}$ (days)** |
| | Commerce 20 °C | 5.9 | 19.7 |
| | Commerce 10 °C | 21.5 | 71.3 |
| | Gross Umstadt 20 °C | 1.7 | 5.7 |
| | Drummer#6 20 °C | 17.5 | 58.2 |
| **DMOA** | **Soil** | **$DT_{50}$ (days)** | **$DT_{90}$ (days)** |
| | Commerce 20 °C | 3.6 | 12.1 |
| | Commerce 10 °C | 65.9 | 219.1 |
| | Gross Umstadt 20 °C | 3.4 | 11.2 |

**Conclusions:**

Following application at a nominal rate of 2 mg a.s./kg dry soil, oxamyl was degraded extensively, under aerobic conditions in the dark, in Commerce (silt loam, USA) and Gross Umstadt (silt loam, Germany) soils

incubated at 20 °C and in Commerce soil incubated at 10°C. Degradation was rapid in these incubations, with oxamyl accounting for <50% AR by day 7 in both 20 °C Commerce soil and Gross Umstadt soil and by day 21 in 10 °C Commerce soil. By the end of these incubations, oxamyl accounted for less than detectable levels in 20°C Commerce soil (day 32 onwards), 0.2% AR in Gross Umstadt soil (day 31) and less than detectable levels in 10 °C Commerce soil (day 179).

Degradation of oxamyl was less extensive in Drummer #6 soil (silty clay loam, USA), incubated at 20 °C, with oxamyl accounting for 51.2% AR by the end of this incubation (day 123). Mineralisation in Drummer #6 soil was also substantially lower than in the other incubations but was still significant (25.6% AR maximum). It appears that Drummer #6 soil may have had a stressed microbial population due to its strongly acidic nature (pH of 4.8) and that, therefore, it may not have been representative of a typical European agricultural soil. The acidic nature of Drummer #6 soil may also have inhibited degradation of oxamyl by hydrolysis to IN-A2213, which occurs faster under alkaline conditions.

The predominant degradation product in all cases was carbon dioxide, which increased throughout each incubation, reaching final levels of 73.1% AR (Commerce, 20 °C – day 60), 76.1% AR (Gross Umstadt, 20 °C – day 31), 25.6% AR (Drummer #6, 20 °C – day 123) and 81.0% AR (Commerce, 10 °C – day 179). Unextracted soil radioactivity also increased during the incubations, reaching maximum values of 26.4% AR (Commerce, 20 °C – day 32), 26.4% AR (Gross Umstadt, 20 °C – day 21), 20.7% AR (Drummer #6, 20 °C – day 123) and 17.7% AR (Commerce, 10 °C – day 179).

The main degradation products extracted from soil were IN-A2213 and IN-D2708. IN-A2213 was detected in all soils, while IN-D2708 was observed in the Commerce and Gross Umstadt soils only. No other significant degradation products were identified apart from $^{14}CO2$, IN- A2213 and IN-D2708.

Maximum detected levels of IN-A2213 were 51.0% AR (Commerce, 20 °C – day 7), 24.9% AR (Gross Umstadt, 20 °C – day 2), 7.6% AR (Drummer #6, 20 °C – day 60) and 33.8% AR (Commerce, 10 °C – day 32). By the end of the incubations, IN-A2213 had declined to final detected levels of 3.4% AR (Commerce, 20 °C – day 32), 0.2% AR (Gross Umstadt, 20 °C – day 31), 6.0% AR (Drummer #6, 20 °C – day 123) and 0.5% AR (Commerce, 10 °C – day179).

IN-D2708 became a major soil degradation product in the Commerce and Gross Umstadt soils as IN-A2213 declined, reaching maximum levels of 25.7% AR (Commerce, 20 °C – day 11), 34.7% AR (Gross Umstadt, 20 °C – day 10) and 39.5% AR (Commerce, 10 °C – day 90). By the end of these incubations, IN-D2708 had declined to less than detectable levels in 20 °C Commerce soil, 0.3% AR (Gross Umstadt, 20 °C) and 20.1% AR (Commerce, 10 °C). It was surmised that IN-D2708 was not seen in Drummer #6 soil due to the slow rate of oxamyl degradation in this soil, which resulted in lower levels of IN-A2213 (the precursor for IN- D2708 formation).

With regard to the effect of temperature on the incubations, although degradation of oxamyl in 10 °C Commerce soil was slower than in 20 °C Commerce soil (as expected), the route of degradation was the same in both cases. Overall, the study confirms the findings from Study 1 in this section (report no. DuPont-2958), indicating that the major degradation pathway for oxamyl during aerobic incubation in microbially viable agricultural topsoil is hydrolysis to form IN-A2213, which subsequently degrades to IN-D2708, with carbon dioxide and unextractable soil residues being the final products.

The aerobic degradation of the active substance study DuPont-2957, originally submitted under EU Rev8 Point IIA 7.1.1.2.1.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), and U.S. EPA 162-1 (1982). A review of this study indicates that it partially meets the current guideline (OECD 307); deviations include the Drummer#6 soil having soil properties outside of those recommended in the guideline. In particular, the Drummer#6 soil had a pH of 4.8, which is outside the guideline recommended range of 5.5–8.0. Microbial measurements of this soil at the start and termination of the incubation demonstrated that the soil did not meet the additional guideline criteria of a microbial biomass of at least 1% of the total organic carbon. It is believed that the extremely low pH of this Drummer#6 soil hindered its microbial health and rendered it a non-viable soil. As a result of the low microbial biomass of the Drummer#6 soil, the apparent half-life in this soil was much longer than in any other soil tested. Since this soils has been shown to not be microbially viable, results of the Drummer#6 soil have not been taken forward in any kinetic assessments or PEC modelling. However, reconduct is unlikely to yield a significantly different result because the two remaining soils used in this study provide an adequate data set for the rate of oxamyl degradation in aerobic soils when they are considered with the results from DuPont-2958, DuPont-39014, and AMR1851-90. Therefore, this study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
A review of this study indicates that it partially meets the current guideline (OECD 307); deviations include the Drummer#6 soil having soil properties outside of those recommended in the guideline. Since this soil has been shown to not be microbially viable, results of the Drummer#6 soil have not been taken forward in any kinetic assessments or PEC modelling.
The two remaining soils used in this study provide an adequate data set for the rate of oxamyl degradation in aerobic soils when they are considered with the results from other studies.

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.1.2.1.1/02**

| **Reference:** -- | **Report:** | Smyser, B.P., Mattson, S.L. (2000); Route of degradation of oxamyl in aerobic soil<br>**DuPont Report No.:** DuPont-2958<br>**Guidelines:** SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), U.S. EPA 162-1 (1982) |
|---|---|---|

**Summary:**

Oxamyl degraded with a first order $DT_{50}$ ($DT_{90}$) = 7.9 (26) days. Oxamyl oxime (IN-A2213) and DMOA (IN-D2708) metabolites were found at levels up to 15 and 30% of the applied radioactivity, respectively. Oxamyl oxime and DMOA degraded with $DT_{50}$ ($DT_{90}$) values of 1.8 (6 days) and 7.6 (25.4 days), respectively. No other metabolite was observed at >1% of the applied radioactivity. $^{14}$C-Carbon dioxide, found in the caustic trap, was the final and most significant degradate. Essentially all the applied radioactivity was converted to $^{14}$C-carbon dioxide (108% of applied radioactivity) by the end of the study.

**Materials and Methods:**

The rate of degradation of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410) in aerobic soil was studied in a field-fresh loam soil, from Nijmegen, The Netherlands. Soil samples were used within three months of collection from the field, and soil biomass determinations (made at the time of treatment and the end of the study) indicated the presence of a viable microbial population. Details of the soil characteristics are given in Table 25.

**Table 25 Characteristics of Nijmegen loam soil used to investigate the aerobic soil metabolism of oxamyl**

| **Characteristic** | **Soil** |
|---|---|
| Soil designation | Nijmegen |
| Origin | Nijmegen, The Netherlands |
| pH | 7.0 |
| % sand (2000-50 □m) | 45.2 |
| % silt (<50-2 □m) | 40.8 |
| % clay (<2 □m) | 14.0 |
| USDA texture | loam |
| Organic matter (%) | 2.4 |
| Organic carbon (%) | 1.4 |
| Cation exchange capacity (meq/100 g) | 10.1 |
| Moisture-holding capacity at 0 bar (%) | 33.3 |
| Initial microbial biomass (mg microbial C/100 g soil) | 18.71 |
| Final microbial biomass (mg microbial C/100 g soil) | 13.45 |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

Prior to incubation under study conditions, the soil was adjusted to 40% of its 0-bar moisture content by the addition of purified water, resulting in a soil moisture content of 13.3% (g water/100 g dry soil).
Following a pre-incubation period of seven days, samples of the moist soil (equivalent to 50 g dry soil) were treated with ~100 µg [1- $^{14}$C]-oxamyl. This treatment rate of 2 mg a.s./kg dry soil corresponds to an application rate of 3 kg a.s./ha (based on a soil incorporation depth of 10 cm and a default value of 1.5 g/cm3 for bulk dry soil density), which is 54.5% of the maximum recommended use rate for oxamyl (5.5 kg a.s./ha).
Sufficient samples were prepared so that replicates could be taken for analysis at each sampling timepoint. The treated soil samples were incubated for up to 59 days under aerobic conditions in the dark at 20 °C, with the moisture content being maintained at 40% of the 0- bar moisture-holding capacity throughout the incubation period (by the addition of purified water as necessary).
Duplicate treated soil samples were removed for analysis at intervals of 0, 1, 2, 3, 7, 10, 14, 21, 31 and 59 days after test substance application. Qualitative and quantitative determination of the distribution of [1- $^{14}$C]-oxamyl and its degradates in the soil extracts was undertaken using reversed phase high performance liquid chromatography (HPLC) with radiochemical detection, fraction collection and liquid scintillation analysis.
The limits of detection (LOD) for extract analysis, HPLC analysis and combustion analysis were respectively 0.20% applied radioactivity (0.004 mg oxamyl equivalents/kg soil), 0.39% applied radioactivity (0.0078 mg oxamyl equivalents/kg soil) and 0.03% applied radioactivity (0.0007 mg oxamyl equivalents/kg soil).

**Findings:**

The levels (expressed as a percentage of applied radioactivity) of parent compound and degradates recovered at each sampling interval in the study, together with mass balances, are shown in Table 26.
Total recoveries of applied radioactivity (mass balance values) ranged from 100.3% (day 14) to 130.3% (day 59). In each case, the mass balance was calculated as the sum of volatile radioactivity in the KOH traps ( $^{14}CO_2$), volatile radioactivity in the ethylene glycol trap, radioactivity extracted from the soil and unextracted radioactivity remaining in the soil.
There was very extensive mineralisation of the test substance during the study, as indicated by the increase in $^{14}CO_2$ throughout the incubation period, which reached a level of 74.3% AR (applied radioactivity) by day 31 and a level of 108.5% AR by day 59. Concomitant with this was an increase in unextracted soil radioactivity, which reached a plateau towards the end of the study (19.7% AR on day 31, 19.8% AR on day 59). [An increase in unextractable radioactivity is expected when a microorganism utilises an exogenous compound for its growth and maintenance.] Volatile radioactivity other than $^{14}CO_2$ was minimal, with a maximum level of 0.19% AR being found in the ethylene glycol trap by day 59.

**Table 26 Mass balance and distribution of applied radioactivity in aerobic Nijmegen loam soil after application of 2 mg/kg of [1-$^{14}$C]-oxamyl**

| DAA | Oxamyl | IN-A2213 | IN-D2708 | $^{14}CO^2$ | Volatiles | Others | NER | Total |
|---|---|---|---|---|---|---|---|---|
| 0 | 102.4 | 4.8 | - | - | - | 0.7 | 0.7 | 108.6 |
| 1 | 85.4 | 15.0 | 2.9 | 1.1 | 0.00 | 0.3 | 1.7 | 107.1 |
| 2 | 93.2 | 14.5 | 6.0 | 1.9 | 0.00 | 0.65 | 2.3 | 118.6 |
| 3 | 73.4 | 11.7 | 11.7 | 2.8 | 0.00 | 0.45 | 3.8 | 103.9 |
| 7 | 56.8 | 13.4 | 19.8 | 7.3 | 0.01 | 0.75 | 6.4 | 104.4 |
| 10 | 42.1 | 13.1 | 25.9 | 12.6 | 0.01 | 0.30 | 9.3 | 103.2 |
| 14 | 28.9 | 7.8 | 29.6 | 21.0 | 0.04 | 0.45 | 12.1 | 100.3 |
| 21 | 17.8 | 3.7 | 28.8 | 37.3 | 0.04 | - | 15.9 | 103.6 |
| 31 | 6.4 | 1.9 | 16.5 | 74.3 | 0.18 | - | 19.7 | 118.9 |
| 59 | <1.9 | <1.9 | <1.9 | 108.5 | 0.19 | - | 19.8 | 130.3 |

DAA = days after application, NER = non-extractable radioactivity

Day-59 extracts were not analysed by HPLC, since the extractable radioactivity at this timepoint accounted for only 1.9% of the applied radioactivity.
Soil-extractable radioactivity (comprising parent oxamyl, degradates IN-A2213 and IN-D2708 and other extractable radioactivity) declined from 107.9% AR on day 0 to 1.9% AR by day 59. Oxamyl itself declined rapidly from an initial level of 102.4% AR, accounting for <50% AR by day 10 and accounting for <1.9% AR by day 59. The incubation period for the study did not extend beyond 59 days because the degradation of oxamyl was essentially complete by this stage. The very good recovery of oxamyl on day 0 was considered to demonstrate test substance extractability and stability.

IN-A2213 (oxamyl oxime – the hydrolysis product of oxamyl) was the main soil degradation product initially. It reached a peak value of 15.0% AR on day 1 and then declined, accounting for 1.9% AR by day 31. IN-D2708, which was detected on day 1 at a level of 2.9% AR, subsequently became the main soil degradation product, reaching a maximum value of 29.6% AR on day 14. Levels of IN-2708 then decreased, such that it accounted for <1.9% AR by the end of the incubation period. Apart from $^{14}CO2$, IN-A2213, IN-D2708 and unextractable soil residues, no other degradation products were detected at levels exceeding 1% AR. The identities of IN-A2213 and IN-D2708 were confirmed by matching the retention time in samples to that of authentic standards using two separate HPLC analytical methods. Selected soil extracts were also directly fortified with IN-D2708 to confirm the retention time match.
The results of the fractionation of the unextracted soil radioactivity from the day-31 replicates (average of 19.7% AR) to determine the distribution of radioactivity in the fulvic acid, humic acid and humin fractions of the soil are given in Table 27.

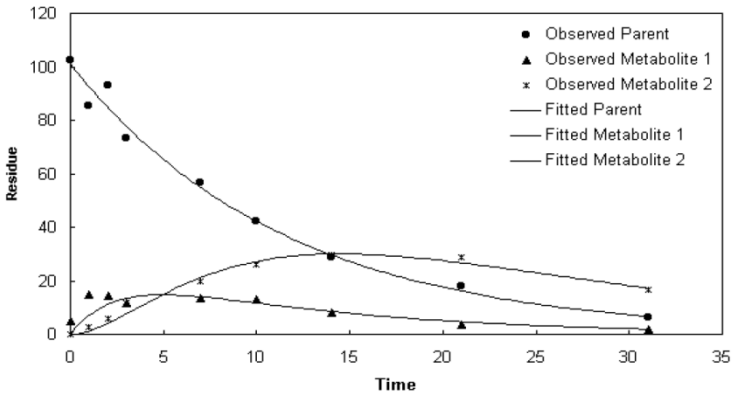
**Table 27 Distribution of unextracted soil radioactivity in soil organic matter at day 31**

| Soil fraction | % of applied radioactivity | % of non-extractable fraction |
|---|---|---|
| Fulvic acid | 6.6 | 33.4 |
| Humic acid | 6.1 | 31.2 |
| Humin | 2.2 | 11.4 |

Average of unextracted soil radioactivity in the day-31 replicates was 19.7% AR. The fulvic acid, humic acid and humin fractions from the post-extracted day-31 soil samples respectively contained 6.6%, 6.1% and 2.2% of applied radioactivity, showing that the soil- bound (unextracted) portion of radioactivity in these samples was associated with all three fractions of the soil organic matter.

Degradation rates for oxamyl, oxamyl oxime (IN-A2213) and DMOA (IN-D2708) were determined from the reported data using a nonlinear regression of conventional first-order kinetic equations. The software used for this fitting procedure was ModelManager, Version 1.1 (Cherwell Scientific Limited, 1999).
Table 26 contain values for both the average amount of oxamyl, oxamyl oxime, and DMOA (average of the two replicates, as %AR) remaining at each sampling point. The average %AR of each was plotted as a function of time and the best fit determined in ModelManager are depicted in Figure 6 .

**Figure 6 Soil degradation kinetics of oxamyl, oxamyl-oxime and DMOA**




A $DT_{50}$ (half-life) and $DT_{90}$ for oxamyl, oxamyl oxime, and DMOA were determined. These values are as follows:

| Analyte | $DT_{50}$ (days) | $DT_{90}$ (days) |
|---|---|---|
| **Oxamyl** | 7.9 | 26 |
| **Oxime** | 1.8 | 6 |
| **DMOA** | 7.6 | 25.4 |

**Conclusions:**

Following application at a nominal rate of 2 mg a.s./kg dry soil, oxamyl was degraded extensively at 20 °C, under aerobic conditions in the dark, in the Dutch loam soil (Nijmegen) used in this laboratory study. Degradation was rapid with oxamyl accounting for <50% AR by day 10 and <1.9% AR by the end of the incubation period (day 59). The ultimate, and principal, degradation product was carbon dioxide, which increased throughout the study, to reach a level of 74.3% AR by day 31 and 108.5% AR by day 59. Unextracted soil radioactivity also increased during the incubation (reaching a plateau of approximately 20% AR towards the end of the study), and was shown to be primarily associated with the fulvic and humic acid fractions of the soil in approximately equal proportions and to a lesser extent with the humin fraction (based on fractionation of post-extracted day-31 soil samples).

The main soil degradation products were IN-A2213 and IN-D2708, which were detected at maximum respective levels of 15.0% AR (day 1) and 29.6% AR (day 14). Both these substances had declined to <1.9% AR by the end of the study. No other metabolite was observed at >1% AR.

Overall, the study shows that oxamyl was readily utilised by soil microorganisms to form carbon dioxide, via the intermediate degradation products IN-A2213 and IN-D2708. On the basis of this study, the primary degradation pathway for oxamyl in aerobic soil appears to be hydrolysis to form IN-A2213, which subsequently degrades to IN-D2708, with carbon dioxide and unextractable soil residues being the final products.

The aerobic degradation of the active substance study DuPont-2958, originally submitted under EU Rev8 Point IIA 7.1.1.2.1.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines SETAC Europe (1995), OECD Guidelines for the Testing of Chemicals: Aerobic and Anaerobic Transformation in Soil (Draft, October 1999), and U.S. EPA 162-1 (1982). A review of this study indicates it fully meets the current guideline (OECD 307). Therefore, this study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl
The soil used in this study provide an adequate data set for the rate of oxamyl degradation in aerobic soil when it was considered with the results from other studies.

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.1.2.1.1/03**

| | | |
|---|---|---|
| **Reference:** -- | **Report:** | Spare, W.C. (1991); Anaerobic soil metabolism of [1-$^{14}$C]oxamyl in Madera, California soil<br><br>**DuPont Report No.:** AMR 1851-90<br><br>**Guidelines:** U.S. EPA 162-2 (1982) |

**Summary:**

The metabolism and degradation were studied in Madera, California soil to provide data on the rate of degradation and the pattern of metabolism under aerobic and aneorobic conditions. The application rate is 9.5 ppm. The test vessels were incubated at 25 °C in the dark under aerobic conditions for one half-life. The rate of degradation of oxamyl in aerobic soil was calculated, using least square regression analysis, to be 11 days with a correlation coefficient of 0.997 using all data generated through 51 days. The major degradation products under aerobic conditions were identified as oxime and DMOA.The half-life of oxamyl in Madera, California soil at 25 °C in the dark was 6 days under anaerobic conditions

**Materials and Methods:**

The rate of degradation of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410) in aerobic soil were studied in a US sandy clay loam soil (from Madera, California) under aerobic conditions followed by anaerobic conditions. The determination of degradation rates for oxamyl and metabolites IN-A2213 and IN-D2708 during the initial aerobic portion of the study is described here.

The aerobic portion of the study involved incubation of Madera soil (pH 7.7) at 25°C, with soil moisture content being maintained at 70-75% of field capacity. A nominal treatment rate of 9.5 mg a.s./kg dry soil was used, corresponding to an application rate of 11.875 kg a.s./ha (based on a soil incorporation depth of 10 cm and the reported bulk soil density of 1.25 g/cm3).

The experimental procedures for the aerobic portion of the study have already been described in section B.8.1.1.1 and details of the soil characteristics have been given previously in Table 6.

Degradation rates for oxamyl, IN-A2213 and IN-D2708 were determined on the basis of the amounts of these substances extracted from the soil at each sampling interval. The figures for the amounts of the substances detected during the study (expressed as a percentage of applied radioactivity) have been given in Table 7.

The original study only reported a degradation rate for oxamyl, which was obtained assuming first order degradation (linear regression of the natural log (ln) of the percentage of oxamyl remaining against time, least-squares fit). However, in order to obtain degradation rates for IN-A2213 and IN-D2708 as well, the notifier re-estimated first order DT50 and DT90 values from the residue data in the same manner as described previously for report nos. DuPont-2957 and DuPont-2958. First order degradation rate constants for oxamyl, IN-A2213 and IN- D2708 were obtained by using ModelManager® (version 1.1) to perform non-linear regressions of experimental residue versus time data to integrated equations obtained from differential rate equations that assumed first order degradation for oxamyl and both first order formation and degradation for metabolites IN-A2213 and IN-D2708, with parent and metabolites being considered in series. It was assumed that IN-A2213 and IN-D2708 formed exclusively as a result of a fraction of oxamyl degrading directly to IN-A2213 and a fraction of IN-A2213 degrading directly to IN-D2708.

**Findings:**

The outputs from the various degradation rate calculations for the aerobic portion of the study are summarised in Table **28.**

**Table 28 $DT_{50}$ and $DT_{90}$ values for oxamyl, IN-A2213 and IN-D2708 in Madera sandy clay loam soil incubated aerobically at 25 °C, with soil moisture content at 70-75% of field capacity**

| Substance | k (days$^{-1}$) | DT50 (DAY S) | DT90 (DAY S) | $r^2$ | Method |
|---|---|---|---|---|---|
| Original calculation in study report | | | | | |
| Oxamyl | $k_p$ = 0.0645 | 11 | 36 | 0.997 | First order degradation – linear regression of the natural log of the percentage of oxamyl remaining against time, least-squares fit. |
| Notifier's re-estimation | | | | | |
| Oxamyl | $k_p$ = 0.0602 | 11.5 | 38.2 | 0.987 | Non-linear regression of integrated rate equations based on simple first order kinetics, with parent and two metabolites considered in series. |
| IN-A2213 | $k_1$ = 0.108 | 6.4 | 21.3 | | |
| IN-D2708 | $k_2$ = 0.139 | 5.0 | 16.5 | | |

$k_p$ is the first order degradation rate constant for oxamyl, $k_1$ is the first order degradation rate constant for IN-2213 and $k_2$ is the first order degradation rate constant for IN-D2708.

In the non-linear regression technique used by the notifier, C1 (the fraction of oxamyl forming IN-A2213) and C2 (the fraction of IN-A2213 forming IN-D2708) were both fixed to 1.

The notifier's re-estimation provided a very good fit to the data (r2 = 0.987) and showed that oxamyl, IN-A2213 and IN-D2708 all degraded rapidly in the aerobic portion of the study, as indicated by respective DT50 values of 11.5, 6.4 and 5.0 days (and corresponding DT90 values of 38.2, 21.3 and 16.5 days). These results are consistent with the previously reported degradation rates obtained from the other laboratory aerobic soil metabolism studies involving application of oxamyl (report nos. DuPont-2957 and DuPont-2958) for incubations in microbially viable soils.

The oxamyl degradation rate estimated by the notifier (DT50 = 11.5 days) is very similar to the aerobic degradation rate reported in the original study (DT50 = 11 days), where oxamyl was considered on its own. Fitting the residue data for oxamyl in conjunction with the residue data for IN-A2213 and IN-D2708 only had a marginal effect on the degradation rate obtained by considering oxamyl in isolation.

**Conclusions:**

Following treatment at a rate of 9.5 mg a.s./kg dry soil, corresponding to an application rate of 11.875 kg a.s./ha, oxamyl degraded rapidly in Madera sandy clay loam soil (pH 7.7) incubated under laboratory aerobic conditions at 25 °C, with soil moisture content being maintained at 70-75% of field capacity. First order DT50 and DT90 values for oxamyl were estimated by the notifier to be 11.5 and 38.2 days respectively. The first order degradation rate constants used to calculate the DT50 and DT90 values were obtained by non-linear regression of integrated equations resulting from differential rate equations that assumed first order degradation for oxamyl and both first order formation and degradation for metabolites IN-A2213 and IN- D2708, with parent and metabolites being considered in series.
The non-linear regression of the experimental residue versus time data also provided first order degradation rate constants for the metabolites IN-A2213 and IN-D2708, showing that these metabolites degraded quickly under the incubation conditions. First order DT50 values for these substances were estimated to be 6.4 and 5.0 days respectively (with corresponding DT90 values of 21.3 and 16.5 days).
The oxamyl DT50 value of 11.5 days estimated by the notifier is in accord with the aerobic DT50 value of 11 days reported in the original study, where degradation of oxamyl was considered in isolation (first order degradation – linear regression of the natural log of the percentage of oxamyl remaining against time, least-squares fit).
The DT50 and DT90 values obtained for oxamyl, IN-A2213 and IN-D2708 from the aerobic portion of the study are consistent with the degradation rate results from the other laboratory aerobic soil metabolism studies for incubations in microbially viable soils.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl
The soil used in this study provide an adequate data set for the rate of oxamyl degradation in aerobic soil when it was considered with the results from other studies.

**Aerobic degradation (studies involving metabolite application)**

Laboratory aerobic degradation rates for metabolites IN-A2213 and IN-D2708 were derived from the results of studies involving application of oxamyl, as previously described. In addition, aerobic rates of degradation for the metabolites IN-D2708 and IN-N0079 were determined experimentally in two separate studies, each of which involved fortification of three soil types under laboratory conditions with the appropriate metabolite.

The aerobic part of degradation study AMR 1851-90, originally submitted under EU Rev8 Point IIA 7.1.1.1.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline U.S. EPA 162-2 (1982). A review of this study indicates that the aerobic soil experiment in this study meets the current guideline (OECD 307). The only minor deviation is a mass balance slightly below the guideline recommended 90-100%AR at the last 2 sampling points. This deviation does not impact the outcome of the study since the degradation pathway of oxamyl was well established before the final timepoints. Losses are attributed to slight ineffieciencies in trapping the large amounts of $^{14}C0_2$ generated and are not losses of parent oxamyl or metabolites. Therefore reconduct is unlikely to yield a significantly different results and the study is still suitable for deriving rate data. Therefore, this study is relied upon.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.1.1/04**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.1.2.1.1/01** | **Report:** | Clark, B. (2015); Aerobic rate of degradation of [$^{14}$C]-DPX-D1410 (oxamyl) in four acidic soils<br>**DuPont Report No.:** DuPont-39014<br>**Guidelines:** OECD 307, OPPTS 835.4100, SETAC (1995)<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** 80581<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

A full description of this study can be found in Point B.8.1.1.1 in this document.

The rate of degradation of oxamyl was examined in four acidic soils in the laboratory under aerobic conditions. The test soils were treated with radiolabeled oxamyl at a concentration of 5.0 µg a.s./g soil. Samples were incubated in darkness at 20 ± 2°C with a soil moisture of 100% of maximum water holding capacity (0.1 bar moisture, pF2). The major transformation products detected were IN-A2213 and IN-D2708, accounting for a maximum of 13.5 and 78.0 AR%, respectively, after 91 days of incubation.
Oxamyl degraded at 20°C in all test soils with $DT_{50}$ values ranging from 1.6 to 12.1 days and $DT_{90}$ values ranging from 5.3 to 40.2 days. The relative rate of degradation of oxamyl by the four soils was related to the microbial activity, pH, and organic matter content of the soils.

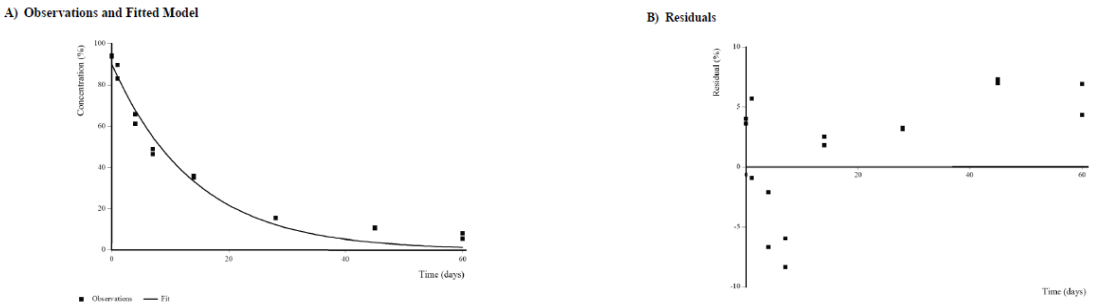
Four soils were chosen to represent conditions of use; Speyer loamy sand (Hanhofen, Germany), Tama light clay (Stark County, Illinois), LRA-D sandy loam (Derbyshire, England), and Goch sandy loam (Northrhine-Westphalia, Germany). Prior to use, each test soil was homogenized and passed through a 2-mm mesh sieve. Soils were then stored at approximately 4°C in the dark in closed bags when not in use. Pre-incubation was performed to acclimate soil samples to the test temperature and achieve aerobic conditions prior to study initiation. Samples were pre-incubated for 9 days for the Definitive Test sample series before the test substance was applied.

TRANSFORMATION OF PARENT COMPOUND

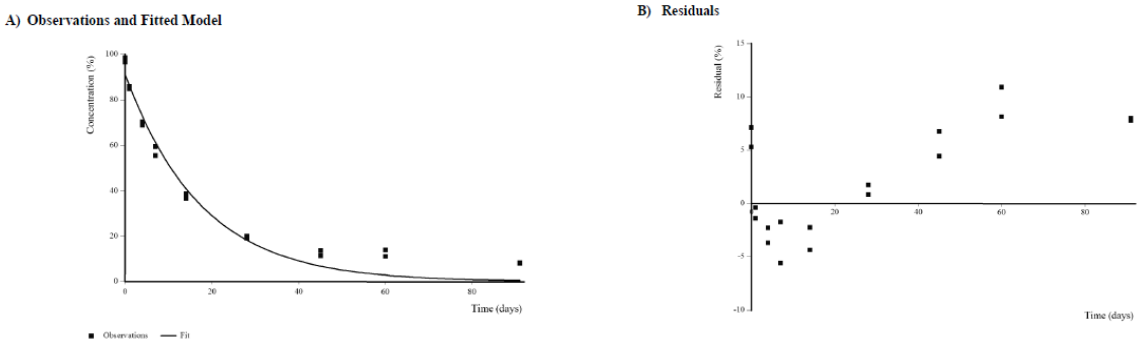
Plots of the observed and fitted data for oxamyl using the single first-order (SFO) are presented below. It is noted that due a rapid degradation observed in the Goch soil, robust kinetic fits could not be obtained ($\chi^2$=26.7). Since nearly 90% of the oxamyl was degraded between day 1 and day 4 sampling points, the $DT_{50}$ for this soil can be conservatively estimated as 4 days.

Figure 7 Single First Order regression analysis of the degradation data in four soil treated with oxamyl

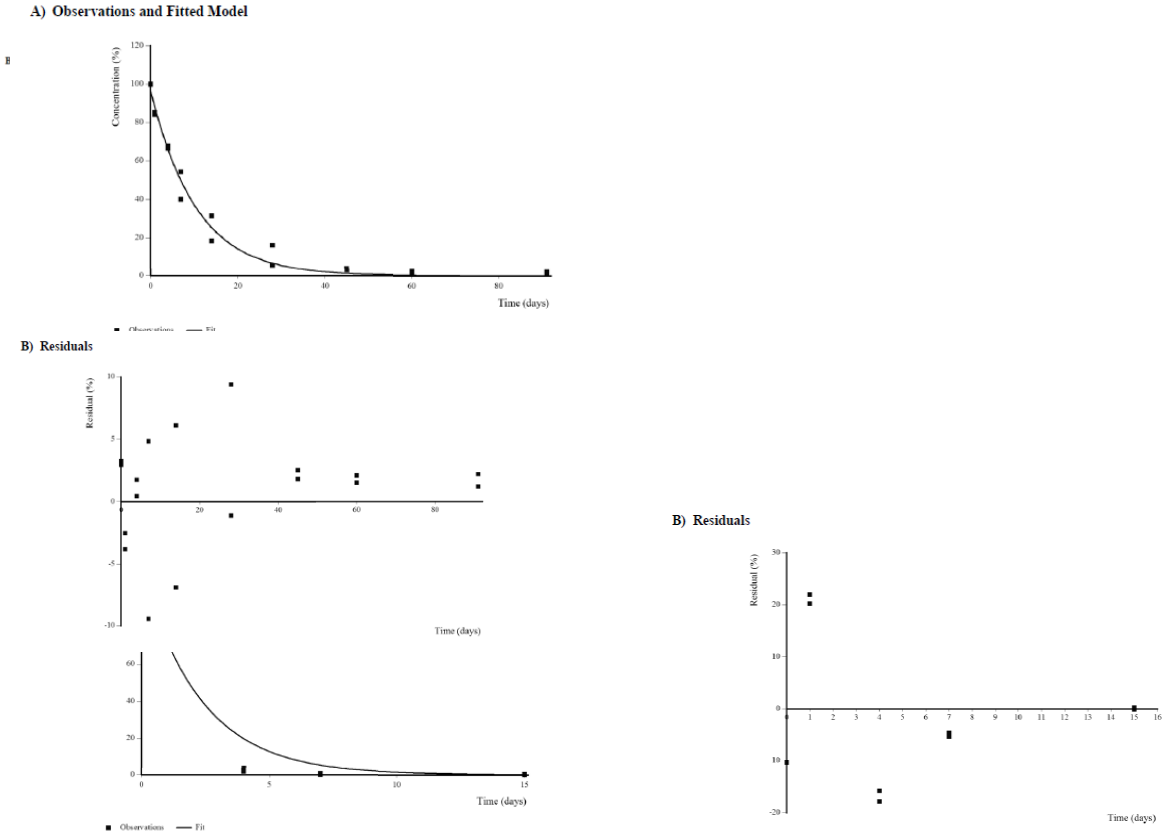
1) Soil: Tama



2) Soil: LRA-D




3) Soil: Speyer




Levels of oxamyl in the soil declined continuously over a period of 91 days incubation. The $DT_{50}$ and $DT_{90}$ values for oxamyl ranged from 1.6 to 12.1 days and 5.3 to 40.2 days, respectively as shown in Table 29.

**Table 29 Summary of degradation parameters as persistence triggers for oxamyl**

| Study | Soil/Condition | $r^2$ | $\chi^2$ | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|---|---|
| DuPont-39014 | Tama (Light Clay) 20°C | 0.9810 | 8.59 | 9.8 | 32.4 | SFO |
| | LRA-D (Sandy Loam) 20°C | 0.9824 | 9.66 | 12.1 | 40.2 | SFO |
| | Speyer (Loamy Sand) 20°C | 0.9863 | 5.17 | 7.2 | 24.0 | SFO |
| | [a]Goch (Sandy Loam) 20°C | 0.9261 | 26.7 | 1.6 | 5.3 | SFO |

[a] Due a rapid degradation, enough data points were not available to produce robust kinetic fits.

The kinetics presented in this report are superseded by the FOCUS kinetic results derived in the modelling position paper DuPont-41859 EU (summarised in Point 0 of this document).

Two major degradation products were formed in soil. IN-A2213 accounted for 0.2% of applied radioactivity for [1-$^{14}$C]oxamyl after 91 days incubation. IN-D2708 accounted for 36.9% of applied radioactivity for [1-$^{14}$C]oxamyl after 91 days incubation.

**CONCLUSION**

Oxamyl degraded at 20°C in all test soils with $DT_{50}$ values ranging from 1.6 to 12.1 days and $DT_{90}$ values ranging from 5.3 to 40.2 days. The relative rate of degradation of oxamyl by the four soils was related to the microbial activity and organic matter content of the soils.
The results demonstrate that oxamyl is rapidly transformed in acidic soils over time and would extensively degrade to $CO_2$ and non-extractable residues.

(Clark, B., 2014)

**RMS comments and conclusion**

Study was submitted for the first time in this submission. The study was conducted in accordance with OECD 307 and SETAC (1995) guidelines. The study was conducted according to Good Laboratory Practice. The study is considered acceptable.
The $DT_{50}$ and $DT_{90}$ values for oxamyl ranged from 1.6 to 12.1 days and 5.3 to 40.2 days, respectively
The soil used in this study provide an adequate data set for the rate of oxamyl degradation in aerobic soil when it was considered with the results from other studies.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.1.1/05**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.1.2.1.1/02** | **Report:** | Ghafoor, A., Zillgens, B. (2015); Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) from laboratory soil degradation studies<br>**DuPont Report No.:** DuPont-41859 EU<br>**Guidelines:** Not applicable<br>**Deviations:** Not applicable<br>**Testing Facility:** Dr. Knoell Consult GmbH, Mannheim, Germany<br>**Testing Facility Report No.:** DuPont-41859 EU<br>**GLP:** No<br>**Certifying Authority:** Not applicable |

**Executive Summary**

A summary of this modelling position paper can be found in the Oxamyl EU Renewal Dossier, Document M-CP, Section 9 (DuPont-40953 EU and DuPont-42129 EU). Four aerobic soil degradation studies in nine soils for oxamyl under laboratory conditions (DuPont-2957, AMR 1851-90, DuPont-2958, and DuPont-39014), one aerobic degradation study for metabolite IN-D2708 (DMOA) in three soils under laboratory conditions (DuPont-2675), one aerobic degradation study for metabolite IN-N0079 (DMCF) in three soils under laboratory conditions (DuPont-2674), and one photodegradation study for oxamyl and its metabolite IN-N0079 (DMCF) in one soil under laboratory conditions (DuPont-31501) were carried out. Since the studies do not contain a kinetic evaluation of the data according to recent FOCUS recommendations (FOCUS, 2006, 2011), residue data of these studies were re-evaluated to derive persistence and modelling endpoints for oxamyl and its metabolites IN-A2213, IN-D2708, and IN-N0079 under aerobic soil conditions, and for oxamyl and its photolytic metabolite IN-N0079 under irradiated conditions (photodegradation). The results of the kinetic evaluations of persistence endpoints of the active substance are summarised below. This was done for the sake of clarity and easier reading. The persistence and modelling endpoints, derived from laboratory (photo-) degradation studies and chosen according to FOCUS (2006, 2011) guidelines, are summarised in Table below.

**Table 29a Summary of degradation parameters as persistence triggers for oxamyl**

| Study | Soil/Condition | $DegT_{50}$ (days) | $DegT_{90}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|
| DuPont-2957 | Commerce 20°C | 2.8 | 9.3 | 7.8 | SFO |
| | Commerce 10°C | 15.8 | 52.3 | 4.4 | SFO |
| | Gross-Umstadt 20°C | 3.7 | 13.9 | 4.4 | DFOP |
| AMR 1851-90 | Madera 25°C | 11.1 | 36.8 | 5.5 | SFO |
| DuPont-2958 | Nijmegen 20°C | 7.8 | 25.8 | 6.6 | SFO |
| DuPont-39014 | Goch 597 20°C | 0.6 | 2.0 | 0.7 | SFO |
| | LRA-D 588 20°C | 9.7 | 79.5 | 2.8 | DFOP |
| | Speyer 582 20°C | 7.2 | 24.0 | 5.2 | SFO |
| | Tama 583 20°C | 7.8 | 48.5 | 3.3 | FOMC |

**Table 29 Summary of degradation parameters as persistence triggers for oxamyl degradation in light (photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|
| DuPont-31501 | Tama 20°C | 4.6 | 15.2 | 4.5 | SFO |

Modelling endpoints derived from the kinetic analyses were corrected for soil moisture content at field capacity (pF2.0) and temperature (20°C), if necessary, to be used in environmental fate modelling tools.
Moisture correction was carried out by multiplying the obtained SFO $DegT_{50}$ values by a moisture correction factor, which is espressed as follows:

$$\left(\frac{\theta}{\theta ref}\right)^{B}$$

where:
θ = actual soil moisture
$\theta_{REF}$= gravimetric water content at pF2
B = 0.7

The actual soil moisture was calculated from the reference moisture holding capacity (MHC) typical for the soil textural class ("gravimetric water content at MWHC") multiplied by soil moisture at which the study had been conducted (% MWHC). The gravimetric water content at MWHC and the gravimetric water content at pF2, based upon the textural class, were taken from the FOCUS groundwater guidance (FOCUS, 2000), if it was not reported in the study report.
If the moisture correction factor was > 1 due to the actual soil moisture being greater than the gravimetric water content at pF2 no adjustment was made, i.e. the moisture correction factor was set to 1.

Temperature correction was carried out using a $Q_{10}$ value of 2.58.

$$f_{temp} = Q_{10}^{\frac{Tact-Tref}{10}}$$

for: Tact > 0°C and Tref = 20°C
where: $f_{temp}$ = temperature correction factor
$Q_{10}$= 2.58
Tact = actual temperature
Tref = reference temperature

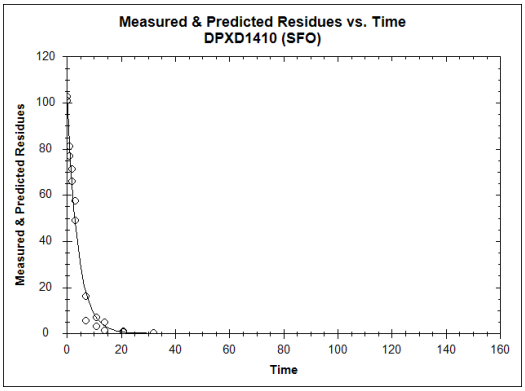
**Summary of degradation parameters as modelling endpoints for oxamyl**

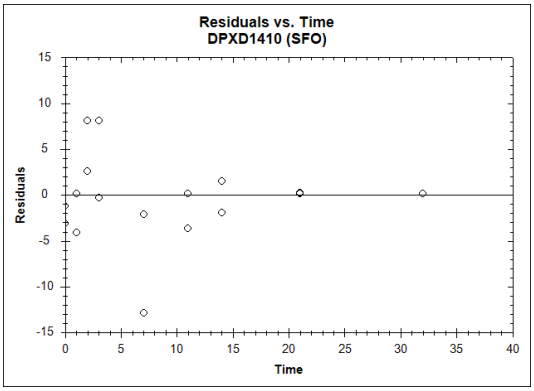
| Study | Soil/Condition | $DegT_{50}$ at 20 °C (days) | Moisture Content (w/w %) | | | $DegT_{50}$ at 20 °C & 10kPa (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2957 | Commerce 20°C | 2.8 | 33.3 | 13.3 | 26 | 1.8 | SFO |
| | Commerce 10°C | 6.1 | 33.3 | 13.3 | 26 | 3.8 | SFO |
| | Gross-Umstadt 20°C | 4.0 | 50 | 20 | 26 | 3.3 | SFO |
| AMR 1851-90 | Madera 25°C | 17.8 | 15.4 | 11.6 | 22 | 11.4 | SFO |
| DuPont-2958 | Nijmegen 20°C | 7.8 | 33.3 | 13.3 | 25 | 5.0 | SFO |
| DuPont-39014 | Goch 597 20°C | 0.6 | - | pF2 | - | 0.6 | SFO |
| | LRA-D 588 20°C | 19.4 | - | pF2 | - | 19.4 | FOMC $DegT_{90}$/3.32 |
| | Speyer 582 20°C | 7.2 | - | pF2 | - | 7.2 | SFO |
| | Tama 583 20°C | 14.3 | - | pF2 | - | 14.3 | FOMC $DegT_{90}$/3.32 |

The kinetics fittings for degradation of oxamyl for all nine soils were derived from the respective best-fit model and are presented in figure below

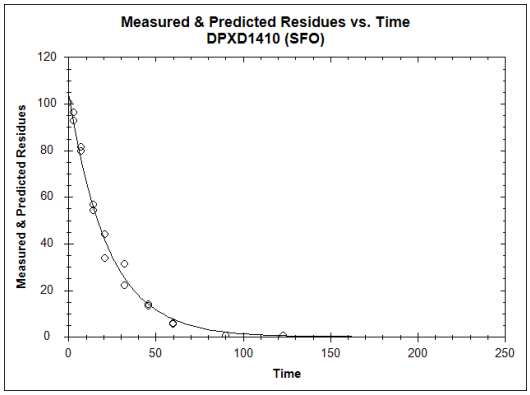
**Figure: The kinetics fittings for degradation of oxamyl in soil**

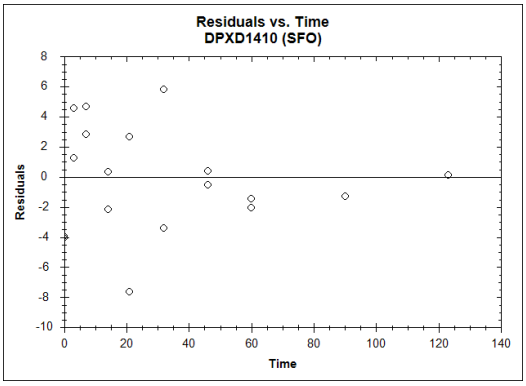
*Commerce soil at 20ºC*



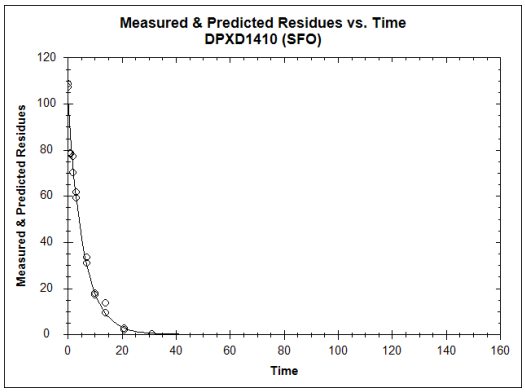



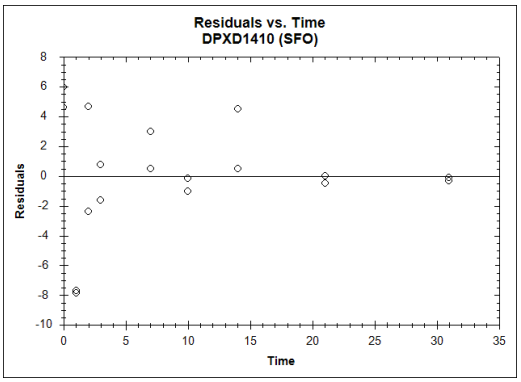

*Commerce soil at 10ºC*



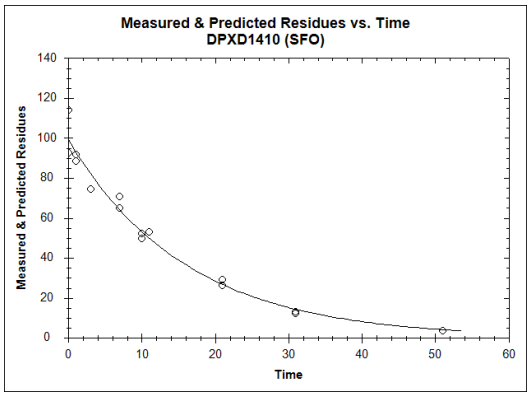



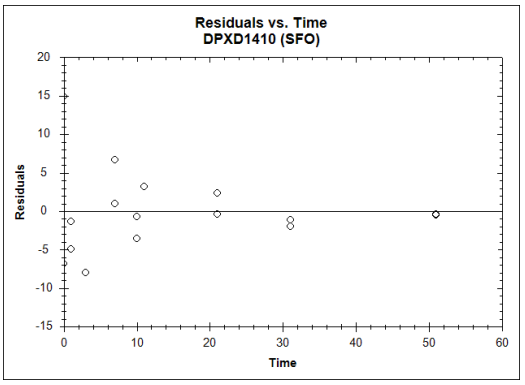

*Gross-Umstadt soil at 20ºC*



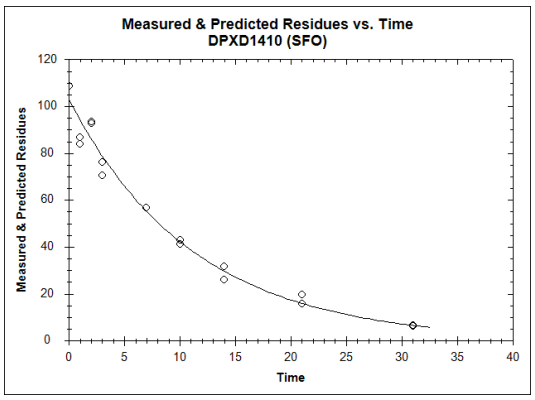



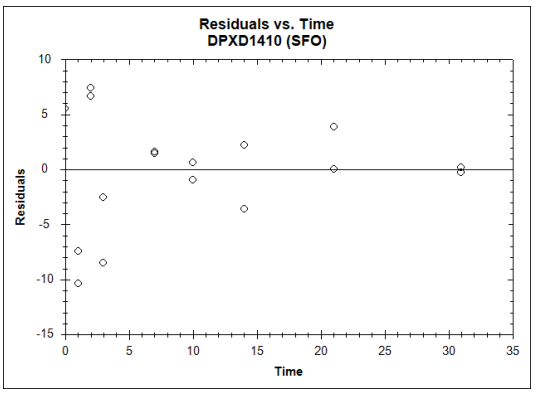

*Madera soil at 25°C*



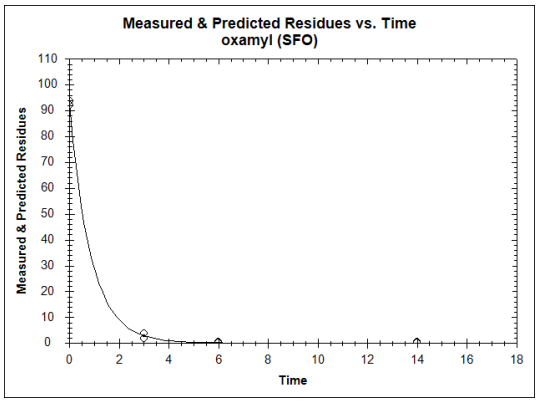



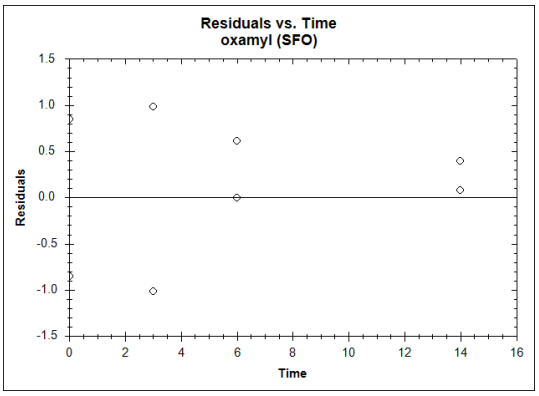

*Nijmegen soil at 20°C*



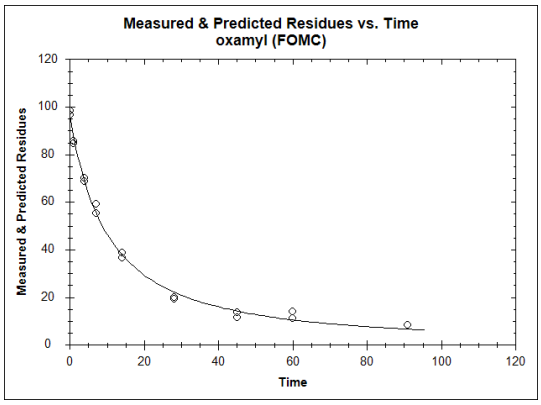



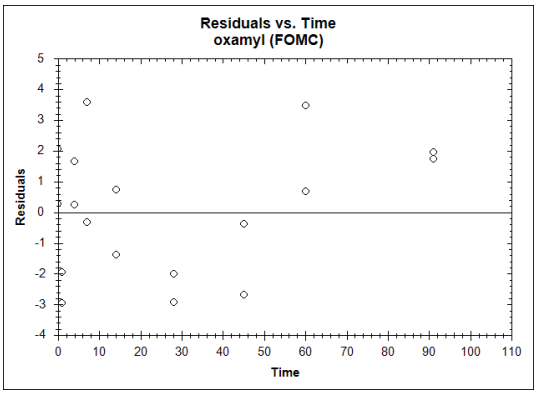

*Goch-597 soil at 20°C*



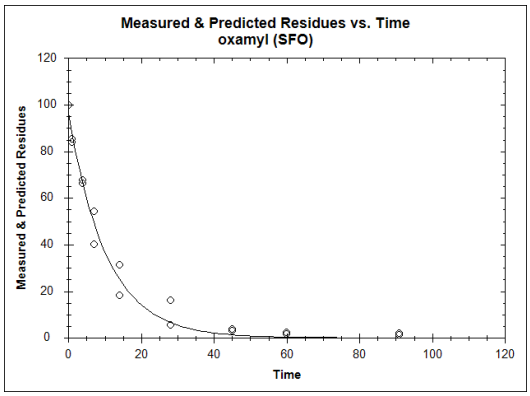



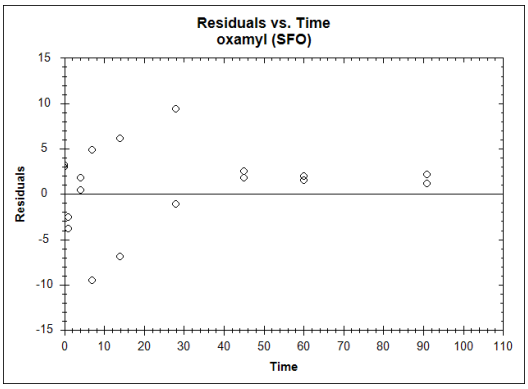

*LRA-D-588 soil at 20°C*



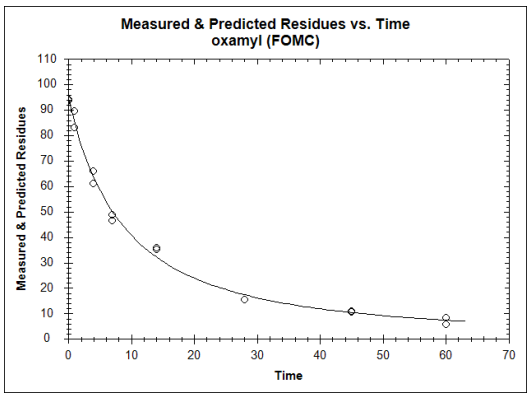



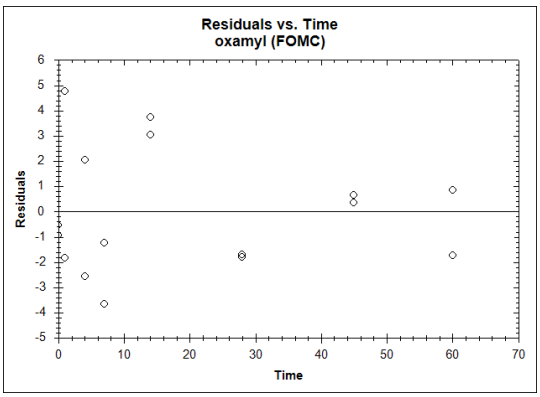

*Speyer 582 soil at 20ºC*




*Tama 583 soil at 20ºC*




Endpoints derived for the metabolites can be found under Point B.8.1.2.1.2 below.

**RMS comments and conclusion**

This study is considered corrected to harmonize the derivation of degradation parameters from soil metabolism. Residue data of aerobic soil degradation studies in nine soils for oxamyl under laboratory conditions (DuPont-2957, AMR 1851-90, DuPont-2958, and DuPont-39014) and one photodegradation study for oxamyl in one soil under laboratory conditions (DuPont-31501) were re-evaluated to derive persistence and modelling endpoints for oxamyl under aerobic soil conditions and under irradiated conditions (photodegradation).
The kinetic assessments conducted are in full compliance with the FOCUS kinetics guidance and the imput parameters can be used for ground water and surface water risk assessment.

*B.8.1.2.1.2 Aerobic degradation of metabolites, breakdown and reaction products*

RMS comments and conclusion

zRMS precise that all kinetic evaluations performed in studies B.8.1.2.1.2/01 and 02 are superseded by the FOCUS kinetic results derived in study summarised in Points B.8.1.2.1.2/03

**B.8.1.2.1.2/01**

| **Reference:** -- | **Report:** | Swain, R.S., Anderson, J. J. (2000); Rates of degradation of oxamyl metabolite [$^{14}$C](dimethylamino) oxoacetic acid (IN-D2708) in three aerobic soils<br>**DuPont Report No.:** DuPont-2675<br>**Guidelines:** SETAC Europe (1995) |
|---|---|---|

1. Test material: [$^{14}$C]IN-D2708
   Lot/Batch #: HOTC 549
   Purity: Radiochemical purity - >95%

**Materials and Methods:**

The aerobic rate of degradation of [ $^{14}$C]-IN-D2708 ([1,2- $^{14}$C]-IN-D2708, specific activity 49.53 µCi/mg, radiochemical purity >95%) was investigated in three field-fresh soils at 20 °C. The soils employed were the same ones that were used in the main laboratory degradation rate study on oxamyl (report no. DuPont-2957), i.e. 'Commerce' (silt loam, USA), 'Gross Umstadt' (silt loam, Germany) and 'Drummer #6' (silty clay loam, USA).

Soil samples were used within three months of collection from the field, and were stored at 5°C and sieved (2 mm) before use. Details of the soil characteristics are given in Table 30. [The figures in Table 30are the same as those previously given for these soils in Table 4, apart from the initial and final microbial biomass measurements.]

Prior to incubation under study conditions, purified water was added to the Commerce and Drummer #6 soils, with Commerce being adjusted to 40% of its 0-bar moisture content (resulting in 13.3 g water/100 g dry soil) and Drummer #6 being adjusted to 47% of its 0-bar moisture content (resulting in 23.2 g water/100 g dry soil). Gross Umstadt soil was used as received, with a moisture content of 44% of its 0-bar moisture-holding capacity (corresponding to 22 g water/100 g dry soil).

**Table 30 Characteristics of Commerce, Gross Umstadt and Drummer #6 soils used to investigate the aerobic soil degradation rate of IN-D2708**

| **Parameter** | **Commerce** | **Gross Umstadt** | **Drummer #6** |
|---|---|---|---|
| Origin | Greenville, MS, USA | Gross Umstadt, Germany | Rochelle, IL, USA |
| USDA texture | silt loam | silt loam | silty clay loam |
| % sand | 32.8 | 5.6 | 8.4 |
| % silt | 56.4 | 77.2 | 60.8 |
| % clay | 10.8 | 17.2 | 30.8 |
| pH | 7.0 | 7.8 | 4.8 |
| Organic matter (%) | 0.4 | 2.1 | 4.4 |
| Organic carbon (%) | 0.2 | 1.2 | 2.6 |
| Cation exchange capacity (meq/100 g) | 6.7 | 9.6 | 26.3 |
| Bulk density (g/cm$^3$) | 1.23 | 0.93 | 1.17 |
| 0-bar moisture (%) | 33.3 | 50.0 | 49.4 |
| Initial biomass (mg microbial C/100 g soil) | 2.37 | 32.52 | -6.86* |
| Final biomass (mg microbial C/100 g soil) | 3.84 | 37.09 | 11.11 |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

* The negative initial biomass value for Drummer #6 soil is considered erroneous.

Following a pre-incubation period of 27 days (at 20 °C), samples of the moist soils (equivalent to 10 g dry soil) were treated with ~10 µg [ $^{14}$C]-IN-D2708. This treatment rate of 1 mg/kg dry soil is in line with concentrations of IN-D2708 that would likely develop in these soils following an application of 5.5 kg oxamyl/ha, and is estimated to be less than half of the maximum possible concentration of IN-D2708 that could develop in these soils. [Assuming a 1:1 molar relationship between parent and IN-D2708 and 100% conversion, an application of 5.5 kg oxamyl/ha would give IN-D2708 concentrations of 2.4 mg/kg soil (Commerce), 3.2 mg/kg soil (Gross Umstadt) and 2.5 mg/kg soil (Drummer #6); based on a soil incorporation depth of 10 cm, respective reported bulk soil densities of 1.23 g/cm3, 0.93 g/cm3 and 1.17 g/cm3 and molecular weights (g/mole) of 219.26 for oxamyl and 117.1 for IN-D2708.]

Sufficient samples were prepared so that replicates could be taken for analysis at each sampling timepoint. The treated soil samples were incubated under aerobic conditions in the dark at 20 ±1 °C (for up to 45 days), with soil moisture contents being maintained at 40-50% of 0-bar moisture-holding capacity throughout the incubation periods (by the addition of purified water as necessary). Humidified air was drawn over the soil samples by vacuum during the incubations, with the effluent air from each treated sample being passed through caustic traps for acidic volatiles (1N KOH and 0.1N KOH with phenolphthalein indicator) before exiting the experimental apparatus.

Duplicate treated soil samples were removed for analysis at each sampling timepoint. The sampling intervals (days after test substance application) were 0, 3, 7, 15, 22 and 30 (Commerce soil), 0, 3, 7, 15 and 22 (Gross Umstadt soil), and 0, 3, 7, 15, 22, 30 and 45 (Drummer #6 soil). Volatile traps for each sample train were also collected and replenished at the same timepoints as the soil samples were removed for analysis.

The collected soil samples were typically extracted four times with an acetonitrile/0.5% ammonium carbonate mixture (50:50 v/v; 20 mL, 10 mL, 10 mL, 10 mL), with the extracts being separated from the soil by centrifugation, decanted off and combined for each sample, in preparation for radioactivity quantification by LSC. Qualitative and quantitative determination of [ $^{14}$C]-IN-D2708 in the soil extracts was undertaken using reversed phase HPLC with radiochemical detection, fraction collection and liquid scintillation analysis.

Radioactivity remaining in the soil samples after the extractions was determined by LSC, following combustion of air-dried samples. Volatile radioactivity, collected in the caustic trapping solutions, was quantified using LSC. It was characterised as $^{14}CO_2$ by applying a standard chemical test involving precipitation of the barium salt of [ $^{14}$C]-carbonate.

**Findings:**

The limits of detection for extract analysis, HPLC analysis and combustion analysis were respectively 0.7% AR (0.007 mg IN-D2708 equivalents/kg soil), 0.05% AR (0.0005 mg IN- D2708 equivalents/kg soil) and 0.02% AR (0.0002 mg IN-D2708 equivalents/kg soil).

Table 31 shows the distribution of total recovered radioactivity into volatiles ($CO_2$), extractable and unextractable radioactivity, for each sampling interval for each incubation.

**Table 31 Radioactive residue distribution and recovery in Commerce, Gross Umstadt and Drummer #6 soils treated with [$^{14}$C]-IN-D2708 (all values as % of applied radioactivity)**

| DAA | Replicate 1 | | | | Replicate 2 | | | |
|---|---|---|---|---|---|---|---|---|
| | $CO_2$ | Extracted | NER | Total | $CO_2$ | Extracted | NER | Total |
| **Commerce soil (silt loam)** | | | | | | | | |
| 0 | nd | 98.5 | 1.5 | 100.0 | nd | 98.1 | 1.2 | 99.3 |
| 3 | 1.7 | 97.3 | 1.6 | 100.6 | 1.8 | 94.2 | 1.6 | 97.5 |
| 7 | 7.2 | 83.5 | 3.3 | 94.0 | 8.2 | 85.6 | 4.1 | 97.9 |
| 15 | 40.7 | 46.5 | 11.4 | 98.6 | 43.5 | 37.9 | 12.5 | 93.8 |
| 22 | 68.7 | 10.3 | 17.0 | 96.0 | 72.3 | 5.5 | 19.0 | 96.7 |
| 30 | 74.9 | 2.4 | 17.8 | 95.1 | 77.2 | 2.9 | 17.3 | 97.4 |
| **Gross Umstadt soil (silt loam)** | | | | | | | | |
| 0 | nd | 97.4 | 1.0 | 98.4 | nd | 92.5 | 1.1 | 93.6 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 3 | 3.4 | 96.0 | 1.9 | 101.3 | 3.4 | 94.5 | 1.9 | 99.7 |
| 7 | 11.3 | 77.9 | 3.4 | 92.6 | 10.6 | 81.0 | 3.4 | 95.0 |
| 15 | 42.9 | 39.8 | 10.3 | 93.0 | 45.1 | 37.2 | 10.6 | 92.9 |
| 22 | 63.5 | 6.4 | 15.8 | 85.7 | 65.4 | 5.2 | 16.2 | 86.8 |
| **Drummer #6 soil (silty clay loam)** | | | | | | | | |
| 0 | nd | 94.0 | 6.1 | 100.0 | nd | 91.9 | 4.8 | 96.7 |
| 3 | 1.3 | 88.2 | 8.5 | 98.1 | 1.2 | 86.1 | 8.4 | 95.7 |
| 7 | 3.9 | 80.1 | 13.9 | 97.9 | 3.6 | 79.4 | 10.8 | 93.8 |
| 15 | 18.4 | 62.4 | 18.6 | 99.4 | 16.3 | 62.1 | 19.8 | 98.2 |
| 22 | 34.7 | 36.6 | 27.2 | 98.6 | 30.6 | 39.7 | 26.9 | 97.2 |
| 30 | 49.8 | 16.4 | 32.7 | 98.9 | 44.5 | 15.5 | 33.3 | 93.3 |
| 45 | 58.7 | 4.4 | 33.3 | 96.3 | 55.9 | 4.7 | 34.5 | 95.1 |

DAA = days after application, NER = non-extractable radioactivity, nd = not determined

Total recoveries of applied radioactivity (mass balance values) from the different incubations ranged from 93.8-100.6% (Commerce), 85.7-101.3% (Gross Umstadt) and 93.3-100.0% (Drummer #6). In each case, the mass balance was calculated as the sum of volatile radioactivity in the caustic traps ( $^{14}CO_2$), radioactivity extracted from the soil and unextracted radioactivity remaining in the soil.

There was extensive mineralisation of IN-D2708 in all three soils as indicated by the substantial increase in $^{14}CO_2$ throughout the incubations. By the end of the incubations, detected levels of $^{14}CO_2$ had reached maximum mean values of 76.1% AR (Commerce – day 30), 64.5% AR (Gross Umstadt – day 22) and 57.3% AR (Drummer #6 – day 45). Levels of unextracted soil radioactivity also increased during the incubations to reach maximum mean values of 18.0% AR (Commerce – day 22), 16.0% AR (Gross Umstadt – day 22) and 33.9% AR (Drummer #6 – day 45).

The soil-extractable radioactivity was largely comprised of IN-D2708, which accounted for over 90% of this radioactivity up to day 15 for all soils. Other degradates were present after day 15 but were not identified. Levels of soil-extractable radioactivity declined sharply during the incubations as IN-D2708 was degraded to carbon dioxide and unextractable soil residues. The results of the analysis of the treated soil samples, showing the mean level of IN-D2708 determined at each sampling interval, are given in Table 32.

**Table 32 Determined residue levels of IN-D2708 in Commerce (silt loam), Gross Umstadt (silt loam) and Drummer #6 (silty clay loam) soils**

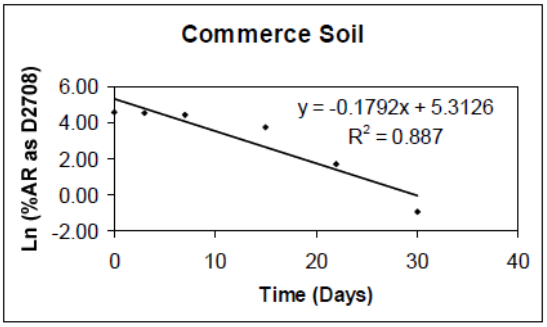
| DAA | [$^{14}$C]-IN-D2708 (% applied radioactivity) | | |
|---|---|---|---|
| | Commerce | Gross Umstadt | Drummer #6 |
| 0 | 97.7 | 94.3 | 92.3 |
| 3 | 95.5 | 94.6 | 86.8 |
| 7 | 84.1 | 79.2 | 79.6 |
| 15 | 41.9 | 38.3 | 61.9 |
| 22 | 5.4 | 3.3 | 34.3 |
| 30 | 0.4 | nd | 11.3 |
| 45 | nd | nd | 0.4 |

DAA = days after application, nd = not determined Residues quoted are the mean of two replicates in each case.

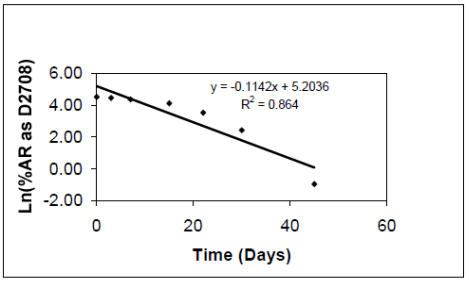
It was assumed that the degradation of IN-D2708 followed simple first order kinetics in each case and so $DT_{50}$ and $DT_{90}$ values for each soil were estimated by performing a linear regression of the natural log (ln) of the amount of IN-D2708 remaining (% AR) against time (days), using the data given in Table 32. The natural logarithm of the average was plotted as a function of time, and a straight line fit to the data; the equation for these lines as well as their variance are depicted in Figure below.

**Figure Soil Degradation Kinetics of [$^{14}$C]-IN-D2708**

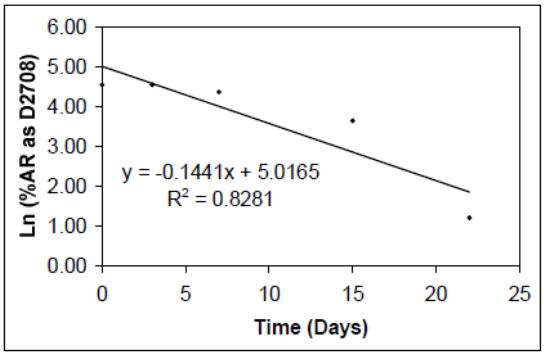
A. Commerce Soil




C. Drummer Soil




B. Gross Umstadt Soil




The outputs from the degradation rate calculations are summarised in Table 33.

**Table 33 $DT_{50}$ and $DT_{90}$ values for IN-D2708 in three aerobic soils under laboratory conditions at 20 °C**

| Soil | k ($days^{-1}$) | $DT_{50}$ (days) | $DT_{90}$ (days) | $r^2$ | Method |
|---|---|---|---|---|---|
| Commerce | 0.1792 | 3.9 | 12.8 | 0.8870 | First order degradation – linear regression of the natural log of the amount of IN-D2708 remaining (% AR) against time (days). |
| Gross Umstadt | 0.1441 | 4.8 | 16.0 | 0.8281 | |
| Drummer #6 | 0.1142 | 6.1 | 20.2 | 0.8640 | |
| Mean | | 5 | 16 | | |

k = first order degradation rate constant ($DT_{50}$ = ln2/k, $DT_{90}$ = ln10/k)

Simple first order kinetics provided an acceptable fit to the data, as indicated by $r^2$ values in the range 0.83-0.89. From the results shown in Table 33, it can be seen that IN-D2708 degraded quickly under the conditions of the study in all three soils tested, with $DT_{50}$ values ranging from 3.9 days to 6.1 days (mean $DT_{50}$ = 5 days). The $DT_{90}$ values ranged from 12.8 days to 20.2 days, with a mean of 16 days. [According to OECD Test Guideline 307 ('Aerobic and Anaerobic Transformation in Soil' – adopted 24 April 2002), the sample of Drummer #6 soil used in this study would be regarded as microbially non-viable (microbial carbon content <1% of total soil organic carbon content). The fact that IN-D2708 degraded only slightly slower in this soil than in the other soils may indicate that significant abiotic degradation occurred in this study as well as microbial degradation.]

**The degradation rate values given in Table 33 are in agreement with the previously reported first order aerobic degradation rates for IN-D2708 that were derived from the results of laboratory soil metabolism studies involving application of oxamyl. The main laboratory degradation rate study on oxamyl (report no. DuPont-2957) yielded IN-D2708 first order $DT_{50}$ and $DT_{90}$ values at 20 °C of 3.6 and 12.1 days (Commerce soil), and 3.4 and 11.2 days (Gross Umstadt soil) (see Table 8.1.2.1-1). The other laboratory**

**soil metabolism studies involving application of oxamyl and providing first order aerobic degradation rates for IN-D2708 (report nos. DuPont-2958 and AMR-1851-90) yielded respective $DT_{50}$ and $DT_{90}$ values for this metabolite of 7.6 and 25.4 days (Nijmegen loam soil, 20 °C – see Table 8.1.2.1-2), and 5.0 and 16.5 days (Madera sandy clay loam soil, 25 °C – see Table 28)**

**Conclusions:**

Following treatment at a rate of 1 mg/kg dry soil (estimated to be less than half of the maximum possible concentration of IN-D2708 that could develop in the test soils), IN-D2708 degraded quickly in three test soils of varying pH, organic matter and clay content (incubated under laboratory aerobic conditions at 20 °C and 40-50% of 0-bar moisture-holding capacity). Degradation was most likely microbially mediated, since the majority of the radiolabel applied as [ $^{14}$C]-IN-D2708 was recovered as $^{14}CO2$ and non-extractable residues. However, the results for Drummer #6 soil, which would be regarded as microbially non-viable according to OECD Test Guideline 307, suggest that significant abiotic degradation may have occurred as well.

DT50 and DT90 values for IN-D2708 were obtained using simple first order kinetics and were estimated to be 3.9 and 12.8 days respectively for Commerce soil (silt loam), 4.8 and 16.0 days respectively for Gross Umstadt soil (silt loam), and 6.1 and 20.2 days respectively for Drummer #6 soil (silty clay loam). These values are in agreement with the previously reported first order aerobic degradation rates for IN-D2708 that were derived from the results of laboratory soil metabolism studies involving application of oxamyl.

The aerobic degradation of metabolites, breakdown and reaction products study DuPont-2675, originally submitted under EU Rev8 Point IIA 7.1.1.2.1.1 and conducted with test material [ $^{14}$C]IN-D2708, was conducted under guideline SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (OECD 307) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl
The degradation rate values given in Table 33 are in agreement with the previously reported first order aerobic degradation rates for IN-D2708 that were derived from the results of laboratory soil metabolism studies involving application of oxamyl.

**B.8.1.2.1.2/02**

| **Reference:** -- | **Report:** | Hashinger, B.M., III, Gaddamidi, V. (2000); Rates of degradation of oxamyl metabolite [$^{14}$C]dimethylcarbonocyanidic amide [IN-N0079] in three aerobic soils<br>**DuPont Report No.:** DuPont-2674<br>**Guidelines:** SETAC Europe (1995) |
|---|---|---|

1. Test material: [$^{14}$C]IN-N0079
   Lot/Batch #: HOTC 548
   Purity: Radiochemical purity - 99.4%

**Materials and Methods:**

The aerobic rate of degradation of [ $^{14}$C]-IN-N0079 ([cyano- $^{14}$C]-IN-N0079, batch 548, specific activity 45.00 µCi/mg, radiochemical purity 99.4%) was investigated in three field- fresh soils at 23 °C. The soils employed were the same ones that were used in the main laboratory degradation rate study on oxamyl (report no. DuPont-2957) and in the laboratory degradation rate study on IN-D2708 (report no. DuPont-2675), i.e. 'Commerce' (silt loam, USA), 'Gross Umstadt' (silt loam, Germany) and 'Drummer #6' (silty clay loam, USA).

Soil samples were used within three months of collection from the field, and were stored at 5°C and sieved (2 mm) before use. Details of the soil characteristics are given in Table 34. [The figures in Table 34 are the same as those previously given for these soils in Tables 8.1.1.1-4 and Table 30, apart from the microbial biomass measurements.] The study report states that the moisture contents of the received soil samples were not adjusted, since they were close to desired levels (40-50% of 0-bar moisture-holding capacity).

Samples of the moist soils (equivalent to 50 g dry soil) were treated with 53.82 µg [ $^{14}$C]-IN- N0079. This treatment rate of ~1 mg/kg dry soil is in line with concentrations of IN-N0079 that could develop in these soils following an application of 5.5 kg oxamyl/ha, and is estimated to be no more than half of the maximum possible concentration of IN-N0079 that could develop in these soils. [Assuming a 1:1 molar relationship between parent

and IN- N0079 and 100% conversion, an application of 5.5 kg oxamyl/ha would give IN-N0079 concentrations of 2.0 mg/kg soil (Commerce), 2.6 mg/kg soil (Gross Umstadt) and 2.1 mg/kg soil (Drummer #6); based on a soil incorporation depth of 10 cm, respective reported bulk soil densities of 1.23 g/cm3, 0.93 g/cm3 and 1.17 g/cm3 and molecular weights (g/mole) of 219.26 for oxamyl and 98.1 for IN-N0079.]

Sufficient samples were prepared so that replicates could be taken for analysis at each sampling timepoint. The treated soil samples were incubated under aerobic conditions, at 23 ± 2 °C, for up to 2.5 hours. No provision was made for the collection of volatile components, as the incubation periods were so short. Duplicate treated soil samples were removed for analysis at each sampling timepoint. The sampling intervals (minutes after test substance application) were 0, 30, 60, 120 and 150 (Commerce soil), 0, 5, 10, 15 and 30 (Gross Umstadt soil), and 0, 15, 30 and 60 (Drummer #6 soil).

**Table 34 Characteristics of Commerce, Gross Umstadt and Drummer #6 soils used to investigate the aerobic soil degradation rate of IN-N0079**

| Parameter | Commerce | Gross Umstadt | Drummer #6 |
|---|---|---|---|
| Origin | Greenville, MS, USA | Gross Umstadt, Germany | Rochelle, IL, USA |
| USDA texture | silt loam | silt loam | silty clay loam |
| % sand | 32.8 | 5.6 | 8.4 |
| % silt | 56.4 | 77.2 | 60.8 |
| % clay | 10.8 | 17.2 | 30.8 |
| pH | 7.0 | 7.8 | 4.8 |
| Organic matter (%) | 0.4 | 2.1 | 4.4 |
| Organic carbon (%) | 0.2 | 1.2 | 2.6 |
| Cation exchange capacity (meq/100 g) | 6.7 | 9.6 | 26.3 |
| Bulk density ($g/cm^3$) | 1.23 | 0.93 | 1.17 |
| 0-bar moisture (%) | 33.3 | 50.0 | 49.4 |
| Biomass (mg microbial C/100 g soil) | 5.25 | 45.3 | 12.4 |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

Soils were tested for microbial viability at the time of study initiation. Since the study was complete within a day, it was not necessary to test the soils at the end of the study also.

The collected soil samples were typically extracted three times with an acetonitrile/water mixture (50:50 v/v, 75 mL per extraction), with the extracts being separated from the soil by centrifugation, decanted off and combined for each sample, in preparation for radioactivity quantification by LSC. [The 15-minute, 30-minute and 60-minute samples from the Drummer#6 incubation were subjected to a fourth extraction, which was analysed separately by LSC and not analysed further, instead of being combined with the first three extracts.] Qualitative and quantitative determination of [ $^{14}$C]-IN-N0079 in the soil extracts was undertaken using reversed phase HPLC with radiochemical detection. Radioactivity remaining in the soil samples after the extractions was determined by LSC, following combustion of air-dried samples.

**Findings:**

The limits of detection for extract analysis, HPLC analysis and combustion analysis were respectively 0.01 mg IN-N0079 equivalents/kg soil, 0.1 mg IN-N0079 equivalents/kg soil and 0.0006 mg IN-N0079 equivalents/kg soil.

Table 35 shows the distribution of total recovered radioactivity into extractable and unextractable radioactivity. As noted previously, volatile radioactivity was not monitored during the study, since the maximum incubation period was only 2.5 hours.

Recoveries of applied radioactivity from the different incubations (calculated as the sum of radioactivity extracted from the soil and unextracted radioactivity remaining in the soil) ranged from 98.61-102.85% (Commerce), 98.94-100.26% (Gross Umstadt) and 97.74-99.01% (Drummer #6). Soil-extractable radioactivity only declined slightly during the incubations, being in the ranges 97.32-101.38% AR (Commerce), 97.36-98.91% AR (Gross Umstadt) and 90.10-95.77% AR (Drummer #6). Levels of unextracted soil radioactivity were in the respective ranges 1.08-1.85% AR, 0.85-1.58% AR and 1.97-8.68% AR for the Commerce, Gross Umstadt and Drummer #6 incubations.

The results of the analysis of the soil extracts, showing the mean level of IN-N0079 determined at each sampling interval, are given in Table 36. IN-N0079 was very rapidly converted to a more polar product (or products), which eluted as a single peak under the analytical conditions used. This accounts for why the soil-extractable radioactivity only declined slightly during the incubations. The product (or products) was not evaluated further.

**Table 35 Average extracted and unextracted radioactivity in Commerce, Gross Umstadt and Drummer #6 soils treated with [$^{14}$C]-IN-N0079 (values as % of applied radioactivity)**

| Time (mins) | Commerce | | | Gross Umstadt | | | Drummer #6 | | |
|---|---|---|---|---|---|---|---|---|---|
| | Extract | NER | Total | Extract | NER | Total | Extract | NER | Total |
| 0 | 101.38 | 1.47 | 102.85 | 98.73 | 0.85 | 99.58 | 95.77 | 1.97 | 97.74 |
| 5 | nd | nd | nd | 97.77 | 1.24 | 99.01 | nd | nd | nd |
| 10 | nd | nd | nd | 98.91 | 1.35 | 100.26 | nd | nd | nd |
| 15 | nd | nd | nd | 97.36 | 1.58 | 98.94 | 95.70 | 2.85 | 98.55 |
| 30 | 97.53 | 1.08 | 98.61 | 97.89 | 1.41 | 99.30 | 90.10 | 8.68 | 98.78 |
| 60 | 99.60 | 1.58 | 101.18 | nd | nd | nd | 91.40 | 7.61 | 99.01 |
| 120 | 97.89 | 1.85 | 99.74 | nd | nd | nd | nd | nd | nd |
| 150 | 97.32 | 1.41 | 98.73 | nd | nd | nd | nd | nd | nd |

NER = non-extractable radioactivity, nd = not determined Figures quoted are the mean of two replicates in each case.

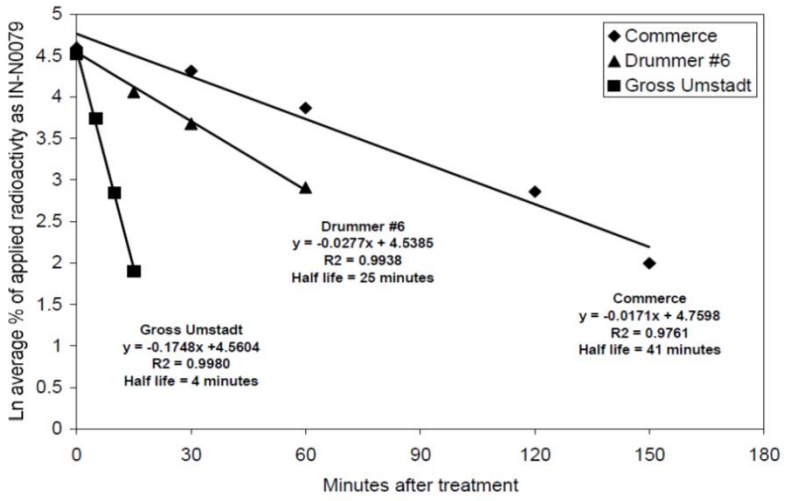
**Table 36 Determined residue levels of IN-N0079 in Commerce (silt loam), Gross Umstadt (silt loam) and Drummer #6 (silty clay loam) soils**

| Time (mins) | [$^{14}$C]-IN-N0079 (% applied radioactivity) | | |
|---|---|---|---|
| | Commerce | Gross Umstadt | Drummer #6 |
| 0 | 98.76 | 91.24 | 99.49 |
| 5 | nd | 42.14 | nd |
| 10 | nd | 17.18 | nd |
| 15 | nd | 6.68 | 57.99 |
| 30 | 74.81 | 0.00 | 39.60 |
| 60 | 47.79 | nd | 18.30 |
| 120 | 17.44 | nd | nd |
| 150 | 7.38 | nd | nd |

nd = not determined
Residues quoted are the mean of two replicates.

It was assumed that the degradation of IN-N0079 followed simple first order kinetics in each case and so $DT_{50}$ and $DT_{90}$ values for each soil were estimated by performing a linear regression of the natural log (ln) of the amount of IN-N0079 remaining (% AR) against time (minutes), using the data given in Table 36. The natural logarithm of the average was plotted as a function of time, and a straight line fit to the data; the equation for these lines as well as their variance are depicted in Figure below.

**Figure Plot of dissipation rate data for IN-N0079 in three soils**




The outputs from the degradation rate calculations are summarised in Table 37.

**Table 37 $DT_{50}$ and $DT_{90}$ values for IN-N0079 in three aerobic soils under laboratory conditions at 23 °C**

| Soil | k ($mins^{-1}$) | $DT_{50}$ (mins) | $DT_{90}$ (mins) | $r^2$ | Method |
|---|---|---|---|---|---|
| Commerce | 0.0171 | 41 | 135 | 0.9761 | First order degradation – linear regression of the natural log of the amount of IN-N0079 remaining (% AR) against time (minutes). |
| Gross Umstadt | 0.1748 | 4 | 13 | 0.9980 | |
| Drummer #6 | 0.0277 | 25 | 83 | 0.9938 | |
| Mean | | 23 | 77 | | |

k = first order degradation rate constant ($DT_{50}$ = ln2/k, $DT_{90}$ = ln10/k)

Simple first order kinetics provided a very good fit to the data, as indicated by $r^2$ values in the range 0.976-0.998. From the results shown in Table 37, it can be seen that IN-N0079 degraded very rapidly under the conditions of the study in all three soils tested, with $DT_{50}$ values ranging from 4 minutes to 41 minutes (mean $DT_{50}$ = 23 minutes). The $DT_{90}$ values ranged from 13 minutes to 135 minutes, with a mean of 77 minutes. [According to OECD Test Guideline 307 ('Aerobic and Anaerobic Transformation in Soil' – adopted 24 April 2002), the sample of Drummer #6 soil used in this study would be regarded as microbially non-viable (microbial carbon content <1% of total soil organic carbon content). The fact that IN-N0079 degraded in this soil at a rate equivalent to that observed in the other soils may indicate that the rapid IN-N0079 degradation observed in this study was largely abiotic in nature.]

**Conclusions:**

Following treatment at a rate of 1 mg/kg dry soil (estimated to be no more than half of the maximum possible concentration of IN-N0079 that could possibly develop in the test soils), IN-N0079 degraded very rapidly in three test soils of varying pH, organic matter and clay content (incubated under laboratory aerobic conditions at 23 ±2 °C and 40-50% of 0-bar moisture-holding capacity). IN-N0079 was converted to a more polar product (or products), which eluted as a single peak under the analytical conditions employed. The results for Drummer #6 soil, which would be regarded as microbially non-viable according to OECD Test Guideline 307, suggest that the degradation was largely abiotic in nature.

$DT_{50}$ and $DT_{90}$ values for IN-N0079 were obtained using simple first order kinetics and were estimated to be 41 and 135 minutes respectively for Commerce soil (silt loam), 4 and 13 minutes respectively for Gross Umstadt soil (silt loam), and 25 and 83 minutes respectively for Drummer #6 soil (silty clay loam).

The aerobic degradation of metabolites, breakdown and reaction products study DuPont-2674, originally submitted under EU Rev8 Point IIA 7.1.1.2.1.1 and conducted with test material [$^{14}$C]IN-N0079, was conducted under guideline SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (OECD 307) and is relied upon.

The rate of degradation for IN-A2213 was clearly defined in the aerobic soil studies performed with oxamyl (DuPont-2958, DuPont-2957, DuPont-2958, and DuPont-39014). Kinetically robust persistence endpoints for IN-A2213 were derived according to FOCUS kinetics in the modelling position paper DuPont-41859 EU. The persistence $DT_{50}$ values for IN-A2213 are summarized under Point B.8.1.2.1.5 in this document and presented in Table .

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.1.2/03**

| | | |
|---|---|---|
| **Reference:** CA 7.1.2.1.2/01 | **Report:** | Ghafoor, A., Zillgens, B. (2015); Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) from laboratory soil degradation studies<br>**DuPont Report No.:** DuPont-41859 EU<br>**Guidelines:** Not applicable<br>**Deviations:** Not applicable<br>**Testing Facility:** Dr. Knoell Consult GmbH, Mannheim, Germany<br>**Testing Facility Report No.:** DuPont-41859 EU<br>**GLP:** No<br>**Certifying Authority:** Not applicable |

A description of this study can be found in Point 0 in this document.

Modelling endpoints derived from the kinetic analyses were corrected for soil moisture content at field capacity (pF2.0) and temperature (20°C), if necessary, to be used in environmental fate modelling tools.
Moisture correction was carried out by multiplying the obtained SFO $DegT_{50}$ values by a moisture correction factor, which is espressed as follows:

$$\left(\frac{\theta}{\theta ref}\right)^{B}$$

where:
θ = actual soil moisture
$\theta_{REF}$= gravimetric water content at pF2
B = 0.7

The actual soil moisture was calculated from the reference moisture holding capacity (MHC) typical for the soil textural class ("gravimetric water content at MWHC") multiplied by soil moisture at which the study had been conducted (% MWHC). The gravimetric water content at MWHC and the gravimetric water content at pF2, based upon the textural class, were taken from the FOCUS groundwater guidance (FOCUS, 2000), if it was not reported in the study report.
If the moisture correction factor was > 1 due to the actual soil moisture being greater than the gravimetric water content at pF2 no adjustment was made, i.e. the moisture correction factor was set to 1.

Temperature correction was carried out using a $Q_{10}$ value of 2.58.

$$f_{temp} = Q_{10}\,\frac{Tact-Tref}{10}$$

for: Tact > 0°C and Tref = 20°C
where: $f_{temp}$ = temperature correction factor
$Q_{10}$= 2.58
Tact = actual temperature
Tref = reference temperatureSummaries of the rate data derived for the major soil metabolites in this position paper are presented in the following tables.

## IN-D2708

**Table 38 Summary of degradation parameters as persistence triggers for IN-D2708**

| Study | Soil/Condition | $DegT_{50}$ (days) | $DegT_{90}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|
| DuPont-2957 | Commerce 20°C | 3.5 | 11.8 | 18.1 | SFO-SFO |
| | Gross-Umstadt 20°C | 3.2 | 10.8 | 21.8 | DFOP-SFO |
| DuPont-2958 | Nijmegen 20°C | 8.8 | 29.4 | 9.0 | SFO-SFO |
| DuPont-2675[a] | Commerce 20°C | 7.6 | 25.3 | 17.7 | SFO |
| | Gross-Umstadt 20°C | 9.5 | 31.6 | 15.1 | SFO |
| | Drummer 20°C | 12.7 | 42.2 | 14.7 | SFO |
| DuPont-39014 | Tama 583 20°C | 6.8 | 22.4 | 15.7 | FOMC-SFO |

[a] IN-D2708 was directly dosed into the soils.

**Table 39a Summary of degradation parameters as modelling endpoints for IN-D2708**

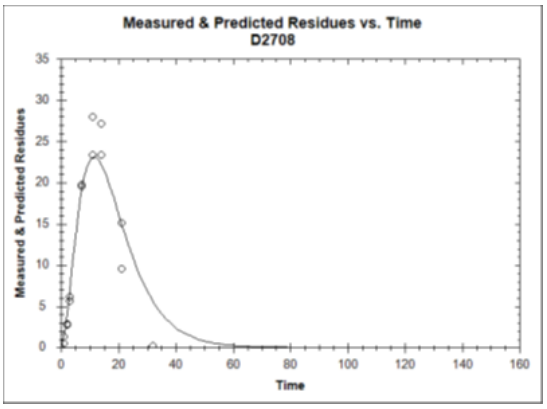
| Study[a] | Soil/Condition | $DegT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DegT_{50}$ at 20°C & 10 kPa[b] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2957 | Commerce 20°C | 3.5 | 33.3 | 13.3 | 26 | 2.2 | SFO-SFO |
| | Gross-Umstadt 20°C | 3.1 | 50 | 20 | 26 | 2.6 | SFO-SFO |
| DuPont-2958 | Nijmegen 20°C | 8.8 | 33.3 | 13.3 | 25 | 5.7 | SFO-SFO |
| DuPont-2675[c] | Commerce 20°C | 7.6 | 33.3 | 13.3 | 26 | 4.8 | SFO |
| | Gross-Umstadt 20°C | 9.5 | 50 | 22 | 26 | 8.5 | SFO |
| | Drummer 20°C | 12.7 | 49.4 | 23.2 | 30 | 10.6 | SFO |
| DuPont-39014 | Tama 583 20°C | 6.8 | - | pF2 | - | 6.8 | FOMC-SFO |

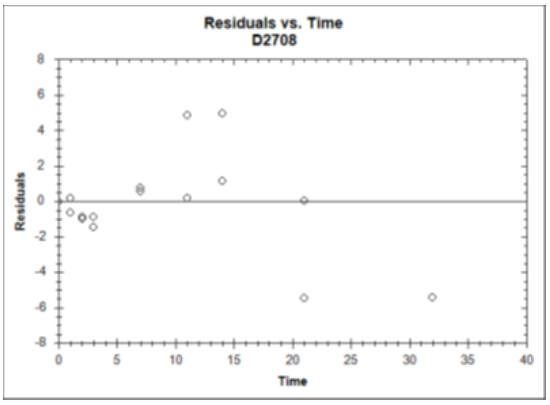
[a] All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU
[b] Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$
[c] IN-D2708 was dosed directly into the soils

The kinetics fittings for degradation of oxamyl's metabolite, IN-D2708 for all seven soils were derived from the respective best-fit model and are presented in figure below

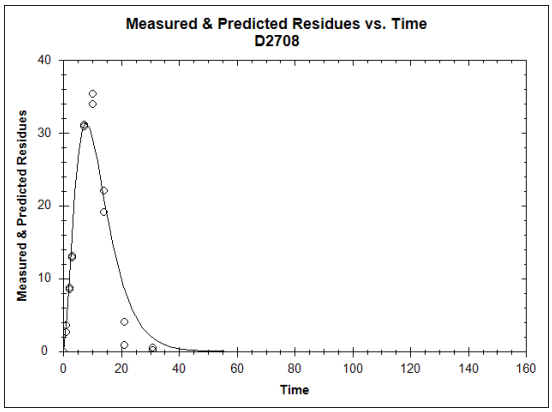
**Figure: The kinetics fittings for degradation of IN-D2708 in soil**

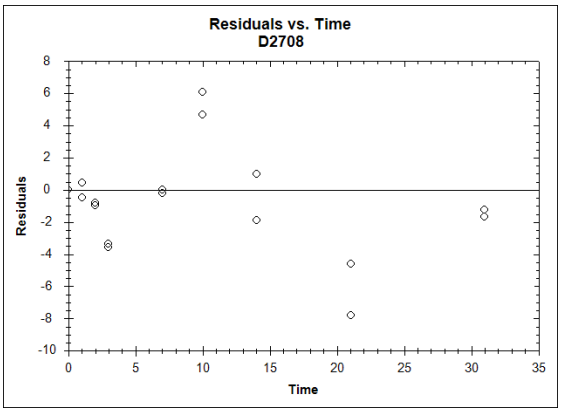
*Commerce soil at 20°C*



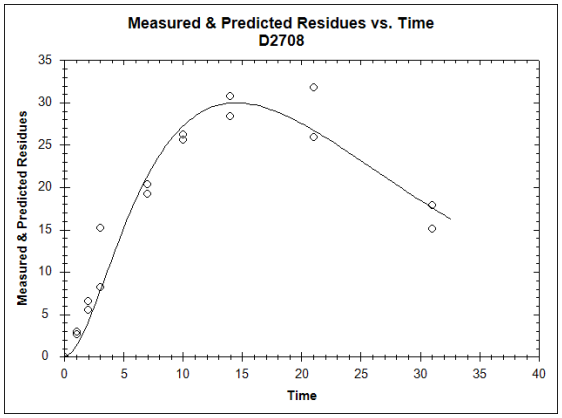



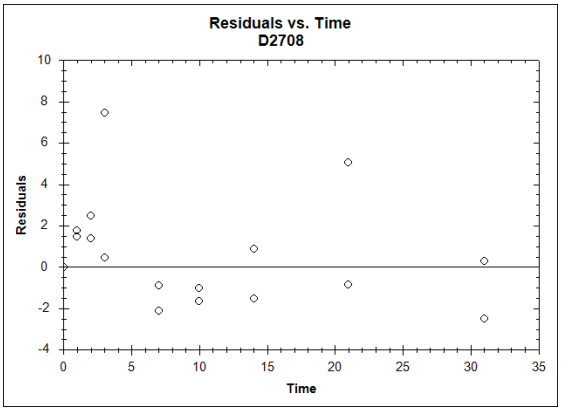

*Gross-Umstadt soil at 20°C*



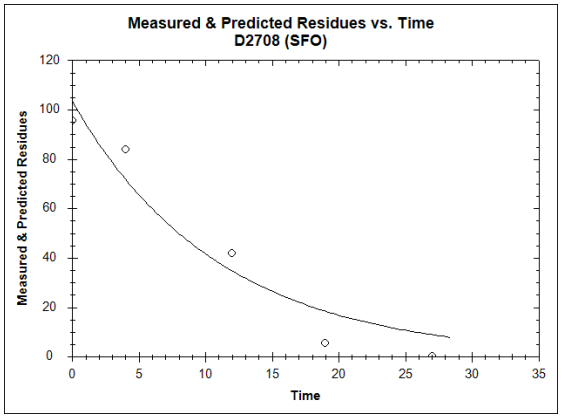



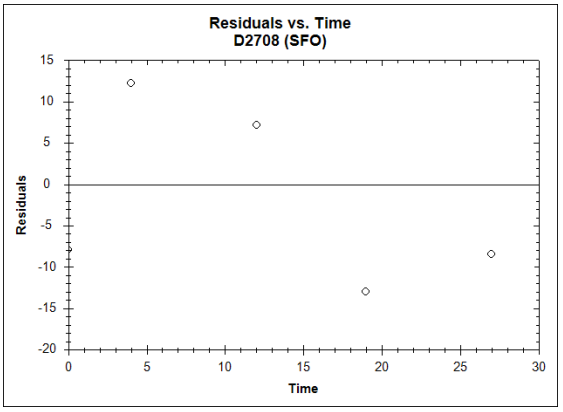

*Nijmegen soil at 20°C*



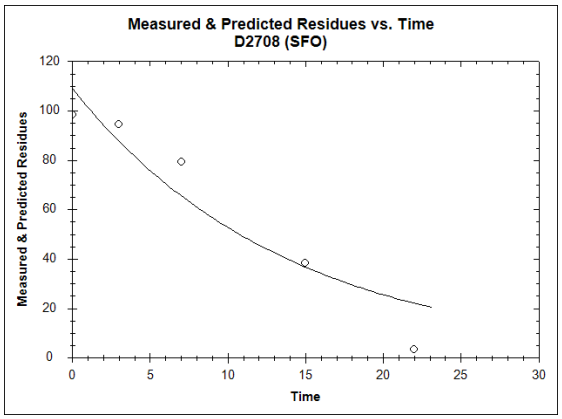



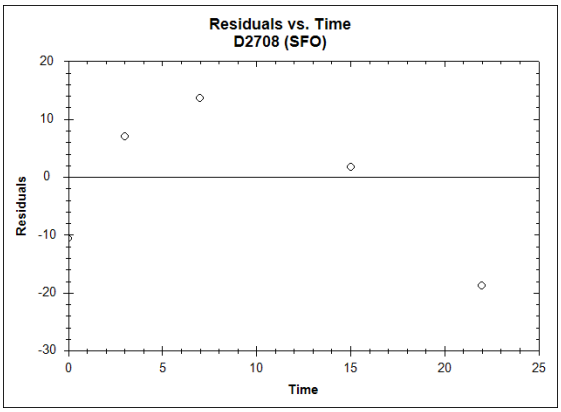

*Commerce soil at 20°C*



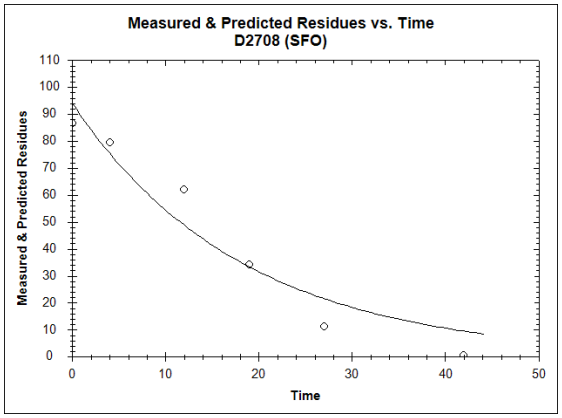



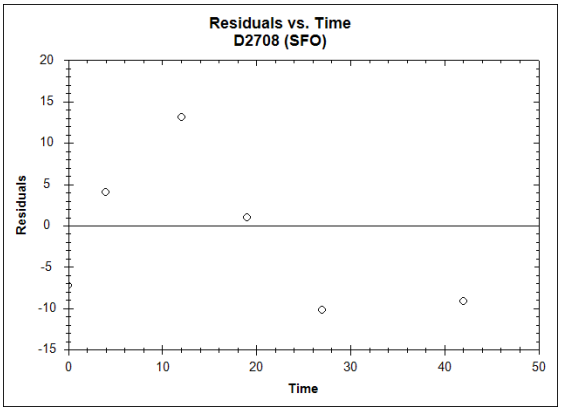

*Gross-Umstadt soil at 20°C*



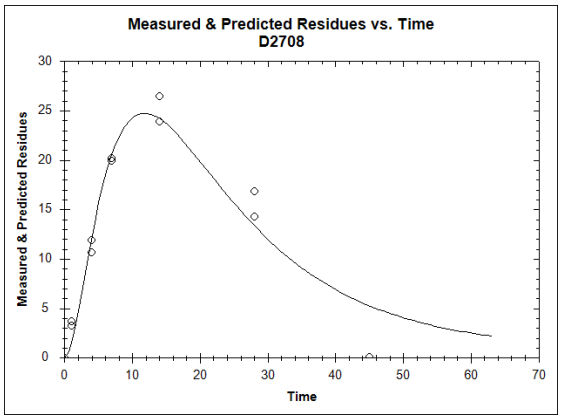



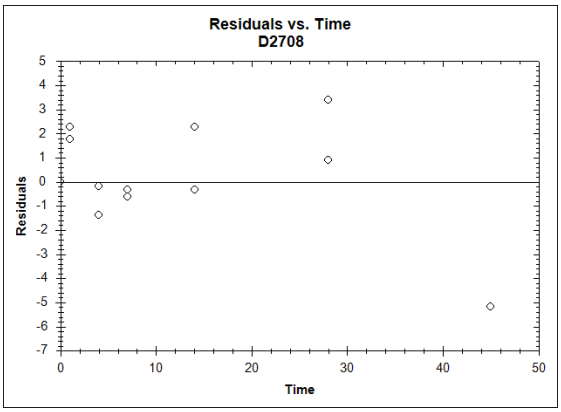

*Drummer soil at 20°C*




*Tama 583 soil at 20°C*




## IN-A2213

**Table 40 Summary of degradation parameters as persistence triggers for IN-A2213**

| Study | Soil/Condition | $DegT_{50}$ (days) | $DegT_{90}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|
| DuPont-2957 | Commerce 20°C | 5.8 | 19.1 | 6.3 | SFO-SFO |
| | Commerce 10°C | 22.1 | 73.3 | 7.5 | SFO-SFO |
| | Gross-Umstadt 20°C | 1.7 | 5.7 | 9.8 | DFOP-SFO |
| DuPont-2958 | Nijmegen 20°C | 1.7 | 5.5 | 23.2 | SFO-SFO |
| DuPont-39014 | Speyer 582 20°C | 1.4 | 4.5 | 16.3 | SFO-SFO |
| | Tama 583 20°C | 1.8 | 5.9 | 17.0 | FOMC-SFO |

**Table 40a Summary of degradation parameters as modelling endpoints for IN-A2213**

| Study[a] | Soil/Condition | $DegT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DegT_{50}$ at 20°C & 10 kPa[c] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2957 | Commerce 20°C | 5.8 | 33.3 | 13.3 | 26 | 3.6 | SFO-SFO |
| | Commerce 10°C[b] | 8.6 | 33.3 | 13.3 | 26 | 5.4 | SFO-SFO |
| | Gross-Umstadt 20°C | 1.6 | 50 | 20 | 26 | 1.3 | SFO-SFO |
| DuPont-2958 | Nijmegen 20°C | 1.7 | 33.3 | 13.3 | 25 | 1.1 | SFO-SFO |
| DuPont-39014 | Speyer 582 20°C | 1.4 | - | pF2 | - | 1.4 | SFO-SFO |
| | Tama 583 20°C | 1.8 | - | pF2 | - | 1.8 | FOMC-SFO |

a All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

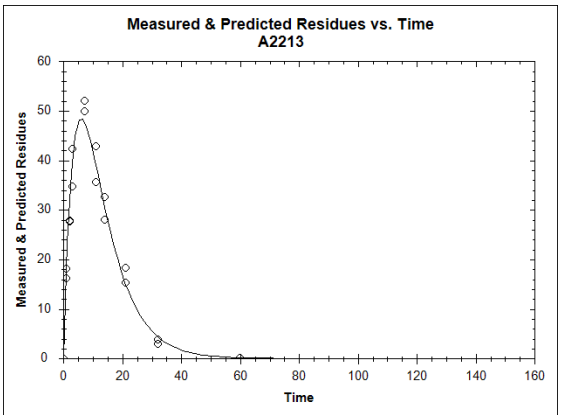
b Corrected to 20°C

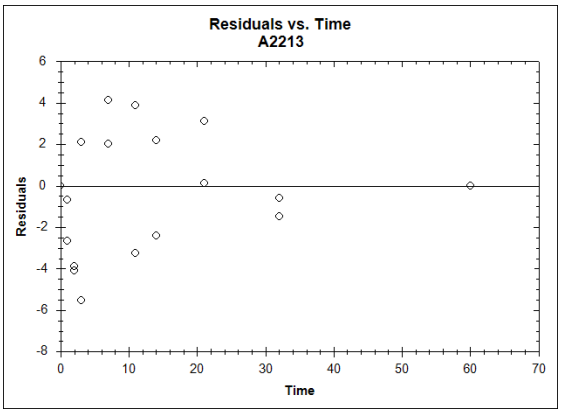
c Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

The kinetics fittings for degradation of oxamyl's metabolite, IN-A2213 for all six soils were derived from the respective best-fit model and are presented in figure below

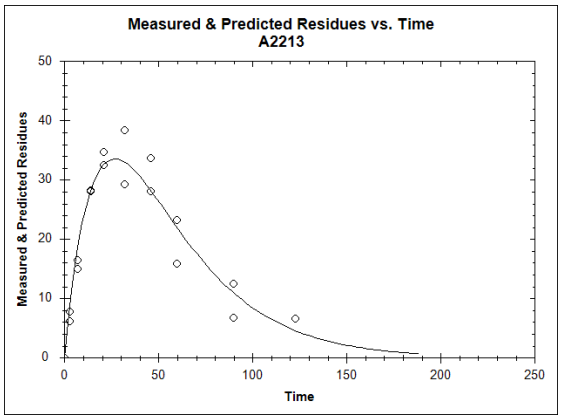
**Figure: The kinetics fittings for degradation of IN-A2213 in soil**

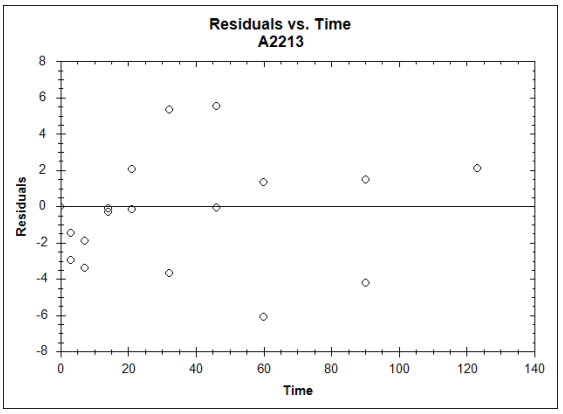
*Commerce soil at 20°*



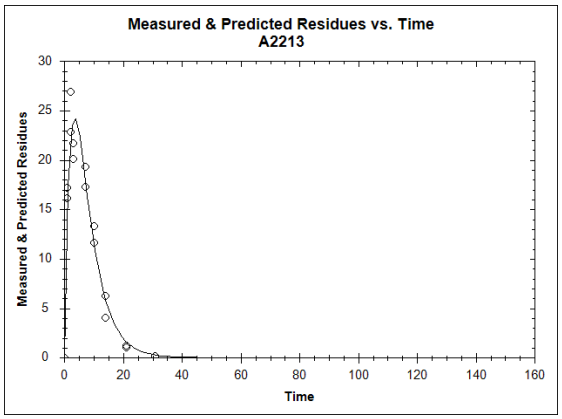



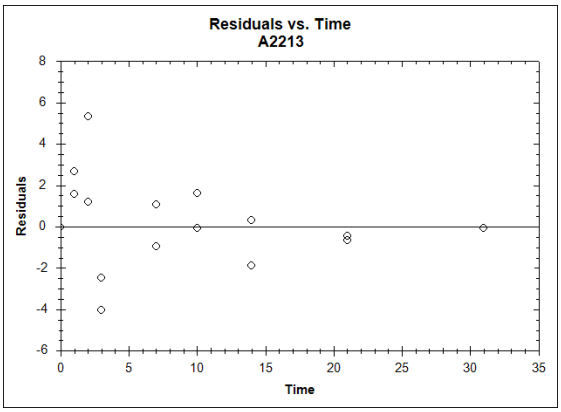

*Commerce soil at 10°C*



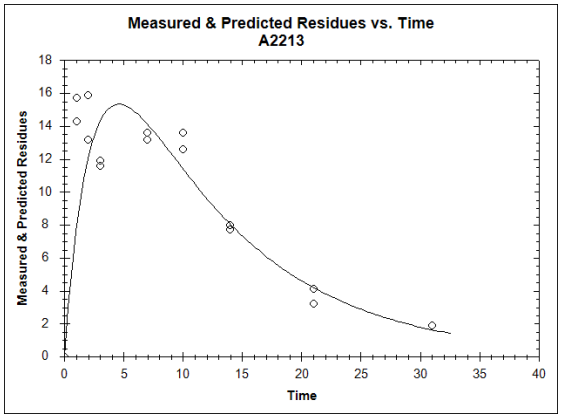



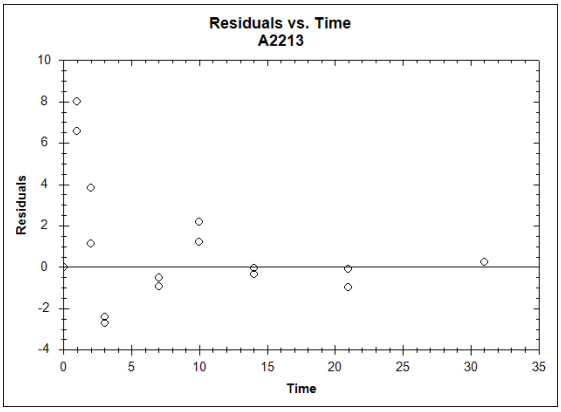

*Gross-Umstadt soil at 20°C*



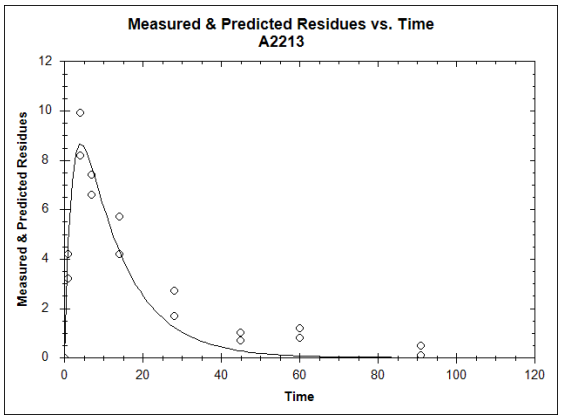



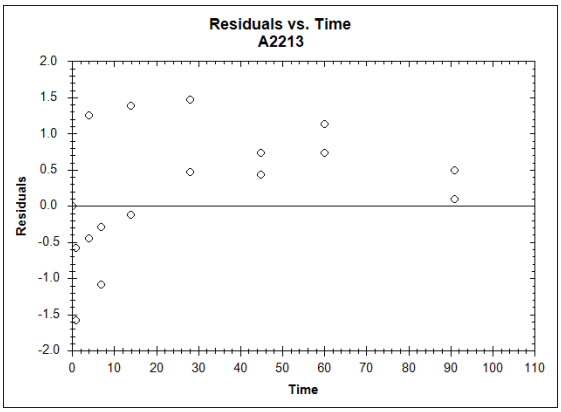

*Nijmegen soil at 20°C*



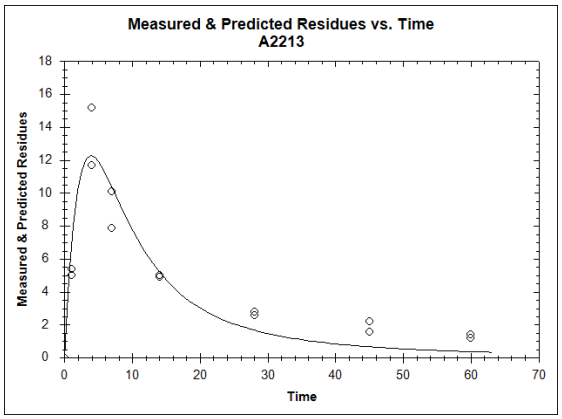



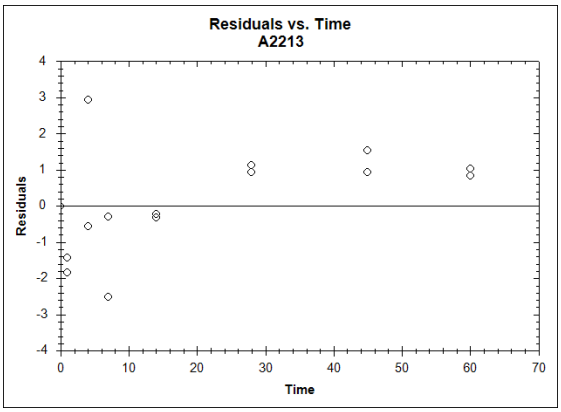

*Speyer 582 soil at 20°C*




*Tama 583 soil at 20°C*




## IN-0079

**Table 41 Summary of degradation parameters as persistence triggers for IN-N0079 (derived from oxamyl photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|
| DuPont-31501 | Tama 20 °C | 2 | 6.5 | 18.0 | SFO-SFO |

**Table 41a Summary of degradation parameters as modelling endpoints for IN-N0079 (derived from oxamyl photodegradation study)**

| Study[a] | Soil/Condition | $DT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DT_{50}$ at 20°C & 10 kPa[b] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-31501 | Tama 20°C | 2 | 30 | 22.5 | 30 | 1.6 | SFO |

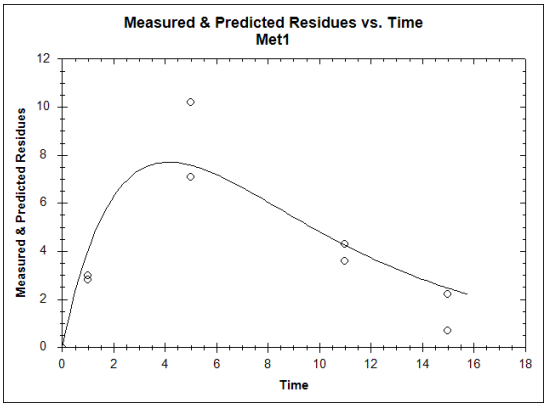
a All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

b Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

The kinetics fittings for degradation of oxamyl's metabolite, IN-N0079 derived from oxamyl photodegradation study were derived from the respective best-fit model and are presented in figure below

**Figure: The kinetics fittings for degradation of IN-N0079 in soil (derived from oxamyl photodegradation study)**

*Tama soil at 20°*



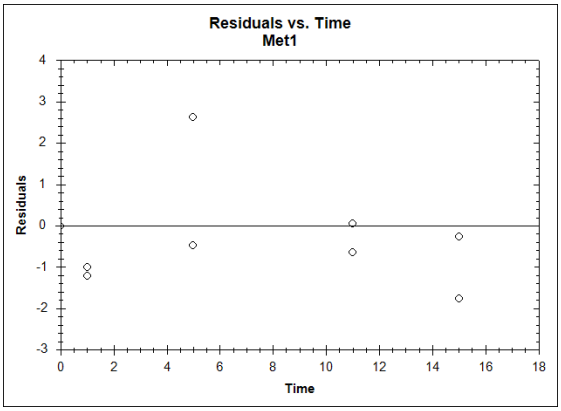



**Table 42 Summary of degradation parameters as persistence triggers for IN-N0079 (applied as parent)**

| Study[a] | Soil/Condition | $DegT_{50}$ (Minutes) | $DegT_{90}$ (Minutes) | Model |
|---|---|---|---|---|
| DuPont-2674[b] | Commerce 23°C | 49.5 | 164.4 | SFO |
| | Gross-Umstadt 23°C | 4.0 | 13.2 | SFO |
| | Drummer #6 23°C | 23.0 | 76.5 | SFO |

a All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

b IN-N0079 was directly dosed into the soils.

**Table 42a Summary of degradation parameters as modelling endpoints for IN-N0079 (applied as parent)**

| Study[a] | Soil/Condition | $DegT_{50}$ at 20°C (minutes) | Moisture content (w/w %) | | | $DegT_{50}$ at 20°C & 10 kPa[b] (minutes) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2674[c] | Commerce 23°C | 65.8 | 33.3 | 13.3 | 26 | 41.2 | SFO |
| | Gross-Umstadt 23°C | 5.3 | 50 | 20 | 26 | 4.4 | SFO |
| | Drummer #6 23°C | 30.8 | 49.4 | 19.9 | 30 | 23.0 | SFO |

a All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

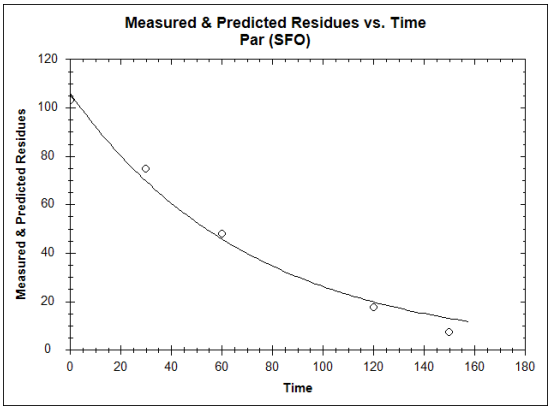
b Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

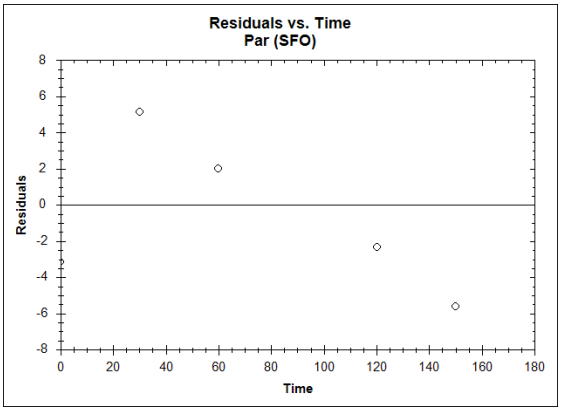
c IN-N0079 was dosed directly into the soils

The kinetics fittings for degradation of oxamyl's metabolite, IN-N0079 for all three soils (applied as parent) were derived from the respective best-fit model and are presented in figure below

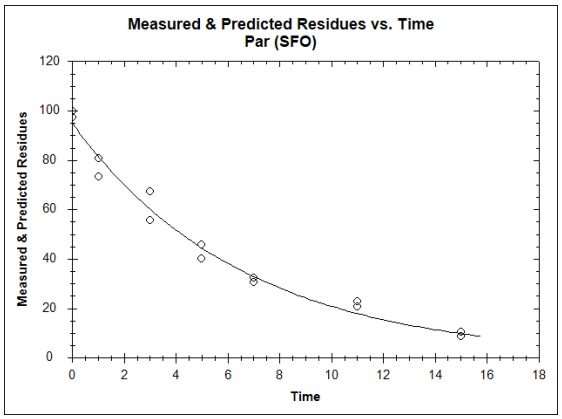
**Figure: The kinetics fittings for degradation of IN-N0079 in soil applied as parent**

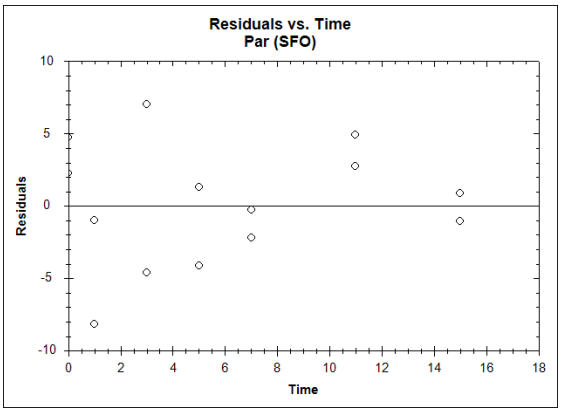
*Commerce soil at 23°C*



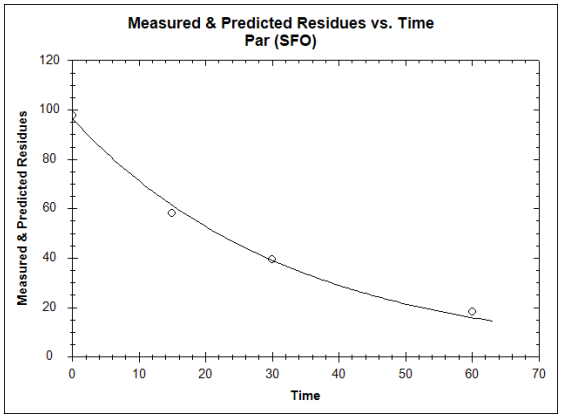



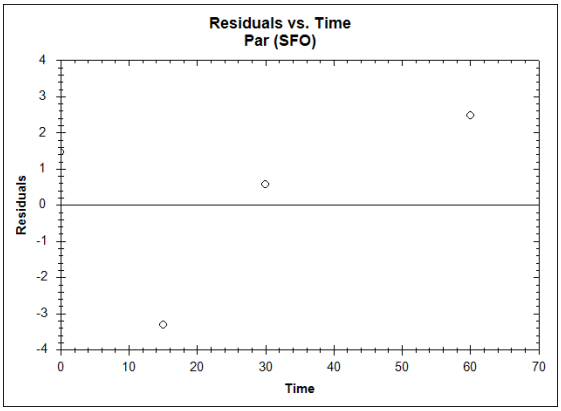

*Gross-Umstadt soil at 23°C*




*Drummer soil at 23°C*




**RMS comments and conclusion**

This study is considered corrected to harmonize the derivation of degradation parameters from soil metabolism. Residue data of aerobic soil degradation studies in 6 soils for metabolite IN-A2213 under laboratory conditions (DuPont-2957, DuPont-2958, and DuPont-39014); 7 soils for metabolite IN-D2708 under laboratory conditions (DuPont-2957, DuPont-2958, DuPont-2675 and DuPont-39014) and 3 soils for metabolite IN-N0079 under laboratory conditions (DuPont-2674) were re-evaluated to derive persistence and modelling endpoints for oxamyl under aerobic soil conditions. One photodegradation study for metabolite IN-N0079 in one soil under laboratory conditions (DuPont-31501) was re-evaluated to derive persistence and modelling endpoints for metabolite IN-N0079 under irradiated conditions (photodegradation).
The kinetic assessments conducted are in full compliance with the FOCUS kinetics guidance and the imput parameters can be used for ground water and surface water risk assessment.

*B.8.1.2.1.3 Anaerobic degradation of the active substance*

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.1.2.1.3/01**

| | | |
|---|---|---|
| **Reference:**<br>-- | **Report:** | Spare, W.C. (1991); Anaerobic soil metabolism of [1-$^{14}$C]oxamyl in Madera, California soil<br><br>**DuPont Report No.:** AMR 1851-90<br><br>**Guidelines:** U.S. EPA 162-2 (1982) |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 311
   Purity: Radiochemical purity - >99%

**Materials and Methods:**

The metabolism and degradation of [1- $^{14}$C]-oxamyl ([1- $^{14}$C]-DPX-D1410, batch 311, specific activity 78.2 µCi/mg, radiochemical purity >99%) were studied in a US sandy clay loam soil (from Madera, California) under aerobic conditions followed by anaerobic conditions. The determination of degradation rates for oxamyl and metabolites IN-A2213 and IN-D2708 during the anaerobic portion of the study is described here.

The initial aerobic portion of the study involved incubation of Madera soil (pH 7.7) at 25 °C, with soil moisture content being maintained at 70-75% of field capacity. A nominal treatment rate of 9.5 mg a.s./kg dry soil was used, corresponding to an application rate of 11.875 kg a.s./ha (based on a soil incorporation depth of 10 cm and the reported bulk soil density of 1.25 g/cm3). After 11 days aerobic incubation (corresponding approximately to one half-life for oxamyl under these aerobic conditions), 20 of the 38 flasks originally prepared were converted to anaerobic conditions by adding deionised water (to a depth of about 1 cm) and purging nitrogen through the test vessels into the traps for volatiles.

The experimental procedures for the subsequent anaerobic incubation have already been described in section B.8.1.1.2.1 and details of the soil characteristics have been given previously in Table 6.

Whole-system degradation rates (soil and flood water combined) for oxamyl, IN-A2213 and IN-D2708 were determined on the basis of the amounts of these substances extracted from the flooded soil samples. [This procedure constitutes a point of departure from SETAC guidance, which recommends that the overlying flood water be first removed from the soil solids, so that these components can be analysed separately.] The figures for the amounts of the substances detected during the anaerobic portion of the study (expressed as a percentage of applied radioactivity) have been given in Table 11.

The original study reported an anaerobic degradation rate for oxamyl, which was obtained assuming first order degradation (linear regression of the natural log (ln) of the percentage of oxamyl remaining against time, least-squares fit), using the anaerobic timepoints 0, 1, 3, 7 and 10 days. However, the notifier also estimated DT50 and DT90 values for IN-A2213 and IN- D2708. These calculations were performed in the same way as for the parent, except that they used the timepoint at which the maximum level of metabolite was detected as the starting point. The maximum detected level of IN-A2213 (69.5% AR) occurred after 20 days anaerobic incubation, and so the timepoints day 20, day 32, day 45 and day 60 were used in this case. For IN-D2708, the timepoints day 32, day 45 and day 60 were used, since the maximum detected level of this metabolite (23.1% AR) occurred after 32 days anaerobic incubation. This approach to calculating anaerobic DT50 and DT90 values for IN-A2213 and IN-D2708 should provide conservative estimates, since there should have been metabolite formation during the considered time periods, in addition to metabolite degradation, and no allowance was made for the contribution of metabolite formation.

**Findings:**

The outputs from the degradation rate calculations for the anaerobic portion of the study are summarised in Table .

**Table 43 Whole-system $DT_{50}$ and $DT_{90}$ values for oxamyl, IN-A2213 and IN-D2708 in flooded Madera sandy clay loam soil incubated anaerobically at 25 °C**

| Substance | k (days$^{-1}$) | $DT_{50}$ (days) | $DT_{90}$ (days) | $r^2$ | Method |
|---|---|---|---|---|---|
| oxamyl | 0.123 | 6 | 19 | 0.945 | linear simple first order kinetics |
| IN-A2213 | 0.0285 | 24 | 81 | 0.968 | linear simple first order kinetics |
| IN-D2708 | 0.0340 | 20 | 68 | 0.741 | linear simple first order kinetics |

k = first order degradation rate constant ($DT_{50}$ = ln2/k, $DT_{90}$ = ln10/k)

Anaerobic timepoints day 0, day 1, day 3, day 7 and day 10 were used for oxamyl, day 20, day 32, day 45 and day 60 were used for IN-A2213, and day 32, day 45 and day 60 were used for IN-D2708 (see Table 11).

Simple first order kinetics provided an acceptable fit to the data, with r2 values ranging from 0.741-0.968. Oxamyl, IN-A2213 and IN-D2708 all degraded readily in the anaerobic portion of the study, as indicated by respective whole-system DT50 values of 6, 24 and 20 days (and corresponding DT90 values of 19, 81 and 68 days). In comparison with the degradation rates obtained from the initial portion of the study, it can be seen that oxamyl degraded more rapidly under anaerobic conditions than under aerobic conditions (aerobic DT50 = 11.5 days), whereas IN-A2213 and IN-D2708 both degraded more rapidly under aerobic conditions (respective aerobic DT50 values of 6.4 and 5.0 days) (see Table 28). However, as noted above, the anaerobic DT50 and DT90 values obtained for IN-A2213 and IN-D2708 may be conservative estimates.

It was noted previously (during the discussion of this study in section B.8.1.1.2.1) that the peak value for IN-D2708 under anaerobic conditions (23.1% AR, day 32) was similar to its aerobic peak value (20.3%, day 21), whereas the anaerobic peak value for IN-A2213 (69.5% AR, day 20) was significantly higher than its aerobic peak value (24.3% AR, day 10); suggesting that, compared to aerobic incubation, anaerobic incubation increased the rate of oxamyl degradation to IN-A2213 but slowed the rate of degradation of IN-A2213 to IN-D2708.

**Conclusions:**

Oxamyl degraded rapidly under anaerobic conditions in the dark, at 25 ±1 °C, following application at a nominal rate of 9.5 mg a.s./kg dry soil to a US sandy clay loam soil (Madera, California) and aerobic incubation in the dark for 11 days at 25 ±1 °C. Whole-system oxamyl DT50 and DT90 values (soil and flood water combined) were obtained using simple first order kinetics and were estimated to be 6 and 19 days respectively.

IN-A2213 and IN-D2708 also degraded readily under anaerobic conditions. Whole-system DT50 and DT90 values for these metabolites were obtained by applying simple first order kinetics, using the timepoint at which the maximum level of metabolite was detected as the starting point, and were estimated to be 24 and 81 days respectively for IN-A2213, and 20 and 68 days respectively for IN-D2708.

A comparison of the results from the aerobic and anaerobic portions of the study indicates that oxamyl degraded more rapidly under anaerobic conditions (aerobic DT50 = 11.5 days), whereas IN-A2213 and IN-D2708 both degraded more rapidly under aerobic conditions (respective aerobic DT50 values of 6.4 and 5.0 days).

The anaerobic degradation of the active substance study AMR 1851-90, originally submitted under EU Rev8 Point IIA 7.1.1.2.1.1, 7.1.1.2.1.2 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline U.S. EPA 162-2 (1982). A review of this study indicates it partially meets the current guideline (OECD 307); deviations include a mass balance below 90% AR at the last two sampling points. However, reconduct is unlikely to yield a significantly different result because the rate of degradation of oxamyl was already well demonstrated by the previous seven sampling points with compliant mass balance. At the last two sampling points, where the mass balance was low, oxamyl had already degraded to less than 0.1% AR, and thus the degradation had already proceeded through the $DT_{50}$ and $DT_{90}$ when the mass balance dropped outside the guideline range of 90–110% AR. Acceptable mass balance was maintained long enough in the study for robust kinetics of the degradation rate to be determined. Therefore, this study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.

*B.8.1.2.1.4 Anaerobic degradation of relevant metabolites, breakdown and reaction products*

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.1.2.1.4/01**

| | | |
|---|---|---|
| **Reference:** -- | **Report:** | Spare, W.C. (1991); Anaerobic soil metabolism of [1-$^{14}$C]oxamyl in Madera, California soil<br>**DuPont Report No.:** AMR 1851-90 |

| | | |
|---|---|---|
| | | **Guidelines:** U.S. EPA 162-2 (1982) |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 311
   Purity: Radiochemical purity - >99%

A full description of this study can be found in Point B.8.1.1.2 in this document.

The anaerobic part of degradation study AMR 1851-90, originally submitted under EU Rev8 Point IIA 7.1.1.1.1 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline U.S. EPA 162-2 (1982). A review of this study indicates that the anaerobic soil experiment performed in this study fully meets the current guideline (OECD 307). Therefore, this study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.1.4/02**

| | | |
|---|---|---|
| **Reference: CA 7.1.2.1.4/01** | **Report:** | Spare, W.C. (1992); Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil, supplemental report<br>**DuPont Report No.:** AMR 1851-90, Supplement No. 1<br>**Guidelines:** U.S. EPA 162-2 (1982)<br>**Deviations:** None<br>**Testing Facility:** Agrisearch, Inc, Frederick, Maryland, USA<br>**Testing Facility Report No.:** 1712<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

A full description of this study can be found in Point B.8.1.1.2 in this document. The purpose of this supplement was to look for the presence of volatile metabolites. Due to the abbreviated sampling schedule, rate data was not derived from this supplement.

*B.8.1.2.1.5 Overall assessment: Rate of degradation in soil*

Six aerobic soil degradation studies have been conducted to investigate the rate of degradation of oxamyl and its metabolites. These studies are DuPont-2958, DuPont-2957, AMR 1851-90, DuPont-39014, DuPont-2675, and DuPont-2674. Since the studies do not contain a kinetic evaluation of the data according to recent FOCUS recommendations, a re-evaluation of the test results has been prepared in the modelling position paper DuPont-41859 EU. The results of this current modelling are presented in the following paragraphs.

The rate of aerobic degradation of oxamyl in the laboratory was measured in 9 different soils. Under laboratory conditions, the persistence $DT_{50}$ values ranged from 0.6 to 15.8 days at 20°C. No correlation was observed between the rate of degradation of oxamyl and soil pH (r = 0.12), as depicted in Figure 4. Laboratory studies show that oxamyl is readily degraded in aerobic soils to form three major metabolites and then extensively mineralized to $CO_2$ (max 81% AR). The persistence endpoints derived for oxamyl are listed in Table .

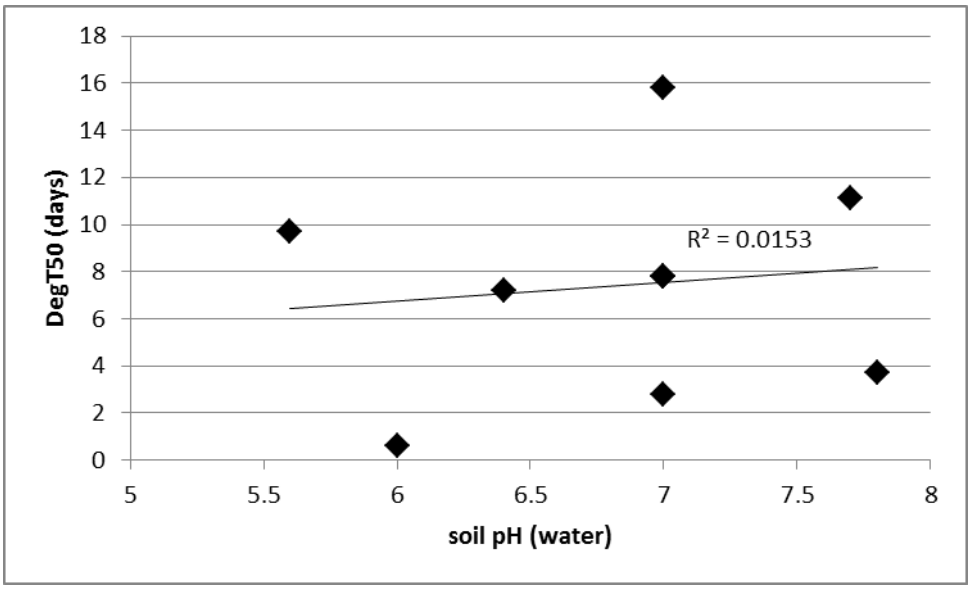
**Table 44 Summary of degradation parameters as persistence triggers for oxamyl**

| Study | Soil/Condition | $DegT_{50}$ (days) | $DegT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-2957 | Commerce 20°C | 2.8 | 9.3 | SFO |
| | Commerce 10°C | 15.8 | 52.3 | SFO |
| | Gross-Umstadt 20°C | 3.7 | 13.9 | DFOP |
| AMR 1851-90 | Madera 25°C | 11.1 | 36.8 | SFO |
| DuPont-2958 | Nijmegen 20°C | 7.8 | 25.8 | SFO |
| DuPont-39014 | Goch 597 20°C | 0.6 | 2.0 | SFO |
| | LRA-D 588 20°C | 9.7 | 79.5 | DFOP |
| | Speyer 582 20°C | 7.2 | 24.0 | SFO |
| | Tama 583 20°C | 7.8 | 48.5 | FOMC |

**Table 44a Summary of degradation parameters as modelling endpoints for oxamyl**

| Study | Soil/Condition | $DegT_{50}$ (days) | Moisture Content (w/w %) | | | $DegT_{50}$ at 20 °C & 10kPa (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2957 | Commerce 20°C | 2.8 | 33.3 | 13.3 | 26 | 1.8 | SFO |
| | Commerce 10°C | 6.1 | 33.3 | 13.3 | 26 | 3.8 | SFO |
| | Gross-Umstadt 20°C | 4.0 | 50 | 20 | 26 | 3.3 | SFO |
| AMR 1851-90 | Madera 25°C | 17.8 | 15.4 | 11.6 | 22 | 11.4 | SFO |
| DuPont-2958 | Nijmegen 20°C | 7.8 | 33.3 | 13.3 | 25 | 5.0 | SFO |
| DuPont-39014 | Goch 597 20°C | 0.6 | - | pF2 | - | 0.6 | SFO |
| | LRA-D 588 20°C | 19.4 | - | pF2 | - | 19.4 | FOMC $DegT_{90}$/3.32 |
| | Speyer 582 20°C | 7.2 | - | pF2 | - | 7.2 | SFO |
| | Tama 583 20°C | 14.3 | - | pF2 | - | 14.3 | FOMC $DegT_{90}$/3.32 |

**Figure 4 Oxamyl degradation rates as a function of soil pH**



Laboratory rate of degradation studies were performed with IN-D2708 and IN-N0079 applied as parent (DuPont-2675 and DuPont-2674). In addition, $DT_{50}$ values for IN-A2213 were derived from the laboratory degradation studies performed with oxamyl. The rate of degradation of IN-D2708 was determined in a laboratory study with three soils (DuPont-2675) and from the robust pathway fits in the oxamyl rate of degradation studies (DuPont-2957, DuPont-2958, and DuPont-39014). Under laboratory conditions, the persistence $DT_{50}$ values ranged from 3.2 to 12.7 days at 20°C. These studies showed that the degradation of IN-D2708 in soil is rapid, resulting in the formation of $CO_2$ and non-extractable residues. The persistence endpoints derived for IN-D2708 are listed in the following table.

**Table 45 Summary of degradation parameters as persistence triggers for IN-D2708**

| Study | Soil/Condition | $DegT_{50}$ (days) | $DegT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-2957 | Commerce 20°C | 3.5 | 11.8 | SFO-SFO |
| | Gross-Umstadt 20°C | 3.2 | 10.8 | DFOP-SFO |
| DuPont-2958 | Nijmegen 20°C | 8.8 | 29.4 | SFO-SFO |
| DuPont-2675[a] | Commerce 20°C | 7.6 | 25.3 | SFO |
| | Gross-Umstadt 20°C | 9.5 | 31.6 | SFO |
| | Drummer 20°C | 12.7 | 42.2 | SFO |
| DuPont-39014 | Tama 583 20°C | 6.8 | 22.4 | FOMC-SFO |

[a] IN-D2708 was directly dosed into the soils.

**Table 45aSummary of degradation parameters as modelling endpoints for IN-D2708**

| Study[a] | Soil/Condition | $DegT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DegT_{50}$ at 20°C & 10 kPa[b] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2957 | Commerce 20°C | 3.5 | 33.3 | 13.3 | 26 | 2.2 | SFO-SFO |
| | Gross-Umstadt 20°C | 3.1 | 50 | 20 | 26 | 2.6 | SFO-SFO |
| DuPont-2958 | Nijmegen 20°C | 8.8 | 33.3 | 13.3 | 25 | 5.7 | SFO-SFO |
| DuPont-2675[c] | Commerce 20°C | 7.6 | 33.3 | 13.3 | 26 | 4.8 | SFO |
| | Gross-Umstadt 20°C | 9.5 | 50 | 22 | 26 | 8.5 | SFO |
| | Drummer 20°C | 12.7 | 49.4 | 23.2 | 30 | 10.6 | SFO |
| DuPont-39014 | Tama 583 20°C | 6.8 | - | pF2 | - | 6.8 | FOMC-SFO |

[a] All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

[b] Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

[c] IN-D2708 was dosed directly into the soils

The rate of degradation of IN-N0079 was determined in a laboratory study with three soils (DuPont-2674). Under laboratory conditions, the persistence $DT_{50}$ values ranged from 4.0 to 49.5 minutes at 20°C. This laboratory study demonstrated that IN-N0079 is rapidly degraded in soils. The persistence endpoints derived for IN-N0079 are listed in Table 6.

**Table 46 Summary of degradation parameters as persistence triggers for IN-N0079 (applied as parent)**

| Study | Soil/Condition | $DegT_{50}$ (minutes) | $DegT_{90}$ (minutes) | Model |
|---|---|---|---|---|
| DuPont-2674[a] | Commerce 23°C | 49.5 | 164.4 | SFO |
| | Gross-Umstadt 23°C | 4.0 | 13.2 | SFO |
| | Drummer #6 23°C | 23.0 | 76.5 | SFO |

[a] IN-N0079 was directly dosed into the soils.

**Table 46a Summary of degradation parameters as modelling endpoints for IN-N0079 (applied as parent)**

| Study[a] | Soil/Condition | $DegT_{50}$ at 20°C (minutes) | Moisture content (w/w %) | | | $DegT_{50}$ at 20°C & 10 kPa[b] (minutes) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2674[c] | Commerce 23°C | 65.8 | 33.3 | 13.3 | 26 | 41.2 | SFO |
| | Gross-Umstadt 23°C | 5.3 | 50 | 20 | 26 | 4.4 | SFO |
| | Drummer #6 23°C | 30.8 | 49.4 | 19.9 | 30 | 23.0 | SFO |

[a] All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU
[b] Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$
[c] IN-N0079 was dosed directly into the soils

Six reliable $DT_{50}$ values for IN-A2213 were derived from the aerobic degradation studies with oxamyl (DuPont-2958, DuPont-2957, and DuPont-39014). In these studies, IN-A2213 was observed to degrade with a $DT_{50}$ of 1.4 to 22.1days at 20°C. The persistence endpoints derived for IN-A2213 are listed in Table 7.

**Table 47 Summary of degradation parameters as persistence triggers for IN-A2213**

| Study | Soil/Condition | $DegT_{50}$ (days) | $DegT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-2957 | Commerce 20°C | 5.8 | 19.1 | SFO-SFO |
| | Commerce 10°C | 22.1 | 73.3 | SFO-SFO |
| | Gross-Umstadt 20°C | 1.7 | 5.7 | DFOP-SFO |
| DuPont-2958 | Nijmegen 20°C | 1.7 | 5.5 | SFO-SFO |
| DuPont-39014 | Speyer 582 20°C | 1.4 | 4.5 | SFO-SFO |
| | Tama 583 20°C | 1.8 | 5.9 | FOMC-SFO |

**Table 47a Summary of degradation parameters as modelling endpoints for IN-A2213**

| Study[a] | Soil/Condition | $DegT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DegT_{50}$ at 20°C & 10 kPa[c] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-2957 | Commerce 20°C | 5.8 | 33.3 | 13.3 | 26 | 3.6 | SFO-SFO |
| | Commerce 10°C[b] | 8.6 | 33.3 | 13.3 | 26 | 5.4 | SFO-SFO |
| | Gross-Umstadt 20°C | 1.6 | 50 | 20 | 26 | 1.3 | SFO-SFO |
| DuPont-2958 | Nijmegen 20°C | 1.7 | 33.3 | 13.3 | 25 | 1.1 | SFO-SFO |
| DuPont-39014 | Speyer 582 20°C | 1.4 | - | pF2 | - | 1.4 | SFO-SFO |
| | Tama 583 20°C | 1.8 | - | pF2 | - | 1.8 | FOMC-SFO |

[a] All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU
[b] Corrected to 20°C
[c] Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

The rate of oxamyl soil photolysis was modelled to occur with a persistence $DT_{50}$ of 4.6 days (Table ). The rate of degradation of the major soil photolysis metabolite, IN-N0079, was also modelled. The persistence $DT_{50}$ for IN-N0079 in the presence of light is 2 days (Table 7).

**Table 48 Summary of degradation parameters as persistence triggers for oxamyl degradation in light (photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-31501 | Tama 20°C | 4.6 | 15.2 | SFO |

**Table 48a Summary of degradation parameters as modelling endpoints for oxamyl in light (photolysis study)**

| Study[a] | Soil/Condition | $DT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DT_{50}$ at 20°C & 10 kPa[b] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-31501 | Tama 20°C | 4.6 | 30 | 22.5 | 30 | 3.8 | SFO |

[a] All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

[b] Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

**Table 49Summary of degradation parameters as persistence triggers for IN-N0079 (derived from oxamyl photolysis study)**

| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Model |
|---|---|---|---|---|
| DuPont-31501 | Tama 20 °C | 2 | 6.5 | SFO-SFO |

**Table 49a Summary of degradation parameters as modelling endpoints for oxamyl IN-N0079 (derived from oxamyl photodegradation study)**

| Study[a] | Soil/Condition | $DT_{50}$ at 20°C (days) | Moisture content (w/w %) | | | $DT_{50}$ at 20°C & 10 kPa[b] (days) | Model |
|---|---|---|---|---|---|---|---|
| | | | At MWHC | During study | At 10 kPa | | |
| DuPont-31501 | Tama 20°C | 2 | 30 | 22.5 | 30 | 1.6 | SFO |

[a] All studies are cited or summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 7, DuPont-40934 EU

[b] Correction factor = $(\theta_{study}/\theta_{pf2})^{0.7}$

### B.8.1.2.2 Field studies

#### *B.8.1.2.2.1 Soil dissipation studies*

**B.8.1.2.2.1/01**

| **Reference:** -- | **Report:** | Zietz, E. (2002); Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10G to bare soil at sites in a typical potato growing region of England – Season 2000<br>**DuPont Report No.:** DuPont-3026<br>**Guidelines:** SETAC Europe (1995) |
|---|---|---|

1. Test material: Oxamyl 10GR
   Lot/Batch #: D1410-394
   Purity: 100 g a.s./kg

**Materials and Methods:**

The field soil dissipation of oxamyl (Vydate 10 GR, granule formulation, 10% oxamyl wt/wt, DPX-D1410-394) was measured following spring application to bare soil at a location near the town of Spalding, Lincolnshire, England. The test site was located in a primary potato production area of England. Soil characteristics are given in Table .

The test site was set up with one control plot and four treated plots (approximate dimensions 55.5 m x 3 m). Each of these five plots was then subdivided into 22 subplots (approximate dimensions 2 m x 2.5 m). The granular test substance, 5.5 kg a.s./ha (55 kg product/ha), was applied directly to the soil surface of the four treated plots using a ground-directed granule applicator, then incorporated to approximately 10 cm depth using a rotary cultivator. At predetermined timepoints up to approximately one year after application, one subplot from each of the five larger plots was sampled to 90 cm depth, except day 0 when sampling was limited to 30 cm. Five soil cores were collected from randomly selected subplots using a zero contamination hydraulic coring system with polyethylene liners. The day-0 sample cores were divided into 0-15 cm and 15-30 cm segments. Cores from all additional sampling intervals were split into 0-15 cm, 15-30 cm, 30-45 cm, 45-60 cm, 60-75 cm, and 75-90 cm segments.

At each sampling interval, soil samples from the same depth segment and plot were composited for analysis. All field samples were frozen within 4 hours of sampling and were stored under frozen conditions at ≤18 □C prior to extraction and analysis. Soil samples were analysed for oxamyl and IN-A2213 using organic solvent extraction followed by LC/MS/MS. The method quantitation limits (LOQ) were 0.005 mg/kg for both analytes. The detection limits (LOD) were approximately 0.001 mg/kg for oxamyl and IN-A2213. The LOQ and LOD were both <0.5% of the theoretical initial soil concentration assuming uniform soil incorporation of 5.5 kg a.s./ha to a depth of 10 cm in soil with a bulk density of 1.5 g/cm3. Samples were analysed for IN-D2708 using a separate analytical method. The method for determination of IN-D2708 in soil was validated by analysis of fortified and unfortified control samples at two levels on two different days using a method based on LC-MS. The limit of quantitation (LOQ) was 0.01 mg/kg for IN-D2708.

DT50 and DT90 values were estimated from the residue data using a non-linear regression of conventional first order kinetic equations, performed with the software package ModelManager®, version 1.1 (Cherwell Scientific Limited, 1999). A detailed description of the equations and calculations involved has been given in the description of the main laboratory degradation rate study (report no. DuPont-2957), as reported in section B.8.1.2.1.

**Table 50 Soil characteristics of test site near Spalding, Lincolnshire, England**

| Parameter | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| Soil cores density ($g/cm^3$)[1] | 1.18 | 1.68 | 1.57 | 1.50 | 1.49 | 1.34 |
| pH (1:5) in 0.01M $CaCl_2$ | 7.3 | 7.4 | 7.6 | 7.6 | 7.7 | 7.6 |
| Cation exchange capacity (mEq/100 g) | 1.80 | 1.40 | 0.87 | 0.93 | 1.93 | 2.95 |
| % organic carbon | 1.4 | 1.1 | 0.8 | 0.8 | 0.9 | 1.2 |
| 0-bar moisture (% w/w) | 42.6 | 41.5 | 41.2 | 41.1 | 50.0 | nd |
| % sand (2 mm - 63 μm) | 23.4 | 32.0 | 30.4 | 36.6 | 16.7 | 6.0 |
| % silt (63 μm - 2 μm) | 66.1 | 59.0 | 60.8 | 56.3 | 54.4 | 54.8 |
| % clay (<2 μm) | 10.6 | 9.0 | 8.8 | 7.2 | 28.9 | 39.3 |
| USDA texture[2] | silt loam | silt loam | silt loam | silt loam | silty clay loam | silty clay loam |
| Particle density ($g/cm^3$) | 2.57 | 2.59 | 2.66 | 2.6 | 2.6 | - |

[1] Average bulk density values for soil samples analysed for oxamyl, IN-A2213 and IN-D2708. The values were based on the freeze-dried dried soil sample weights and sampler volume.
[2] USDA texture determined from the % sand, silt and clay reported. USDA particle size classes (sand = 2 mm – 50 μm, silt = 50 μm - 2 μm, clay <2 μm) vary slightly from those reported.

**Findings:**

**Field information is summarised in**

Table 51.

**Table 51 Description of the field dissipation study conditions**

| Test site | Cropping scenario | Application rate (g a.s./ha) | Date of application | Air temperature (°C) | | | Cumulative rainfall (mm) |
|---|---|---|---|---|---|---|---|
| Near Spalding, Lincolnshire, England | Bare ground | 5500 | 18 May 2000 | min 10.7 | avg 15.2 | max 19.7 | 188 |

Climatic data calculated from the daily weather data given in the report. Air temperature min = average minimum daily temperature, max = average maximum daily temperature, and avg = mean of the average daily temperature, calculated as (daily max + daily min)/2, over the sampling period from 18 May 2000 (day 0) to 18 September 2000 (day 123). Cumulative rainfall is reported for the same period. Data obtained from Kirton weather station located 17.5 km from the field site.

**Oxamyl was present in topsoil, 0-15 cm, at an average concentration of 1.6 mg/kg immediately after and declined to <0.005 mg/kg (LOQ) by day 92 (**

Table ). The average day-0 recovery of oxamyl in soil samples was low (55%, range of 25% to 97% in the four treated plots). The low day-0 recoveries are attributed to the difficulty in obtaining representative day-0 soil samples after incorporation of the granules in the silt loam soil on the day of application. Evaluation of the equipment calibration, application monitoring devices, sample homogenisation and sample analysis data supports this conclusion. The application monitors had as much as 29% variability between them. It is believed that the subsequent incorporation to approximately 9.5 cm by roto-tilling, further increased variability by spreading the formulated material throughout a width greater than the application band.

Oxamyl was initially soil incorporated to approximately 10 cm depth on application day. While the majority of the oxamyl mass remained in the upper core segments, minor levels of oxamyl and IN-A2213 were observed in the deepest core segment sampled, 75-90 cm, in some subplots. The maximum average oxamyl concentration of 0.0096 mg/kg was observed in the 75-90 cm segment on day 21. This average value is above the quantitation limit of 0.005 mg/kg. Average IN-A2213 residues were also above the quantitation limit (0.005 mg/kg) in the 75-90 cm segment with a maximum average concentration of 0.0096 mg/kg on day 21. Samples were not analysed for IN-D2708 below 60 cm because residue levels were below the quantitation limit of 0.01 mg/kg in the 45-60cm segment.

IN-A2213 reached a maximum of 0.27 mg/kg by day 9 in the 0-15 cm segment and declined to <0.005 mg/kg by day 92 (Table ). Based on the total mass of IN-A2213 in the soil profile (Table ), and correcting for molecular weight, IN-A2213 reached a maximum of 12% (day 13) of the target mass of applied oxamyl (5.5 kg/ha, day 0). Expressed as a percentage of the initial mass of oxamyl observed in soil (2.8 kg/ha, day 0) the maximum IN-A2213 level was 24%. IN-D2708 reached a maximum of 10% (day 13) of the target mass of applied oxamyl (5.5 kg/ha, day 0). Expressed as a percentage of the initial mass of oxamyl observed in soil (2.8 kg/ha, day 0) the maximum IN-D2708 level was 20%.

The total mass of oxamyl in the soil profile at each sampling time was calculated in terms of kg/ha (Table ). A non-linear first order regression technique was used to calculate the oxamyl $DT_{50}$ and $DT_{90}$ values using this data. The $DT_{50}$ was 11.0 days and the $DT_{90}$ was 36.0 days (Table ). The $DT_{50}$ and $DT_{90}$ values for IN-A2213 were calculated in the same manner as for oxamyl. The non-linear first-order regression technique used simultaneous optimisation to derive the best model fit for oxamyl, IN-A2213 and IN- D2708. The $DT_{50}$ values for IN-A2213 and IN-D2708 were 4.5 and 3.4 days respectively. The $DT_{90}$ values for IN-A2213 and IN-D2708 were 15.0 and 11 days respectively (Table ).

**Table 52 Average (n = 4) dry weight soil concentration of oxamyl in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 1.6 | 0.0015 | ns | ns | ns | ns |
| 1 | 1.5 | 0.0020 | 0.0025 | na | na | na |
| 3 | 1.4 | 0.0025 | 0.0015 | na | na | na |
| 5 | 1.1 | 0.0025 | 0.0020 | na | na | na |
| 7 | 0.81 | 0.0010 | 0.0005 | na | na | na |
| 9 | 0.79 | 0.0049 | 0.0020 | 0.0020 | na | na |
| 13 | 0.62 | 0.13 | 0.017 | 0.0034 | 0.0025 | 0.0025 |
| 21 | 0.34 | 0.084 | 0.021 | 0.015 | 0.0080 | 0.0096 |
| 28 | 0.19 | 0.047 | 0.023 | 0.014 | 0.0061 | 0.0076 |
| 47 | 0.032 | 0.0089 | 0.0044 | 0.0025 | na | na |
| 60 | 0.010 | 0.0033 | 0.0053 | 0.019 | 0.014 | 0.0062 |
| 92 | 0.0025 | 0.0025 | 0.0020 | na | na | na |
| 123 | 0.0025 | 0.0025 | 0.0025 | na | na | na |

na = not analysed, ns = no samples collected
Only 0-15 cm and 15-30 cm cores were collected on application day. The limit of quantitation (LOQ) was 0.005 mg/kg for oxamyl.
Analytical results from individual plots that were <LOQ were assigned a value of 0.1 to 0.5 times the LOQ for the calculation of average concentration.#

**Table 53 Average (n = 4) dry weight soil concentration of IN-A2213 in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 0.12 | 0.0010 | ns | ns | ns | ns |
| 1 | 0.15 | 0.0020 | 0.0010 | na | na | na |
| 3 | 0.22 | 0.0015 | 0.0005 | na | na | na |
| 5 | 0.23 | 0.0020 | 0.0005 | na | na | na |
| 7 | 0.21 | 0.0025 | 0.0005 | na | na | na |
| 9 | 0.27 | 0.0025 | 0.0020 | 0.0005 | na | na |
| 13 | 0.20 | 0.048 | 0.0062 | 0.0020 | 0.0025 | 0.0025 |
| 21 | 0.16 | 0.027 | 0.0081 | 0.0062 | 0.0038 | 0.0096 |
| 28 | 0.095 | 0.013 | 0.011 | 0.0097 | 0.0048 | 0.0037 |
| 47 | 0.012 | 0.0025 | 0.0025 | 0.0020 | na | na |
| 60 | 0.006 | 0.0025 | 0.0041 | 0.016 | 0.0091 | 0.0047 |
| 92 | 0.0025 | 0.0025 | 0.0025 | na | na | na |
| 123 | 0.0025 | 0.0025 | 0.0025 | na | na | na |

na = not analysed, ns = no samples collected
Only 0-15 cm and 15-30 cm cores were collected on application day. The LOQ was 0.005 mg/kg for IN-A2213. Analytical results from individual plots that were <LOQ were assigned a value of 0.1 to 0.5 times the LOQ for the calculation of average concentration.

**Table 54 Average (n = 4) dry weight soil concentration of IN-A2213 as oxamyl equivalents in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 0.16 | 0.0014 | ns | ns | ns | ns |
| 1 | 0.21 | 0.0027 | 0.0014 | na | na | na |
| 3 | 0.30 | 0.0020 | 0.0007 | na | na | na |
| 5 | 0.31 | 0.0027 | 0.0007 | na | na | na |
| 7 | 0.28 | 0.0034 | 0.0007 | na | na | na |
| 9 | 0.36 | 0.0034 | 0.0027 | 0.0007 | na | na |
| 13 | 0.27 | 0.064 | 0.0083 | 0.0027 | 0.0034 | 0.0034 |
| 21 | 0.22 | 0.037 | 0.011 | 0.0083 | 0.0051 | 0.013 |
| 28 | 0.13 | 0.018 | 0.0151 | 0.013 | 0.0065 | 0.0049 |
| 47 | 0.0016 | 0.0034 | 0.0034 | 0.0027 | na | na |
| 60 | 0.0076 | 0.0034 | 0.0055 | 0.021 | 0.012 | 0.0063 |
| 92 | 0.0034 | 0.0034 | 0.0034 | na | na | na |
| 123 | 0.0034 | 0.0034 | 0.0034 | na | na | na |

na = not analysed, ns = no samples collected
Only 0-15 cm and 15-30 cm cores were collected on application day.
The averaged values of IN-A2213 were converted to oxamyl equivalents. This was achieved by multiplying the averaged IN-A2213 soil concentration by the molecular mass of oxamyl and then dividing by the molecular mass of IN-A2213.

**Table 55 Average (n = 4) dry weight soil concentration of IN-D2708 in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 0.010 | 0.0025 | ns | ns | ns | ns |
| 1 | 0.021 | 0.0025 | na | na | na | na |
| 3 | 0.061 | 0.0025 | na | na | na | na |
| 5 | 0.089 | 0.0025 | na | na | na | na |
| 7 | 0.12 | 0.0025 | na | na | na | na |
| 9 | 0.14 | 0.0025 | na | na | na | na |
| 13 | 0.13 | 0.026 | 0.0038 | na | na | na |
| 21 | 0.07 | 0.025 | 0.0050 | 0.0025 | na | na |
| 28 | 0.031 | 0.0044 | na | na | na | na |
| 47 | 0.0070 | 0.0025 | na | na | na | na |
| 60 | 0.0025 | 0.0025 | na | na | na | na |
| 92 | 0.0025 | 0.0025 | na | na | na | na |
| 123 | 0.0025 | 0.0025 | na | na | na | na |

na = not analysed, ns = no samples collected

Only 0-15 cm and 15-30 cm cores were collected on application day. TheLOQ was 0.01 mg/kg for IN-D2708. Analytical results from individual plots that were <LOQ were assigned a value of 0.1 to 0.5 times the LOQ for the calculation of average concentration.

**Table 5639Average (n = 4) dry weight soil concentration of IN-D2708 as oxamyl equivalents in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 0.019 | 0.0047 | ns | ns | ns | ns |
| 1 | 0.039 | 0.0047 | na | na | na | na |
| 3 | 0.11 | 0.0047 | na | na | na | na |
| 5 | 0.17 | 0.0047 | na | na | na | na |

| | | | | | | |
|---|---|---|---|---|---|---|
| 7 | 0.22 | 0.0047 | na | na | na | na |
| 9 | 0.27 | 0.0047 | na | na | na | na |
| 13 | 0.24 | 0.0048 | 0.0070 | na | na | na |
| 21 | 0.13 | 0.0047 | 0.0094 | 0.0047 | na | na |
| 28 | 0.058 | 0.0082 | na | na | na | na |
| 47 | 0.013 | 0.0047 | na | na | na | na |
| 60 | 0.0047 | 0.0047 | na | na | na | na |
| 92 | 0.0047 | 0.0047 | na | na | na | na |
| 123 | 0.0047 | 0.0047 | na | na | na | na |

na = not analysed, ns = no samples collected
Only 0-15 cm and 15-30 cm cores were collected on application day.
A similar calculation was used to convert IN-D2708 to oxamyl equivalents as that described for the conversion of IN-A2213.

**Table 57 Total mass of oxamyl, IN-A2213 and IN-D2708 in the soil profile after application of 5.5 kg oxamyl/ha**

| Sampling date | Days after application | Oxamyl (kg/ha) | (%) | IN-A2213 (kg/ha) | (%) | IN-D2708 (kg/ha) | (%) |
|---|---|---|---|---|---|---|---|
| 18 May 2000 | 0 | 2.8 | (51.0) | 0.28 | (5.1) | 0.046 | (0.83) |
| 19 May 2000 | 1 | 2.6 | (47.2) | 0.37 | (6.7) | 0.08 | (1.5) |
| 21 May 2000 | 3 | 2.5 | (45.5) | 0.53 | (9.6) | 0.21 | (3.8) |
| 23 May 2000 | 5 | 1.9 | (34.5) | 0.55 | (10.0) | 0.31 | (5.6) |
| 25 May 2000 | 7 | 1.4 | (25.0) | 0.50 | (9.0) | 0.41 | (7.5) |
| 27 May 2000 | 9 | 1.4 | (25.0) | 0.66 | (12.0) | 0.49 | (9.0) |
| 31 May 2000 | 13 | 1.5 | (27.0) | 0.66 | (12.0) | 0.56 | (10.0) |
| 8 June 2000 | 21 | 0.92 | (16.7) | 0.57 | (10.3) | 0.39 | (7.1) |
| 15 June 2000 | 28 | 0.58 | (10.5) | 0.36 | (6.5) | 0.12 | (2.2) |
| 04 July 2000 | 47 | 0.089 | (1.6) | 0.050 | (1.0) | 0.035 | (0.63) |
| 17 July 2000 | 60 | 0.11 | (2.0) | 0.10 | (1.8) | 0.020 | (0.36) |
| 18 Aug. 2000 | 92 | 0.015 | (0.27) | 0.023 | (0.4) | 0.020 | (0.36) |
| 18 Sept. 2000 | 123 | 0.017 | (0.30) | 0.023 | (0.4) | 0.020 | (0.36) |

An average soil bulk density was calculated for each 15 cm depth segment based on the dried sample weight and sampler volume. These average bulk density values were used in the total mass calculations.
% = amount of substance as a percentage of applied oxamyl

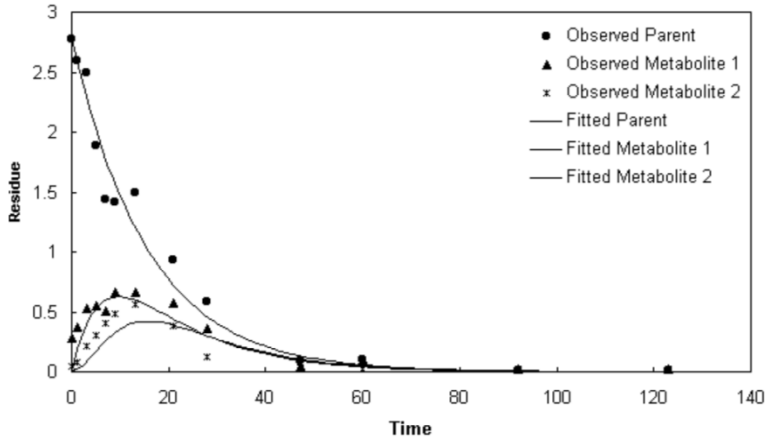
The residue data for oxamyl, oxamyl oxime and DMOA in the soil profile were used to calculate the first order $DT_{50}$ and $DT_{90}$ values of the parent and the degradates.
The first order $DT_{50}$ and $DT_{90}$ values for the test field in Spalding are summarized in the Table 58 and a figure is presented below to show the dissipation curve derived from the average residue data of four plots.

**Table 58 $DT_{50}$ and $DT_{90}$ values for oxamyl, IN-A2213 and IN-D2708 in field soil near Spalding, Lincolnshire, England**

| Substance | $M_0$, $C_1$, $C_2$ | k ($days^{-1}$) | $DT_{50}$ (days) | $DT_{90}$ (days) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| silt loam, pH 7.3 | | | | | | |
| oxamyl | $M_0$ = 2.8<br>$C_1$ = 1 | $k_p$ = 0.0643 | 10.76 | 35.76 | 0.969 | Non-linear regression of integrated rate equations based on simple first order kinetics, with parent and two metabolites considered in series. |
| IN-A2213 | $C_2$ = 1 | $k_1$ = 0.1541 | 4.49 | 14.93 | | |
| IN-D2708 | - | $k_2$ = 0.2019 | 3.43 | 11.40 | | |

$M_0$ is the estimated concentration of oxamyl at time t = 0, $C_1$ is the fraction of oxamyl forming IN-A2213, $C_2$ is the fraction of IN-A2213 forming IN-D2708, $k_p$ is the first order degradation rate constant for oxamyl, $k_1$ is the first order degradation rate constant for IN-A2213 and $k_2$ is the first order degradation rate constant for IN-D2708.

**Figure Dissipation curve of oxamyl (parent) and the degradates oxamyl oxime (met) and DMOA (metabolite 2)**




**Conclusions:**

Average recovery of oxamyl on day 0 was low (55% of applied, with a range of 25% to 97% in the four treated plots). This was attributed to a variable application that was further complicated by incorporation. The application monitors had as much as 29% variability between them. It is believed that the subsequent incorporation to approximately 9.5 cm, by roto-tilling, further increased variability by spreading the formulated material throughout a width greater than the application band.

Oxamyl degraded readily ($DT_{50}$ = 11.0 days) in silt loam soil of pH 7.3, following spring application (18 May) to a non-cropped field. The degradation products IN-A2213 and IN- D2708 also readily degraded ($DT_{50}$ values of 4.5 and 3.4 days respectively).

Average oxamyl residues observed in the 75-90 cm depth segment were above the quantitation limit (0.005 mg/kg) and reached a maximum of 0.0096 mg/kg on day 21. Average IN-A2213 levels exceeded the quantitation limit (0.005 mg/kg) in the 75-90 cm segment at one sampling time only (day 21), and reached a maximum of 0.0096 mg/kg. Samples were not analysed for IN-D2708 residues below 60 cm, as they were below the quantitation limit of 0.01 mg/kg for IN-D2708 at this depth.

The soil dissipation study DuPont-3026, originally submitted under EU Rev8 Point IIA 7.1.1.2.2.1 and conducted with test material Oxamyl 10GR, was conducted under guideline SETAC Europe (1995). A review of this study indicates that it meets the current guidelines EU 1607/VI/97 rev. 1 (1997), EU 7029/VI/95 rev. 5 (1997), SETAC Europe (1995), SANCO/3029/99 rev. 4 (2000) and is relied upon. It is noted that the range of recoveries at day 0 were lower than the target for certain fields, likely due attempts to incorporate the granular test item in to the top soil. However, the amount recovered at day 0 was still a high enough concentration to allow for the dissipation of oxamyl and its major metabolites to be tracked as soil column concentrations were well above the LOD of the analytical method. Therefore, the study is reliable.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Oxamyl dissipates steadily from soil under field conditions. Based on average soil concentrations, the $DT_{50}$ values were 11 days for oxamyl, 5 days for oxamyl oxime and 3 days for DMOA.

**Study submitted in the EU Dossier in 2003 and included in the first EU approval review.**

**B.8.1.2.2.1/02**

| **Reference:** -- | **Report:** | Mol, J.G.J. (2002); Field soil dissipation study of oxamyl in The Netherlands<br>**DuPont Report No.:** DuPont-2815<br>**Guidelines:** SETAC Europe (1995) |
|---|---|---|

1. Test material: Oxamyl 10GR
   Lot/Batch #: D1410-395
   Purity: 100 g a.s./kg

**Material and Methods:**

The field soil dissipation of oxamyl (Vydate 10 GR, granule formulation, 10% oxamyl wt/wt, DPX-D1410-395) was measured following spring application to bare soil at a location near the town of Ottersum, The Netherlands. The test site was representative of typical potato production areas of The Netherlands. Soil characteristics are given in Table .

The test site was set up with one control plot and three treated plots (approximate dimensions 40.5 m x 3 m). Each of these four plots was then subdivided into 20 subplots (approximate dimensions 2 m x 3 m). The granular test substance, 4 kg a.s./ha (40 kg product/ha), was applied directly to the soil surface of the three treated plots using a modified seed drill, then incorporated to approximately 20 cm depth using a rotary cultivator. At predetermined time points up to 1.4 years after application, one subplot from each of the four larger plots was sampled to 90 cm depth, except for day 0 when sampling was limited to 30 cm. Five soil cores were collected from randomly selected subplots using a zero contamination Humax coring system with acetate liners. On day 0, two subplots per larger plot (10 cores total per plot) were sampled to better represent the incorporated granule distribution. The day 0 sample cores were divided into 0-15 cm and 15-30 cm segments. Cores from all additional sampling intervals were split into 0-15 cm, 15-30 cm, 30-45 cm, 45-60 cm, 60-75 cm, and 75-90 cm segments.

At each sampling interval, soil samples from the same depth segment and plot were composited for analysis. All field samples were frozen within 4 hours of sampling and were stored under frozen conditions at ≤18°C prior to extraction and analysis. Soil samples were analysed for oxamyl and IN-A2213 using organic solvent extraction followed by LC/MS/MS. The method quantitation limits (LOQ) and detection limits (LOD) were approximately 0.005 mg/kg and 0.001 mg/kg, respectively, for both analytes. The LOQ and LOD were both <0.5% of the initial soil concentration assuming uniform soil incorporation of 4 kg a.s./ha to a depth of 20 cm in soil with bulk density of 1.5 g/cm3. For DMOA, a separate method using HPLC with single MS detection was used. Both methods were validated before use and met the criteria as laid down in EU guidelines, i.e. average recovery 70-110%, RSD <20%.

DT50 and DT90 values were estimated from the residue data using a non-linear regression of conventional first order kinetic equations, performed with the software package ModelManager®, version 1.1 (Cherwell Scientific Limited, 1999). A detailed description of the equations and calculations involved has been given in the description of the main laboratory degradation rate study (report no. DuPont-2957), as reported in section B.8.1.2.1.

**Table 59 Soil characteristics of test site near Ottersum, The Netherlands**

| **Parameter** | **0-15 cm** | **15-30 cm** | **30-45 cm** | **45-60 cm** | **60-75 cm** | **75-90 cm** |
|---|---|---|---|---|---|---|
| pH (1:5) in water | 6.6 | 6.9 | 6.4 | 5.9 | 5.3 | 5.5 |
| pH (1:5) in 0.01M $CaCl_2$ | 6.7 | 6.4 | 5.8 | 4.8 | 4.4 | 4.5 |
| pH (1:5) in 1M KCl | 6.7 | 6.4 | 5.8 | 4.8 | 4.4 | 4.5 |
| Cation exchange capacity (mEq/100 g) | 1.1 | <0.05 | 1.3 | 0.2 | <0.05 | <0.05 |
| % organic carbon | 2.0 | 1.8 | 1.0 | 0.7 | 0.9 | 1.0 |
| 0-bar moisture (%) | 45.5 | 36.7 | 32.4 | 28.3 | 26.8 | 24.4 |
| % sand (2 mm - 63 μm) | 57.3 | 57.4 | 55.6 | 71.1 | 80.7 | 83.9 |
| % silt (63 μm - 2 μm) | 26.2 | 24.7 | 24.8 | 16.6 | 10.5 | 9.2 |

| | | | | | | |
|---|---|---|---|---|---|---|
| % clay (<2 μm) | 16.4 | 17.9 | 19.5 | 12.3 | 8.8 | 6.9 |
| USDA texture[1] | sandy loam | sandy loam | sandy loam | loamy sand | loamy sand | loamy sand |
| Dry matter content (%m/m) (of air-dried soil) | 99.2 | 99.3 | 99.2 | 99.4 | 99.6 | 99.5 |
| Water content (%m/m) (of air-dried | 0.9 | 0.7 | 0.9 | 0.6 | 0.4 | 0.5 |
| Dry matter content (%m/m) (of field-moist | 86.7 | 83.6 | 85.2 | 89.8 | 89.4 | 91.6 |
| Water content (%m/m)[2] (of field- | 15.4 | 19.7 | 17.4 | 11.3 | 11.9 | 9.2 |

[1] USDA texture determined from the % sand, silt and clay reported. USDA particle size classes (sand = 2 mm – 50 μm, silt = 50 μm - 2μm, clay <2 μm) vary slightly from those reported.
[2] Dry weight basis

**Findings:**

Field information is summarised in Table 60.

**Table 60 Description of field dissipation study conditions**

| Test site | Cropping scenario | Application rate (g a.s./ha) | Date of application | Air temperature (°C) | | | Cumulative rainfall (mm) |
|---|---|---|---|---|---|---|---|
| Near Ottersum, The Netherlands | Bare ground | 4000 | 05 May 1999 | min 9.9 | avg 14.8 | max 20.1 | 427 |

Climatic data calculated from the daily weather data given in the report. Air temperature min = average minimum temperature, max = average maximum temperature, and avg = mean of the average daily temperature over the sampling period from 05 May 1999 (day 0) to 15 Nov 1999 (day 194). Cumulative rainfall is reported for the same period.

Oxamyl was present in topsoil, 0-15 cm, at an average concentration of 1.7 mg/kg immediately after application and declined to at or below 0.001 mg/kg (LOD) by day 100 (Table ). IN-A2213 reached a maximum of 0.062 mg/kg by day 12 in the 0-15 cm segment and declined to <0.001 mg/kg by day 70 (Table ). Based on the total mass of IN-A2213 in the soil profile (Table ), IN-A2213 reached a maximum of ~6% (day 5) of the target mass of applied oxamyl (4.0 kg/ha, day 0). IN-D2708 reached a maximum of 19% (day 16) of the target mass of applied oxamyl (4.0 kg/ha, day 0).

Oxamyl was not detected below the 30-45 cm segment following soil incorporation of oxamyl to approximately 20 cm on application day. Average oxamyl concentrations exceeded LOD (0.001 mg/kg), but remained less than the LOQ (0.005 mg/kg) on days 5 and 16 in the 30-45 cm zone. IN-A2213 was not detected below the 15-30 cm depth segment. Average IN-A2213 residues exceeded the LOD in the 15-30 cm segment only on day 0. IN-D2708 was not detected below the 0-15 cm depth segment.

The total amount of oxamyl in the soil profile at each sampling time was calculated in terms of kg/ha (Table ).

**Table 61 Average (n = 3) dry weight soil concentration of oxamyl in each sample horizon (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 1.7 | 0.031 | ns | ns | ns | ns |
| 5 | 0.94 | 0.28 | 0.0013 | <LOD | <LOD | <LOD |
| 12 | 0.66 | 0.0017 | <LOD | <LOD | <LOD | <LOD |
| 16 | 0.58 | 0.0041 | 0.0010 | <LOD | <LOD | <LOD |
| 27 | 0.20 | 0.0018 | <LOD | <LOD | <LOD | <LOD |
| 40 | 0.081 | 0.0015 | <LOD | <LOD | <LOD | <LOD |
| 70 | 0.0016 | <LOD | <LOD | <LOD | <LOD | <LOD |
| 100 | <LOD | <LOD | na | na | na | na |
| 134 | 0.0010 | <LOD | na | na | na | na |
| 160 | 0.0010 | <LOD | na | na | na | na |

| | | | | | | |
|---|---|---|---|---|---|---|
| 194 | <LOD | <LOD | na | na | na | na |

na = not analysed (due to the absence of detectable residues in the segment above), ns = no samples collected Only 0-15 cm and 15-30 cm cores were collected on application day. The limit of quantification (LOQ) and the limit of detection (LOD) for oxamyl were respectively 0.005 mg/kg and 0.001 mg/kg.

**Table 62 Average (n = 3) dry weight concentration of IN-A2213 in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 0.048 | 0.0013 | ns | ns | ns | ns |
| 5 | 0.078 | <LOD | <LOD | <LOD | <LOD | <LOD |
| 12 | 0.062 | <LOD | <LOD | <LOD | <LOD | <LOD |
| 16 | 0.065 | <LOD | <LOD | <LOD | <LOD | <LOD |
| 27 | 0.035 | <LOD | <LOD | <LOD | <LOD | <LOD |
| 40 | 0.0095 | <LOD | <LOD | <LOD | <LOD | <LOD |
| 70 | <LOD | <LOD | <LOD | <LOD | <LOD | <LOD |
| 100 | <LOD | <LOD | na | na | na | na |
| 134 | <LOD | <LOD | na | na | na | na |
| 160 | <LOD | <LOD | na | na | na | na |
| 194 | <LOD | <LOD | na | na | na | na |

na = not analysed, ns = no samples collected
Only 0-15 and 15-30 cm cores were collected on application day. The LOQ and LOD for IN-A2213 were respectively 0.005 mg/kg and 0.001 mg/kg.

**Table 63 Average (n = 3) dry weight soil concentration of IN-D2708 in each sample depth segment (mg/kg)**

| Days after application | 0-15 cm | 15-30 cm | 30-45 cm | 45-60 cm | 60-75 cm | 75-90 cm |
|---|---|---|---|---|---|---|
| 0 | 0.069 | <LOD | ns | ns | ns | ns |
| 5 | 0.094 | <LOD | na | na | na | na |
| 12 | 0.13 | <LOD | na | na | na | na |
| 16 | 0.17 | <LOD | na | na | na | na |
| 27 | 0.14 | <LOD | na | na | na | na |
| 40 | 0.014 | ns | na | na | na | na |
| 70 | <LOD | <LOD | na | na | na | na |
| 100 | <LOD | <LOD | na | na | na | na |
| 134 | <LOD | <LOD | na | na | na | na |
| 160 | <LOD | ns | na | na | na | na |
| 194 | <LOD | ns | na | na | na | na |

na = not analysed, ns = no samples collected
Only 0-15 and 15-30 cm cores were collected on application day. The LOQ and LOD for IN-D2708were respectively 0.01 mg/kg and 0.005 mg/kg.

**Table 64 Total mass (kg/ha) of oxamyl, IN-A2213 and IN-D2708 as Oxamyl equivalent in the soil profile after application of 4.0 kg oxamyl/ha**

| Sampling date | Days after application | Oxamyl (kg/ha) | (%) | IN-A2213 (kg/ha) | (%) | IN-D2708 (kg/ha) | (%) |
|---|---|---|---|---|---|---|---|
| 05 May 1999 | 0 | 3.8 | (95.0) | 0.15 | (3.8) | 0.30 | (7.5) |
| 10 May 1999 | 5 | 2.1 | (52.5) | 0.25 | (6.3) | 0.41 | (10.3) |
| 17 May 1999 | 12 | 1.5 | (37.5) | 0.20 | (5.0) | 0.55 | (13.8) |
| 21 May 1999 | 16 | 1.3 | (32.5) | 0.21 | (5.3) | 0.75 | (18.8) |
| 01 June 1999 | 27 | 0.47 | (12.0) | 0.12 | (3.0) | 0.62 | (15.5) |
| 14 June 1999 | 40 | 0.19 | (5.0) | 0.037 | (0.93) | 0.071 | (1.8) |
| 14 July 1999 | 70 | 0.0092 | (0.23) | 0.0096 | (0.24) | 0.021 | (0.53) |

| | | | | |
|---|---|---|---|---|
| 13 Aug. 1999 | 100 | 0.0026 (0.065) | 0.0030 (0.08) | 0.021 (0.53) |
| 16 Sept. 1999 | 134 | 0.0034 (0.085) | 0.0030 (0.08) | 0.011 (0.28) |
| 12 Oct. 1999 | 160 | 0.0034 (0.085) | 0.0030 (0.08) | 0.011 (0.28) |
| 16 Nov. 1999 | 194 | 0.0022 (0.055) | 0.0030 (0.08) | 0.011 (0.28) |

% = amount of substance as a percentage of theoretical applied oxamyl A soil bulk density of 1.5g/cm$^3$ was assumed for all depth segments.

A non-linear first-order regression technique was used to calculate the oxamyl $DT_{50}$ and $DT_{90}$ values using this data. The $DT_{50}$ was 9.2 days and the $DT_{90}$ was 30.7 days (**Errore. L'autoriferimento non è valido per un segnalibro.**). The $DT_{50}$ and $DT_{90}$ values for IN-A2213 and IN-D2708 were calculated in the same manner as for oxamyl. The non-linear first-order regression technique used simultaneous optimisation to derive the best model fit for oxamyl, IN-A2213 and IN-D2708. The $DT_{50}$ values for IN-A2213 and IN-D2708 were 1.7 and 6.7 days respectively. The $DT_{90}$ values for IN-A2213 and IN-D2708 were 5.6 and 22 days respectively (**Errore. L'autoriferimento non è valido per un segnalibro.**).

**Figure Decline curves for oxamyl (Parent), oxamyl oxime (Metabolite 1) and DMOA (Metabolite 2)**

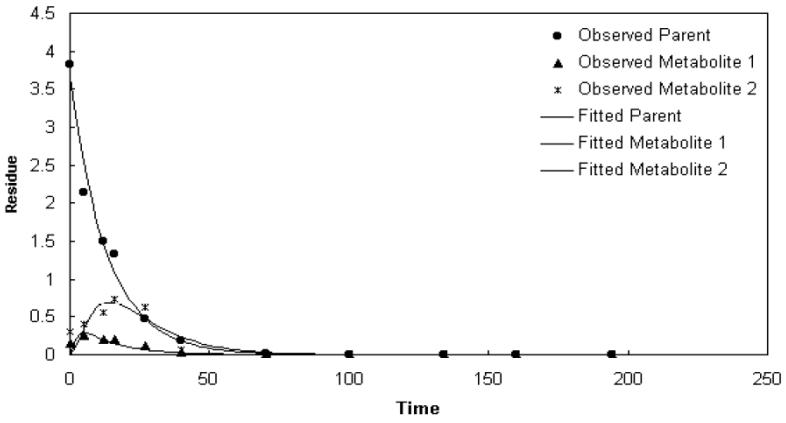



**Table 65 $DT_{50}$ and $DT_{90}$ values for oxamyl, IN-A2213 and IN-D2708 in field soil near Ottersum, Limburg, The Netherlands**

| Substance | $M_0$, $C_1$, $C_2$ | k (days$^{-1}$) | DT50 (days) | DT90 (days) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| sandy loam, pH 6.6 | | | | | | |
| oxamyl | $M_0$ = 3.65<br>$C_1$ = 0.626 | $k_p$ = 0.0750 | 9.24 | 30.70 | 0.979 | Non-linear regression of integrated rate equations based on simple first order kinetics, with parent and two metabolites considered in |
| IN- A2213 | $C_2$ = 1 | $k_1$ = 0.4085 | 1.69 | 5.63 | | |
| IN- D2708 | - | $k_2$ = 0.1037 | 6.68 | 22.19 | | |

$M_0$ is the estimated concentration of oxamyl at time t = 0, $C_1$ is the fraction of oxamyl forming IN-A2213, $C_2$ is the fraction of IN-A2213 forming IN-D2708, $k_p$ is the first order degradation rate constant for oxamyl, $k_1$ is the first order degradation rate constant for IN-A2213 and $k_2$ is the first-order degradation rate constant for IN-D2708.

**Conclusions:**

Oxamyl degraded readily ($DT_{50}$ = 9.2 days) in sandy loam soil of pH 6.6, following spring application (05 May) to a non-cropped field. The degradation products IN-A2213 and IN- D2708 also readily degraded ($DT_{50}$ values of 1.7 and 6.7 days respectively).

The soil dissipation study DuPont-2815, originally submitted under EU Rev8 Point IIA 7.1.1.2.2.1 and conducted with test material Oxamyl 10GR, was conducted under guideline SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (EU 1607/VI/97 rev. 1 [1997], EU 7029/VI/95 rev. 5 [1997], SETAC Europe [1995], SANCO/3029/99 rev. 4 [2000]) and is relied upon.

The following greenhouse dissipation study is being presented as confirmatory data, to expand the pool of field dissipation data for oxamyl. This study has been included because the greenhouse results are consistent with those from the traditional bare field studies in rate and metabolite profile, and thus increase the confidence in the

understanding of oxamyl dissipation under actual use conditions. Since oxamyl degrades so quickly, the more controlled, smaller-scale conditions of the greenhouse allow for a more homogeneous application and better tracking of the material, especially the metabolites, and thus allow for more robust pathway kinetics. Lastly, oxamyl has a substantial chemical component to its degradation, so slight variation in soil microbial populations that may have been present between the greenhouse and outdoors do not impact the validity of the dissipation data.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Oxamyl degraded readily ($DT_{50}$ = 9.2 days) under field conditions. The degradation products IN-A2213 and IN-D2708 also readily degraded, $DT_{50}$ values of 1.7 and 6.7 days respectively.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.2.1/03**

| | | |
|---|---|---|
| **Reference:** CA 7.1.2.2.1/01 | **Report:** | LeNoir, J.S., Zietz, E. (2003); Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10 L by means of drip irrigation to cucurbits in a greenhouse in Spain, season 2000<br>**DuPont Report No.:** DuPont-4719<br>**Guidelines:** SETAC Europe (1995)<br>**Deviations:** None<br>**Testing Facility:** DuPont Stine-Haskell Research Center, Newark, Delaware, USA<br>**Testing Facility Report No.:** DuPont-4719<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive Summary**

The soil dissipation of oxamyl following a single chemigation (drip irrigation) application of oxamyl 100 g/L SL (soluble concentrate) to bare ground and cropped plots was studied under greenhouse conditions in Vinalesa, Spain (near Valencia) over the period of one year, beginning in October 2000.

The study was designed to determine the environmental fate and the rate of decline of oxamyl and its major soil degradates oxamyl oxime (IN-A2213) and dimethyl oxalamic acid (DMOA or IN-D2708) following application of Oxamyl 10SL to cropped and non-cropped plots under actual field conditions. Oxamyl, as Oxamyl 10SL, was applied by chemigation once at 1.5 kg a.s./ha in a greenhouse at a representative vegetable production site in Spain. The greenhouse conditions were maintained according to local practice. The bare ground plots were kept in non-cropped condition throughout the study period while cucumbers (*Cucumis sativus*) were grown on the cropped plots. Three treatment plots and one control plot were established within the four separate plastic greenhouses. Each plot was divided into a non-cropped area and an area cropped with cucumbers. Oxamyl was applied once to each of the three plots using a drip irrigation technique; the control plot was not treated. Application monitors were set out at the time of application to verify the amount applied and the results support that the test item was evenly applied *via* the chemigation system.

Soil cores from the treated and control plots were collected randomly to a depth of 90 cm before application (-1 DAT) and after application, 0, 1, 3, 5, 7, 9, 20, 23, 29, 48, 63, 92, 121, 154, and 178 days after treatment (DAT). All cores were collected directly beneath chemigation drip line emitters. At each sampling time, four cores were collected from each of the treated cropped and non-cropped plots for a total of 24 cores. Four cores were collected from each the cropped and non-cropped untreated control for a total of eight cores. Due to the coring principle, each core was segmented during the sampling procedure. The segments corresponded to 0-10 cm, 10–25 cm, 25–40 cm, 40–55 cm, 55–70 cm, and 70–90 cm. Soil cores were frozen within two hours at the field site. Prior to analysis, the soil segments were composited by sampling time, plot, crop cover, and depth (horizon). Samples collected after 178 days (223 and 373 DAT) were not analysed, since quantifiable residues were no longer present and the $DT_{90}$ of each analyte had been reached.

Soil samples were analysed for oxamyl and oxamyl oxime (IN-A2213) residues by TNO Nutrition and Food Research (The Netherlands) according to method DuPont-2392, summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 4, DuPont-40931 EU. The residues were extracted with acidified acetonitrile:methanol (80:20, v/v) by mechanical shaking (at 50°C), evaporated to dryness, reconstituted in acidified methanol:aqueous ammonium acetate (10:90, v/v), and determined by reversed phase HPLC with MS-MS detection, monitoring ion transitions m/z 237→72 for oxamyl and m/z 163→72 for oxamyl oxime using electrospray ionization operating in the positive ion mode. Samples of untreated soil were fortified at 0.005–2.2 mg/kg when the method was validated (prior to field sample analysis) and 0.005–2 mg/kg, when fortified samples were analysed concurrently with the samples from the field. The method validation recoveries averaged 90–103% (RSD 5–17%, n = 16) for oxamyl and 97–107% (RSD 4–12%, n = 16) for oxamyl oxime and the concurrent recoveries averaged 94% (RSD 15%, n = 49) for oxamyl and 90% (RSD 12%, n = 49) for oxamyl oxime. The limit of quantitation (LOQ) for oxamyl and oxamyl oxime residues in soil was 0.005 mg/kg for each analyte.

Soil samples were analysed for the degradate DMOA (IN-D2708) at TNO Nutrition and Food Research (The Netherlands) using an LC/MS method. The DMOA residues were extracted with an aqueous solvent under basic conditions (10 mM ammonium carbonate in water), cleaned up by SPE, and then analysed by LC/MS operating in the negative ion mode monitoring a single MS-SIM. Quantitation was obtained using an internal calibration curve created with a deuterated (d6)-DMOA analytical standard. Samples of untreated soil were fortified with DMOA at 0.01–0.21 mg/kg for validation, with average method validation recoveries of 101–104% (RSD 1–6%, n = 16) and concurrent recoveries averaging 88% (range 78–105%, RSD 8%, N = 41). The LOQ for DMOA residues in soil was 0.01 mg/kg.

In the samples collected immediately after application, oxamyl was observed in the top three soil segments, though the majority of the residue was found in the 0–10 cm segment. The concentration of oxamyl ranged from 2.9–6.2 mg/kg in the 0–10 cm soil depth, 0.53–2.5 mg/kg in the 10–25 cm segment, and <0.005–0.035 mg/kg in the 25–40 cm segment. Oxamyl oxime was also observed in these segments with concentrations ranging from 0.009–0.36 mg/kg. DMOA was observed only in the top two sample segments on application day with concentrations ranging from 0.01–0.08 mg/kg.

The oxamyl residue declined rapidly within the first month. After approximately four weeks (29 DAT), the concentration of oxamyl was less than 0.02 mg/kg in all samples except two of 0.07 and 0.08 mg/kg. No oxamyl residue above the LOQ (0.005 mg/kg) was detected in samples collected later than five months after the application. The soil degradates oxamyl oxime and DMOA also declined within this time period. Oxamyl oxime was only observed above the LOQ (0.005 mg/kg) in five samples collected later than 63 DAT, all in the 0–10 cm segment. DMOA was not observed in any sample above the LOQ (0.010 mg/kg) after 63 days.

Residues of oxamyl and oxamyl oxime were generally not observed below 55 cm. In a few cases, residues above the LOQ (0.005 mg/kg) were detected at the 55–70 cm soil depth at 23–48 days after treatment. Residues above the LOQ were determined in the deepest segment (70–90) cm only once at 48 DAT for oxamyl (0.006 mg/kg) and in three samples (0.007–0.024 mg/kg, 23–48 DAT) for oxamyl oxime. DMOA was not observed above the LOQ (0.010 mg/kg) in any sample below 40 cm. The soil residue data demonstrate that the majority of the oxamyl remained in the top three sample segments (0–40 cm) where degradation to oxamyl oxime and DMOA occurred.

Apparent residues of each analyte were non-detectable in all control soil samples or the untreated soil samples taken before application.

For each sampling time, the residue concentrations of oxamyl, oxamyl oxime, and DMOA in each depth segment were converted to oxamyl equivalents and expressed on a mass/area basis ($\mu g/cm^2$). The resulting mass/area values for each depth segment were summed for the entire sampled soil profile, which provided the total mass of each analyte present in the sampled soil profile at each sampling time. Due to the similarity of the cropped and non-cropped plot data, the average mass/area values (n = 6, average of three cropped and three non-cropped plots) were used in the primary kinetic analysis. A non-linear simple first order regression technique was used to determine the half-lives. The half-lives (first order $DT_{50}$) were 3.3 days for oxamyl, 2.1 days for oxamyl oxime, and 0.52 days for DMOA. The corresponding $DT_{90}$ values were 11 days for oxamyl, 7.0 days for oxamyl oxime, and 1.7 days for DMOA.

The storage period between sampling in the field and analysis did not exceed 9.1 months for all soil samples analysed for oxamyl and oxamyl oxime. The maximum storage interval between the sampling and extraction date for the DMOA degradate was 15.9 months. The freezer storage stability of the residues in soil has been determined (refer to summary for DuPont-9342).

## I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: Oxamyl 10SL
   Lot/Batch #: D1410-417
   Purity: 100 g/L (nominal); 101.3 g a.s./L (measured)
   Description: Dark green liquid
   CAS #: None for the formulation; 23135-22-0 for oxamyl active substance
   Stability of the test compound: Shown to be stable for at least 2 months

2. Test Site

Test site description is detailed in **Errore. L'origine riferimento non è stata trovata.**. Soil samples collected to 90 cm depth were characterized and the soil characterization data are included in

.

**Table 66 Test site description**

| | |
|---|---|
| Location: | Vinalesa, Spain |
| Region: | Valencia |
| GPS Coordinates: | Not reported |
| Representative Crop Region: | Vegetables cultivated in greenhouses; orchards of oranges |
| Site Selection Criteria: | Typical greenhouse with flat and level soil. Soil allowing coring at least down to 90 cm |
| Weather Station: | Not applicable (indoor environmental conditions provided) |
| Pretreatment Exclusion Criteria: | No other chemical of similar structure applied during the past 3 years. |
| Plot History, Crops Grown: | Pepper grown from 1996 to 2000 |
| Pesticides Used in Preceding 3 Years: | Imidacloprid, methamidophos, abamectin, endosulfan, pyrifenox, dimethoate, chlorpyrifos, and dicofol. Methyl bromide and chloropicrin to fumigate the soil were applied in September/October of every year. The last fumigation was carried out in the year 1999. |
| Cultivation Method: | The soil in the greenhouses was cultivated by mechanical tillage and a fine tilth was prepared. |
| Location/Identification of Weather Station: | Not applicable. Climate data recorded inside the greenhouse were reported (daily average temperatures and humidity) for the period of the field phase October 2000 until October 2001. |
| Distance of Weather Station from Test Site: | Not applicable |
| Depth to Ground Water Table: | Not applicable |

**Table 67 Soil properties at the Spain site**

| | Soil depth (cm) | | | | | |
|---|---|---|---|---|---|---|
| **Soil property** | **0-10** | **10-25** | **25-40** | **40-55** | **55-70** | **70-90** |
| Sand % (0.063-2 mm)[a] | 71.7 | 67.7 | 37.1 | 47.8 | 56.9 | 59.0 |
| Silt % (0.002-0.063 mm)[a] | 20.0 | 22.1 | 45.9 | 36.0 | 28.4 | 28.5 |
| Clay % (<0.002 mm)[a] | 8.3 | 10.2 | 17.1 | 16.2 | 14.7 | 12.6 |
| pH (water) | N/A[b] | N/A | N/A | N/A | N/A | N/A |
| pH (0.01M $CaCl_2$) | 7.79 | 7.72 | 7.78 | 7.68 | 7.67 | 7.71 |
| % Total Organic Carbon (TOC)[c] | 2.0 | 1.4 | 1.2 | 1.1 | 1.1 | 1.1 |
| % Organic matter[d] | 3.5 | 2.4 | 2.0 | 1.9 | 1.9 | 1.9 |
| C.E.C [meq/100g][e] | 10.0 | 7.1 | 7.0 | 6.3 | 7.4 | 7.6 |
| Bulk density (g $cm^{-3}$) [f] | 1.93 | - | - | - | - | - |
| Water Holding Capacity (% of dry wt.) | 43.1 | 38.4 | 37.8 | 35.2 | 33.4 | 30.5 |
| Soil Classification[g] | Medium loamy sand | Medium loamy sand | Slight sandy loam | Strong loamy sand | Strong loamy sand | Strong loamy sand |
| Soil Series Name | N/A | N/A | N/A | N/A | N/A | N/A |

[a] Particle size distribution (%)
[b] Not applicable and/or not reported
[c] Organic carbon content [% or g/100 g of dry soil]
[d] TOC × 1.72 (%)
[e] Cation Exchange Capacity (C.E.C)
[f] The density at the original position in the field (horizon 0–12 cm), average of eight spots in the field taken into account
[g] Soil classification according to USDA system

## B. METHODS

1. Experimental design

The experimental details for the test substance application, application rate, application method, etc., are included in **Errore. L'origine riferimento non è stata trovata.**.

2. Soil sampling

Soil sampling intervals, sampling depths, and number of cores collected are listed in **Errore. L'origine riferimento non è stata trovata.**.

3. Description of Analytical Methods

Soil samples were analysed for oxamyl and oxamyl oxime (IN-A2213) residues by TNO Nutrition and Food Research (The Netherlands) according to method DuPont-2392, summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 4, DuPont-40931 EU, modified with respect to the extraction procedure and the chromatographic conditions (method details are included in the study report). The residues were extracted with acidified acetonitrile:methanol (80:20, v/v) by mechanical shaking (at 50°C), evaporated to dryness, reconstituted in acidified methanol:aqueous ammonium acetate (10:90, v/v), and determined by reversed phase HPLC with MS-MS detection monitoring ion transitions m/z 237→72 for oxamyl and m/z 163→72 for oxamyl oxime using electrospray ionization operating in the positive ion mode. The concentrations (mg/kg) of oxamyl and its degradation product oxamyl oxime were quantitated using an external calibration curve of each analyte. The LOQ for oxamyl and oxamyl oxime residues in soil was 0.005 mg/kg each.

Soil samples were analysed separately for the degradate DMOA (IN-D2708) by TNO Nutrition and Food Research using an LC/MS method (method details are included in the study report). The DMOA residues were extracted with an aqueous solvent under basic conditions (10 mM ammonium carbonate in water), cleaned up by SPE charcoal (ENVI-carb®), and then analysed by LC/MS using anion exchange column and mass spectrometric detection using single MS-SIM in the negative mode (m/z 116 for DMOA and 122 for d6-DMOA). Quantitation was obtained using an internal calibration curve created with a deuterated (d6)-DMOA analytical standard. The LOQ for DMOA residues in soil was 0.01 mg/kg.

The concentrations (mg/kg) of oxamyl and its degradation products in treated field soil samples were calculated on a dry weight basis.

**Table 68 Experimental design, plot set up, and application details for the Spain site**

| Details | Vinalesa, Valencia, Spain (Greenhouse) |
|---|---|
| Duration of study | 178 days |
| Uncropped (bare) or cropped | Bare (maintained weed free) and Cropped (cucumber) |
| Controls used | Yes |
| Number of plot(s) | Three treated (replicates DA, DB, and DC) and one untreated control (DX), each divided into non-cropped and cropped areas, and each in a separate greenhouse. |
| Treated plot dimensions | 46.5 × 1.6 m (The cropped and the non-cropped areas were congruent with each other and measured 22.5 m × 1.6 m each) |
| Untreated control plot dimensions | 46.5 × 1.6 m (The cropped and the non-cropped areas were congruent with each other and measured 22.5 m × 1.6 m each) |
| Distance between control plot and treated plot | Not specified (located in separate greenhouse) |
| Distance between treated plots | 1.5 m |
| Application rate used (g a.s./ha) | 1500 g a.s./ha |
| Was the maximum label rate per ha used in study? | Yes |
| Application date(s) | 10-Oct-00 (cucumber plants at BBCH 12, 8–10 cm high) |
| Application method | Ground-directed drip irrigation technique (chemigation) |
| Type of spray equipment | Commercial drip irrigation equipment |
| Volume of spray solution applied/plot | Approximately 800 L *via* chemigation |
| Identification and volume of carrier (*e.g.*, water), if used | Water<br>(pH adjusted to ~4.5–5.5) |
| Monthly weather reports included (yes/no) | Yes |
| Pan evaporation data available? | No |
| Meteorological conditions during application | |
| Cloud cover (%) | Clouding from 0 to 95% |
| Temperature (air) | 21.9–32.7°C |
| Relative humidity (%) | 24–70% |
| Wind speed | Not applicable |
| Sunlight (hr) [time required for application] | Not reported |
| Supplemental irrigation and method | A permanent drip irrigation system was installed on the surface of the soil of each plot for regular irrigation with water. For the test substance application, a second drip line system was installed on the soil surface of the treated plots in parallel to the irrigation line. |
| Verification of application | Jars were placed under the last emitters at the end of each chemigation line (12 jars in total), and after application, the volume of water and test substance was determined in each jar. |
| Field spikes (transit stability samples) | None; Day 0 sample and application monitor analyses confirmed transit stability |
| Additional modules added to study: run-off, leaching, volatilization | None; irrigation was carefully controlled. Soil sampling to 90 cm (36 in.) to measure movement in soil |

**Table 69 Soil sampling details for the Spain site**

| **Details** | **Vinalesa, Valencia, Spain (Greenhouse)** |
|---|---|
| Method of sampling (random or systematic) | The selection of the subplot was random. Each corer was placed on the spot where an emitter was originally placed at the time of application. |
| Sampling intervals (days after treatment) | -1[a], 0[b], 1, 3, 5, 7, 9, 20, 23, 29, 48, 63, 92, 121, 154, 178, 223, 373<br>(The soil sampling was performed according to the Study Plan for a time period of 373 days until 18 October 2001; however, only the soil taken until 178 DAT was analysed, as each analyte declined rapidly.) |
| Method of soil collection | The 0–10 cm segment was sampled using a plastic cylinder with an inner diameter of 12 cm pushed 10 cm into the soil, and the soil was then scooped out using a hand shovel. The cylinder remained in place during collection of the lower depths to prevent treated soil from falling onto the sampling area and potentially contaminating the lower depths. Soil cores for the 10–90 cm depths were taken with a motor-driven (Geotool model LMSR-R780, acetate liner) coring system. |
| Sampling depth | Nominally to 90 cm depth |
| Number of cores collected per plot | 4 per replicate plot, 12 per time point total |
| Depth and diameter of segments | 0-10 cm (121 mm diameter)<br>10-25 cm (50 mm diameter)<br>25-40 cm (50 mm diameter)<br>40-55 cm (50 mm diameter)<br>55-70 cm (50 mm diameter)<br>70-90 cm (50 mm diameter) |
| Storage conditions | Frozen |
| Maximum storage length | 9.1 months (oxamyl and oxamyl oxime); 15.9 months (DMOA) |

[a] Control soil

[b] Just after application

## II. RESULTS AND DISCUSSION

### A. APPLICATION VERIFICATION

To determine the homogeneity of the application, the entire eluate of the last emitter at the end of each chemigation line was collected in a jar. The total number of jars was twelve. The volume in each jar was measured after the application. The volumes of application solution collected by each single monitoring jar were reported. The volumes collected underneath each of the four emitters of each plot varied little, since the relative standard deviations ranged from 1 to 3%. Therefore, a homogeneous application throughout the entire chemigation system can be assumed.

### B. RESIDUE DECLINE

Residues in mg/kg dry weight basis are listed in Table (cropped plot) and Table (uncropped plot).

The residue data reported reflect the dissipation of oxamyl during several months starting from October 2000. The residue concentrations of oxamyl in the treated cropped and non-cropped plots ranged from <0.005 to 6.2 mg/kg (dry soil) at the day of application and were distributed over the four upper soil segments. The corresponding concentrations of oxamyl oxime ranged from <0.005 to 0.36 mg/kg and were again distributed over the four upper segments. The concentrations of DMOA in the same samples were <0.01 to 0.08 mg/kg. The oxamyl residues declined rapidly during the first twenty days. After approximately seven weeks (DALA 48), no oxamyl residues above 10% of the originally applied test item could be determined in any soil sample. Some residue concentrations were occasionally determined in the top horizons after that time point and ranged from 0.005 to 0.076 mg/kg.

The degradates oxamyl oxime (IN-A2213) and DMOA (IN-D2708) also declined readily within a time period of 63 days. Occasional residues analysed in top soil samples taken thereafter ranged from 0.041 to 0.007 mg/kg. In a few cases, residues were detected in the fifth horizon at DALA 23 through DALA 63. No residues of oxamyl or DMOA were found in any sample from the deepest horizon 70–90 cm. Negligible concentrations of oxamyl oxime were detected three times in the deepest horizon of the cropped and four

times of the non-cropped plots. The maximum concentration was 0.024 mg/kg on DALA 48. Since the $DT_{90}$ concentration was experimentally reached within 60 days, the analysis of samples collected beyond 178 days was terminated.

The residue concentrations indicate that the majority of the test item oxamyl remained in the two top horizons, where it quickly degraded to oxamyl oxime and DMOA, and only minor quantities tended to migrate.

No residues of oxamyl or the degradate oxamyl oxime were observed in any of the control samples and the untreated samples taken before application.

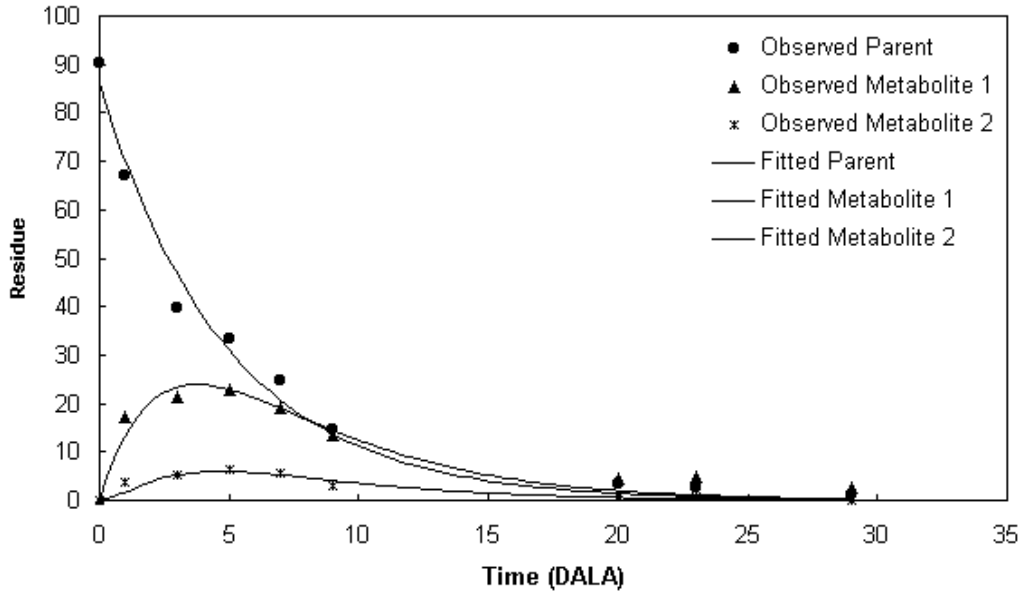
C. MASS BALANCE

Given that chemigation does not deliver a uniform application of the test material, the residue concentrations of each analyte (mg/kg) were converted into mass per area ($\mu g/cm^2$) and the masses of the metabolites were calculated and expressed as oxamyl equivalents. The results of the single plots were averaged, and the masses in the six horizons were summed for each sampling date. As there was little difference in the dissipation between the cropped and non-cropped plots, the data for the cropped and non-cropped plots were combined to calculate an overall half-life for the parent and two metabolites in soil. The resulting masses are tabulated in Table .

D. DISSIPATION KINETICS

The residue data for oxamyl and its degradates oxamyl oxime and DMOA in up to six segments (0–90 cm) for the sampling period 0 through 178 days were used to calculate the $DT_{50}$ and $DT_{90}$ values of the parent and the degradates. The $DT_{50}$ and $DT_{90}$ values for the greenhouse in Vinalesa (Spain) are summarized in the following table and diagram (Figure 5) presented to illustrate the dissipation curve derived from the average of the cropped and non-cropped plots combined.

**Figure 5 Decline of oxamyl at the Spain site**




Y-axis units are $\mu g/cm^2$
X-axis units are days
parent = oxamyl
Metabolite 1 = oxamyl oxime (IN-A2213)
Metabolite 2 = DMOA (IN-D2708)

| | Data sets | | |
|---|---|---|---|
| **Oxamyl** | **Cropped[a]** | **Non-cropped[b]** | **Combined[c]** |
| $DT_{50}$ [d]: | 3.0 | 3.7 | 3.3 |
| $DT_{90}$ [d]: | 10 | 12 | 11 |
| $C_0$ [µg/cm$^2$][d]: | 93 | 78 | 86 |
| $r^{2e}$: | 0.98 | 0.99 | 0.99 |
| **Oxamyl oxime (IN-A2213)** | | | |
| $DT_{50}$ [days]: | 1.9 | 2.3 | 2.1 |
| **DMOA (IN-D2708)** | | | |
| $DT_{50}$ [days]: | 0.36 | 0.72 | 0.52 |

a The residue data of the three cropped plots were averaged prior to regression.

b The residue data of the three non-cropped plots were averaged prior to regression.

c The soil concentration values of the cropped and the non-cropped plots were averaged prior to regression, these values represent the overall dissipation of oxamyl and its soil degradates for this study.

d $C_0$ is the calculated concentration just after application, and derived from the intercept of the ordinate.

e $r^2$ is the coefficient of determination for the sequential fit of the regression model to data for each analyte.

The dissipation curves were generated using ModelManager.

## III. CONCLUSIONS

A field soil dissipation study was conducted with Oxamyl 10SL insecticide and nematicide in a greenhouse at one site in Vinalesa, Spain. The greenhouse was located in a region that exhibited typical conditions of vegetable growing with respect to cultivation, soil, and climate.

The data compiled in the report describe the dissipation of oxamyl and its major soil degradates oxamyl oxime (IN-A2213) and DMOA (IN-D2708) under the conditions investigated. Oxamyl 10SL (liquid formulation containing 10% of oxamyl as the active substance) was applied to the bare soil surface of cropped and non-cropped plots at an application rate of 15 L/ha, anticipated for nematode control in greenhouse-cultivation. The rate corresponds to nominal 1.5 kg of oxamyl per hectare. The test item was applied by chemigation drip irrigation equipment and technique.

The climate conditions were hot and humid throughout the duration of this study as indicated by the climate data provided. The plots were irrigated by drip irrigation frequently according local practice for cucumber cultivation. The cropped and the non-cropped units were irrigated equally.

Soil cores from the treated and control plots were collected randomly to a depth of 90 cm before application and at seventeen time points during one year following the treatment. Soil samples were analysed for residues of oxamyl and oxamyl oxime by LC/MS/MS and for DMOA by a different method using an LC/MS technique. The LOQ for residues in soil was 0.005 mg/kg each for oxamyl and oxamyl oxime and 0.01 mg/kg for DMOA. No residues were detected in any sample taken before the application and in the soil from the untreated control plot.

Oxamyl residue levels declined rapidly to concentrations below 10% of the initial soil concentrations within one month. First order dissipation half-lives ($DT_{50}$) were calculated for each analyte separately for the cropped and the non-cropped plots. The calculated $DT_{50}$ values in the cropped and non-cropped plots ranged from 3.0 to 3.7 days for oxamyl, 1.9 to 2.3 days for oxamyl oxime, and 0.4 to 0.7 days for DMOA. The corresponding $DT_{90}$ value of oxamyl ranged from 10 to 12 days. Since the dissipation of oxamyl and its degradates was very similar between the cropped and non-cropped plots, the two data sets were combined to calculate the overall average degradation values. The averaged $DT_{50}$ values were 3.3 days for oxamyl, 2.1 days for oxamyl oxime, and 0.5 days for DMOA.

Oxamyl and its degradates were observed to a depth of 40 cm on the day of chemigation. This can be expected given the sandy nature of the soil and the high volume of water that was applied to ensure an even application of the test item. During the next month of the study, the analytes were detected in addition in the fourth and fifth horizon 40–70 cm. On 48 DAT, 0.006 mg/kg of oxamyl and 0.024 mg/kg of oxamyl oxime were detected in the lowest horizon (70–90 cm) of one subplot of the non-cropped plot. Otherwise, no residues were found in any of the bottom layer samples.

Since soil residues were tracked up until one clean depth segment, which was confirmed for all but one sample, the calculated half-lives are understood to reflect true degradation half-lives rather than dissipation half-lives. The results of this Oxamyl 10SL chemigation study are consistent with the results of a similar chemigation study of Oxamyl 10SL conducted in Sicily (DuPont-4800).

**Table 70 Residues of oxamyl at each depth for the cropped plot (mg/kg, dry weight basis) for the Spain site**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **9** | **20** | **23** | **29** | **48** | **63** | **92** | **121** | **154** | **178** |
| Oxamyl (0–10 cm) | DA | 3.1 | 2.7 | 1.3 | 0.86 | 1.4 | 0.7 | 0.12 | 0.077 | 0.024 | 0.065 | 0.011 | 0.007 | 0.006 | <0.005 | <0.005 |
| | DB | 6.2 | 2.9 | 1.7 | 1.3 | 1.1 | 0.58 | 0.022 | 0.061 | 0.028 | 0.005 | 0.011 | 0.009 | <0.005 | 0.021 | ND[a] |
| | DC | 3.4 | 3 | 2.7 | 1.2 | 0.89 | 0.39 | 0.029 | 0.092 | 0.029 | 0.014 | 0.076 | 0.01 | 0.008 | 0.006 | <0.005 |
| | **Avg.[b]** | **4.23** | **2.87** | **1.90** | **1.12** | **1.13** | **0.56** | **0.06** | **0.08** | **0.03** | **0.03** | **0.03** | **0.01** | **0.01** | **0.01** | **<0.005** |
| Oxamyl (10–25 cm) | DA | 0.53 | 1 | 1.1 | 0.8 | 0.88 | 0.35 | 0.067 | 0.053 | 0.018 | 0.02 | 0.005 | ND | <0.005 | ND | ND |
| | DB | 2.4 | 2.5 | 0.55 | 1.2 | 0.25 | 0.1 | 0.046 | 0.022 | 0.026 | 0.018 | 0.007 | <0.005 | 0.007 | <0.005 | ND |
| | DC | 2.5 | 1.8 | 1.7 | 0.76 | 0.73 | 0.39 | 0.069 | 0.12 | 0.052 | 0.014 | 0.023 | <0.005 | 0.005 | ND | ND |
| | **Avg.** | **1.81** | **1.77** | **1.12** | **0.92** | **0.62** | **0.28** | **0.06** | **0.07** | **0.03** | **0.02** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxamyl (25–40 cm) | DA | <0.005 | 0.12 | 0.23 | 0.38 | 0.11 | 0.018 | 0.013 | 0.029 | ND | 0.012 | <0.005 | ND | ND | ND | ND |
| | DB | 0.035 | 0.13 | ND | 0.11 | 0.059 | ND | 0.025 | 0.021 | 0.01 | 0.005 | <0.005 | ND | ND | ND | ND |
| | DC | 0.11 | 0.26 | 0.21 | <0.005 | 0.06 | <0.005 | 0.008 | 0.042 | 0.034 | 0.016 | <0.005 | ND | ND | ND | ND |
| | **Avg.** | **0.05** | **0.17** | **0.15** | **0.16** | **0.08** | **0.01** | **0.02** | **0.03** | **0.02** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxamyl (40–55 cm) | DA | ND | ND | ND | ND | ND | ND | ND | <0.005 | ND | ND | <0.005 | n.a.[c] | n.a. | n.a. | n.a. |
| | DB | ND | ND | n.a. | ND | ND | n.a. | 0.043 | 0.023 | 0.009 | 0.019 | ND | n.a. | n.a. | n.a. | n.a. |
| | DC | ND | ND | ND | n.a. | ND | n.a. | <0.005 | 0.009 | 0.011 | 0.014 | <0.005 | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.02** | **0.01** | **0.01** | **0.01** | **<0.005** | --- | --- | --- | --- |
| Oxamyl (55–70 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | ND | ND | ND | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | ND | ND | ND | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | ND | ND | ND | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | --- | --- | --- | --- | --- | --- | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | --- | --- | --- | --- |
| Oxamyl (70–90 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **---** | **<0.005** | **<0.005** | **<0.005** | **---** | **---** | **---** | **---** | **---** |

[a] ND = Not detected.

[b] Mean values calculated by the reviewer using data from Appendix 8 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[c] n.a. = Not analysed.

**Table 70 Residues of oxamyl at each depth for the cropped plot (mg/kg, dry weight basis) for the Spain site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **9** | **20** | **23** | **29** | **48** | **63** | **92** | **121** | **154** | **178** |
| Oxime (0–10 cm) | DA | 0.36 | 0.6 | 0.44 | 0.54 | 0.55 | 0.24 | 0.24 | 0.062 | 0.031 | 0.099 | 0.017 | <0.005 | 0.016 | ND[a] | ND |
| | DB | 0.28 | 0.45 | 0.64 | 0.75 | 0.87 | 0.59 | 0.01 | 0.039 | 0.034 | <0.005 | 0.014 | <0.005 | 0.007 | 0.041 | ND |
| | DC | 0.19 | 0.46 | 1 | 0.48 | 0.57 | 0.3 | 0.054 | 0.096 | 0.026 | 0.014 | 0.12 | 0.008 | <0.005 | 0.008 | ND |
| | **Avg.[b]** | **0.28** | **0.50** | **0.69** | **0.59** | **0.66** | **0.38** | **0.10** | **0.07** | **0.03** | **0.04** | **0.05** | **<0.005** | **0.01** | **0.02** | **<0.005** |
| Oxime (10–25 cm) | DA | 0.045 | 0.27 | 0.47 | 0.82 | 0.37 | 0.2 | 0.036 | 0.041 | 0.026 | 0.007 | <0.005 | ND | <0.005 | ND | ND |
| | DB | 0.22 | 0.52 | 0.26 | 0.45 | 0.27 | 0.071 | 0.019 | 0.026 | 0.029 | 0.007 | <0.005 | <0.005 | <0.005 | <0.005 | ND |
| | DC | 0.24 | 0.39 | 0.61 | 0.28 | 0.45 | 0.23 | 0.03 | 0.17 | 0.036 | 0.005 | 0.018 | <0.005 | <0.005 | ND | ND |
| | **Avg.** | **0.17** | **0.39** | **0.45** | **0.52** | **0.36** | **0.17** | **0.03** | **0.08** | **0.03** | **0.01** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (25–40 cm) | DA | ND | 0.074 | 0.13 | 0.45 | ND | 0.016 | 0.019 | 0.059 | 0.016 | 0.021 | 0.007 | ND | <0.005 | n.a.[c] | ND |
| | DB | <0.005 | 0.018 | ND | 0.034 | 0.042 | ND | 0.015 | 0.02 | 0.02 | 0.006 | <0.005 | <0.005 | <0.005 | n.a. | ND |
| | DC | 0.009 | 0.053 | 0.098 | ND | 0.044 | <0.005 | <0.005 | 0.096 | 0.051 | 0.019 | 0.005 | <0.005 | <0.005 | n.a. | ND |
| | **Avg.** | **0.01** | **0.05** | **0.08** | **0.16** | **0.03** | **0.01** | **0.01** | **0.06** | **0.03** | **0.02** | **<0.005** | **<0.005** | **<0.005** | **n.a.** | **<0.005** |
| Oxime (40–55 cm) | DA | n.a. | ND | ND | ND | ND | ND | ND | 0.008 | <0.005 | 0.008 | 0.033 | n.a. | n.a. | n.a. | n.a. |
| | DB | ND | ND | n.a. | ND | ND | n.a. | 0.043 | 0.026 | 0.017 | 0.045 | ND | n.a. | n.a. | n.a. | n.a. |
| | DC | ND | ND | ND | n.a. | ND | n.a. | <0.005 | 0.035 | 0.022 | 0.025 | 0.006 | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.02** | **0.02** | **0.01** | **0.03** | **0.01** | **---** | **---** | **---** | **---** |
| Oxime (55–70 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | <0.005 | ND | ND | ND | 0.014 | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | 0.013 | ND | 0.023 | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | <0.005 | ND | ND | <0.005 | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **<0.005** | **0.01** | **<0.005** | **0.01** | **0.01** | **---** | **---** | **---** | **---** |
| Oxime (7090 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | <0.005 | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **---** | **<0.005** | **<0.005** | **<0.005** | **---** | **---** | **---** | **---** | **---** |

[a] ND = Not detected.

[b] Mean values calculated by the reviewer using data from Appendix 8 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[c] n.a. = Not analysed.

**Table 70 Residues of oxamyl at each depth for the cropped plot (mg/kg, dry weight basis) for the Spain site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **9** | **20** | **23** | **29** | **48** | **63** | **92** | **121** | **154** | **178** |
| DMOA (0–10 cm) | DA | 0.08 | 0.12 | 0.14 | 0.13 | 0.1 | 0.06 | 0.07 | 0.02 | 0.02 | 0.03 | <0.01 | ND[a] | <0.01 | n.a.[b] | n.a. |
| | DB | 0.04 | 0.08 | 0.08 | 0.1 | 0.12 | 0.08 | ND | ND | <0.01 | ND | ND | ND | ND | n.a. | n.a. |
| | DC | 0.05 | 0.07 | 0.1 | 0.1 | 0.14 | 0.04 | 0.02 | 0.03 | 0.01 | 0.01 | 0.06 | ND | ND | n.a. | n.a. |
| | **Avg.[c]** | **0.06** | **0.09** | **0.11** | **0.11** | **0.12** | **0.06** | **0.03** | **0.02** | **0.01** | **0.02** | **0.02** | **<0.01** | **<0.01** | --- | --- |
| DMOA (10–25 cm) | DA | 0.01 | 0.04 | 0.1 | 0.09 | 0.07 | 0.03 | ND | <0.01 | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | DB | 0.03 | 0.04 | 0.02 | 0.04 | 0.06 | 0.01 | ND | ND | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | DC | 0.02 | 0.03 | 0.08 | 0.02 | 0.06 | 0.03 | <0.01 | 0.01 | ND | ND | <0.01 | <0.01 | n.a. | n.a. | n.a. |
| | **Avg.** | **0.02** | **0.04** | **0.07** | **0.05** | **0.06** | **0.02** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | --- | --- | --- |
| DMOA (25–40 cm) | DA | ND | <0.01 | <0.01 | 0.04 | 0.01 | ND | ND | ND | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | DB | ND | ND | ND | ND | <0.01 | ND | ND | ND | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | DC | ND | ND | <0.01 | ND | ND | ND | ND | 0.01 | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | **Avg.** | **<0.01** | **<0.01** | **<0.01** | **0.02** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | --- | --- | --- |
| DMOA (40–55 cm) | DA | n.a. | n.a. | ND | ND | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | --- | --- | **<0.01** | **<0.01** | **<0.01** | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| DMOA (55–70 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| DMOA (70–90 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |

[a] ND = Not detected.

[b] n.a. = Not analysed.

[c] Mean values calculated by the reviewer using data from Appendix 8 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

**Table 71 Residues of oxamyl at each depth for the bareground (non-cropped) plot (mg/kg, dry weight basis) for the Spain site**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **9** | **20** | **23** | **29** | **48** | **63** | **92** | **121** | **154** | **178** |
| Oxamyl (0–10 cm) | DA | 3.3 | 4.4 | 0.84 | 1.33 | 0.9 | 0.54 | 0.049 | 0.022 | 0.017 | <0.005 | <0.005 | 0.007 | 0.008 | ND[a] | ND |
| | DB | 3.7 | 2.6 | 1.6 | 2.2 | 1.6 | 0.81 | 0.13 | 0.044 | 0.026 | ND | 0.007 | 0.007 | 0.006 | <0.005 | <0.005 |
| | DC | 2.9 | 2.8 | 1.9 | 1.2 | 1.2 | 0.4 | 0.13 | 0.056 | 0.015 | 0.03 | 0.011 | <0.005 | 0.006 | ND | ND |
| | **Avg.[b]** | **3.30** | **3.27** | **1.45** | **1.58** | **1.23** | **0.58** | **0.10** | **0.04** | **0.02** | **0.01** | **0.01** | **0.01** | **0.01** | **<0.005** | **<0.005** |
| Oxamyl (10–25 cm) | DA | 1.2 | 1.5 | 0.67 | 0.88 | 0.09 | 0.25 | 0.075 | 0.012 | 0.02 | 0.008 | <0.005 | 0.005 | ND | ND | <0.005 |
| | DB | 0.79 | 1.4 | 2.2 | 0.87 | 0.48 | 0.83 | 0.18 | 0.051 | 0.045 | 0.006 | 0.014 | <0.005 | <0.005 | ND | <0.005 |
| | DC | 1.8 | 2.2 | 0.46 | 0.93 | 0.68 | 0.29 | 0.067 | 0.087 | 0.016 | 0.017 | <0.005 | <0.005 | <0.005 | ND | ND |
| | **Avg.** | **1.26** | **1.70** | **1.11** | **0.89** | **0.42** | **0.46** | **0.11** | **0.05** | **0.03** | **0.01** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxamyl (25–40 cm) | DA | <0.005 | <0.005 | 0.055 | 0.3 | 0.008 | 0.027 | 0.017 | 0.01 | 0.008 | ND | ND | ND | ND | ND | ND |
| | DB | <0.005 | 0.006 | 0.21 | ND | ND | 0.017 | 0.12 | 0.022 | 0.01 | <0.005 | 0.01 | ND | ND | ND | ND |
| | DC | 0.019 | 0.35 | 0.11 | 0.075 | 0.11 | 0.045 | 0.043 | 0.01 | 0.008 | 0.006 | ND | ND | ND | ND | ND |
| | **Avg.** | **0.01** | **0.12** | **0.13** | **0.13** | **0.04** | **0.03** | **0.06** | **0.01** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxamyl (40–55 cm) | DA | n.a.[c] | n.a. | ND | ND | ND | ND | <0.005 | 0.006 | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | ND | ND | n.a. | n.a. | ND | 0.021 | <0.005 | n.a. | <0.005 | 0.008 | n.a. | n.a. | n.a. | n.a. |
| | DC | ND | ND | ND | ND | ND | ND | ND | ND | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.01** | **<0.005** | **---** | **<0.005** | **0.008** | **---** | **---** | **---** | **---** |
| Oxamyl (55–70 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | 0.006 | 0.007 | ND | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | <0.005 | ND | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **---** | **0.006** | **<0.005** | **<0.005** | **<0.005** | **---** | **---** | **---** | **---** |
| Oxamyl (70–90 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | <0.005 | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | 0.006 | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **---** | **<0.005** | **<0.005** | **0.006** | **---** | **---** | **---** | **---** | **---** |

[a] ND = Not detected.

[b] Mean values calculated by the reviewer using data from Appendix 8 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[c] n.a. = Not analysed.

**Table 71 Residues of oxamyl at each depth for the bareground (non-cropped) plot (mg/kg, dry weight basis) for the Spain site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **9** | **20** | **23** | **29** | **48** | **63** | **92** | **121** | **154** | **178** |
| Oxime (0–10 cm) | DA | 0.31 | 0.72 | 0.52 | 0.69 | 0.49 | 0.21 | 0.043 | 0.011 | 0.007 | 0.009 | <0.005 | <0.005 | <0.005 | ND[a] | ND |
| | DB | 0.23 | 0.27 | 0.67 | 0.44 | 0.86 | 0.49 | 0.076 | 0.067 | 0.012 | ND | <0.005 | <0.005 | <0.005 | 0.005 | ND |
| | DC | 0.21 | 0.35 | 0.69 | 0.6 | 0.63 | 0.33 | 0.33 | 0.03 | 0.015 | 0.019 | 0.022 | <0.005 | <0.005 | ND | ND |
| | **Avg.[b]** | **0.25** | **0.45** | **0.63** | **0.58** | **0.66** | **0.34** | **0.15** | **0.04** | **0.01** | **0.01** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (10–25 cm) | DA | 0.15 | 0.46 | 0.35 | 0.54 | 0.056 | 0.24 | 0.052 | 0.026 | 0.012 | <0.005 | <0.005 | <0.005 | <0.005 | ND | <0.005 |
| | DB | 0.059 | 0.3 | 0.7 | 0.13 | 0.24 | 0.52 | 0.074 | 0.023 | 0.047 | 0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DC | 0.13 | 0.38 | 0.19 | 0.56 | 0.48 | 0.28 | 0.072 | 0.12 | 0.015 | 0.006 | <0.005 | <0.005 | <0.005 | ND | ND |
| | **Avg.** | **0.11** | **0.38** | **0.41** | **0.41** | **0.26** | **0.35** | **0.07** | **0.06** | **0.02** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (25–40 cm) | DA | ND | <0.005 | 0.016 | 0.29 | 0.01 | 0.048 | 0.03 | 0.056 | 0.042 | 0.05 | n.a.[c] | <0.005 | ND | ND | ND |
| | DB | <0.005 | <0.005 | 0.11 | ND | <0.005 | 0.014 | 0.063 | 0.018 | 0.026 | 0.014 | n.a. | ND | ND | ND | ND |
| | DC | <0.005 | 0.057 | 0.055 | 0.082 | 0.073 | 0.094 | 0.14 | 0.033 | 0.07 | 0.01 | n.a. | ND | ND | ND | ND |
| | **Avg.** | **<0.005** | **0.021** | **0.06** | **0.12** | **0.03** | **0.05** | **0.08** | **0.04** | **0.05** | **0.02** | **n.a.** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (40–55 cm) | DA | ND | n.a. | ND | ND | ND | ND | 0.011 | 0.053 | 0.056 | <0.005 | <0.005 | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | ND | ND | n.a. | n.a. | ND | ND | <0.005 | 0.006 | 0.01 | 0.021 | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | ND | ND | ND | ND | ND | ND | ND | 0.012 | ND | <0.005 | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.01** | **0.02** | **0.02** | **0.01** | **0.01** | **---** | **---** | **---** | **---** |
| Oxime (55–70 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | <0.005 | 0.056 | 0.042 | ND | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | n.a. | ND | 0.017 | <0.005 | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **<0.005** | **0.056** | **0.02** | **0.01** | **<0.005** | **---** | **---** | **---** | **---** |
| Oxime (70–90 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | 0.007 | 0.014 | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | 0.024 | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **---** | **---** | **---** | **---** | **---** | **---** | **---** | **0.007** | **0.014** | **0.024** | **---** | **---** | **---** | **---** | **---** |

[a] ND = Not detected.

[b] Mean values calculated by the reviewer using data from Appendix 8 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[c] n.a. = Not analysed.

**Table 71 Residues of oxamyl at each depth for the bareground (non-cropped) plot (mg/kg, dry weight basis) for the Spain site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **9** | **20** | **23** | **29** | **48** | **63** | **92** | **121** | **154** | **178** |
| DMOA (0–10 cm) | DA | 0.08 | 0.16 | 0.29 | 0.15 | 0.1 | 0.04 | 0.01 | ND[a] | ND | 0.02 | ND | ND | ND | n.a.[b] | n.a. |
| | DB | 0.05 | 0.12 | 0.11 | 0.13 | 0.15 | 0.07 | 0.01 | 0.02 | ND | ND | ND | ND | ND | n.a. | n.a. |
| | DC | 0.05 | 0.08 | 0.2 | 0.21 | 0.35 | 0.07 | 0.04 | <0.01 | <0.01 | 0.01 | 0.02 | <0.01 | ND | n.a. | n.a. |
| | **Avg.[c]** | **0.06** | **0.12** | **0.20** | **0.16** | **0.20** | **0.06** | **0.02** | **0.01** | **<0.01** | **0.01** | **0.01** | **<0.01** | **<0.01** | --- | --- |
| DMOA (10–25 cm) | DA | 0.03 | 0.07 | 0.08 | 0.14 | 0.02 | 0.1 | <0.01 | ND | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | DB | <0.01 | 0.03 | 0.09 | 0.09 | 0.03 | 0.05 | 0.01 | ND | <0.01 | ND | ND | ND | n.a. | n.a. | n.a. |
| | DC | 0.02 | 0.05 | 0.03 | 0.13 | 0.14 | 0.05 | <0.01 | 0.01 | ND | ND | ND | <0.01 | n.a. | n.a. | n.a. |
| | **Avg.** | **0.02** | **0.05** | **0.07** | **0.12** | **0.06** | **0.07** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | --- | --- | --- |
| DMOA (25–40 cm) | DA | ND | ND | ND | 0.06 | ND | <0.01 | <0.01 | <0.01 | ND | <0.01 | ND | ND | n.a. | n.a. | n.a. |
| | DB | ND | ND | <0.01 | ND | ND | ND | 0.01 | <0.01 | <0.01 | ND | ND | ND | n.a. | n.a. | n.a. |
| | DC | ND | ND | ND | 0.01 | <0.01 | 0.01 | 0.02 | <0.01 | ND | ND | ND | ND | n.a. | n.a. | n.a. |
| | **Avg.** | **<0.01** | **<0.01** | **<0.01** | **0.03** | **<0.01** | **<0.01** | **0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | --- | --- | --- |
| DMOA (40–55 cm) | DA | n.a. | n.a. | <0.01 | ND | n.a. | ND | ND | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | <0.01 | <0.01 | ND | ND | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | **n.a.** | **n.a.** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | --- | --- | --- | --- | --- | --- | --- |
| DMOA (55–70 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| DMOA (70–90 cm) | DA | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DB | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | DC | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| | **Avg.** | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |

[a] ND = Not detected.

[b] n.a. = Not analysed.

[c] Mean values calculated by the reviewer using data from Appendix 8 of the DuPont-4719. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

**Table 72 Residues of oxamyl in soil from bare ground and cropped plots, combined and expressed in terms of mass per area for the Spain site**

| Time (DALA) | Oxamyl (µg/cm2) | Oxamyl oxime[a] (IN-A2213) (µg/cm$^2$) | DMOA[a] (IN-D2708) (µg/cm$^2$) |
|---|---|---|---|
| 0 | 90 | 0 | 0 |
| 1 | 66 | 17 | 3.9 |
| 3 | 39 | 21 | 5.2 |
| 5 | 33 | 24 | 6.1 |
| 7 | 24 | 19 | 5.6 |
| 9 | 14 | 13 | 3.0 |
| 20 | 3.4 | 4.5 | 0.87 |
| 23 | 2.7 | 5.1 | 0.67 |
| 29 | 1.2 | 2.9 | 0.17 |
| 48 | 0.82 | 1.8 | 0.38 |
| 63 | 0.49 | 1.1 | 0.32 |
| 92 | 0.18 | 0.04 | 0 |
| 121 | 0.09 | 0.09 | 0 |
| 154 | 0.07 | 0.18 | 0 |
| 178 | 0 | 0 | 0 |

[a] Values given in oxamyl equivalents.

The following greenhouse dissipation study is being presented as confirmatory data, to expand the pool of field dissipation data for oxamyl. This study has been included because the greenhouse results are consistent with those from the traditional bare field studies in rate and metabolite profile, and thus increase the confidence in the understanding of oxamyl dissipation under actual use conditions. Since oxamyl degrades so quickly, the more controlled, smaller-scale conditions of the greenhouse allow for a more homogeneous application and better tracking of the material, especially the metabolites, and thus allow for more robust pathway kinetics. Lastly, oxamyl has a substantial chemical component to its degradation, so slight variation in soil microbial populations present between the greenhouse and outdoors do not impact the validity of the dissipation data.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.2.1/04**

| | | |
|---|---|---|
| **Reference: CA 7.1.2.2.1/04** | **Report:** | Zietz, E. (2002); Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10 L by means of drip irrigation to cucurbits in a greenhouse in Italy - Season 2000<br>**DuPont Report No.:** DuPont-4800<br>**Guidelines:** SETAC Europe (1995)<br>**Deviations:** None<br>**Testing Facility:** Institut Fresenius Chemische und Biologische/Gmbh, Taunusstein, Germany<br>**Testing Facility Report No.:** DuPont-4800<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive Summary**

The soil dissipation of oxamyl following a single chemigation (drip irrigation) application of oxamyl 100 g/L SL (soluble concentrate) to bare ground and cropped plots was studied under greenhouse conditions in Punta Secca, Sicily, Italy over the period of one year, beginning in November 2000.

The study was designed to determine the environmental fate and the rate of decline of oxamyl and its major degradates oxamyl oxime (IN-A2213) and dimethyl oxalamic acid (DMOA or IN-D2708) following application of Oxamyl 10SL to cropped and non-cropped plots under actual field conditions. Oxamyl, as Oxamyl 10SL, was applied by chemigation (drip irrigation) once at 1.5 kg a.s./ha in a greenhouse at a representative site in Sicily, Italy. The greenhouse conditions were maintained according to local practice. The soil was a slightly clayey sand (pH, 7.1–7.5, OM, ~1%, WHC, 32–40 g/100g). The bare ground plots were kept in a non-cropped condition throughout the study period, while cucumbers were grown on the cropped plots.

Three treatment plots and one untreated control plot were established within the greenhouse, with each plot divided into a non-cropped area and an area cropped with cucumbers. Oxamyl was applied once to each of the three plots using a drip irrigation technique at 1.5 kg active ingredient per hectare in 480–570 L of water per plot. Application monitors were set out at the time of application to verify the amount applied, which was 101% of the target rate. The application monitoring also indicated that very little remained within the emitters, and that consequently the test item was applied evenly. Given the conditions of chemigation as a spot application and the relatively high amount of water applied to sandy soil, the treatment regime represented a worst case scenario with respect to leaching and dissipation behaviour.

Soil cores from the treated and control plots were collected randomly to a depth of 90 cm before application (-1 DAT) and after application, 0, 1, 3, 5, 7, 10, 14, 20, 28, 48, and 62 days after treatment (samples collected at 90 to 358 DAT were not analysed). At each sampling time, four cores were collected from each of the treated cropped and non-cropped plots for a total of 24 cores. Four cores were collected from each the cropped and non-cropped untreated control for a total of eight cores. Due to the coring principle (system Humax) each core was segmented during the sampling procedure. The segments corresponded to 0–10 cm, 10–25 cm, 25–40 cm, 40–55 cm, 55–70 cm, and 70–90 cm. Soil cores were frozen within two hours at the field site. Prior to analysis, the soil segments were composited by sampling time, plot, crop cover, and depth (horizon).

Soil samples were analysed for oxamyl and oxamyl oxime (IN-A2213) residues by Institut Fresenius (Taunusstein, Germany) according to method DuPont-2392, summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 4, DuPont-40931 EU. The residues were extracted with acidified acetonitrile:methanol (80:20, v/v), concentrated, diluted with acidified water, filtered (0.45 µm PTFE), and determined by LC/MS/MS monitoring ion transitions m/z 237→72 for oxamyl and m/z 163→72 for oxamyl oxime using electrospray ionization. Samples of untreated soil were fortified at 0.005–2.3 mg/kg when the method was validated (prior to field sample analysis) and 0.005–0.46 mg/kg, when fortified samples were analysed concurrently with the samples from the field. The method validation recoveries averaged 94% (RSD 5.1%, n = 16) for oxamyl and 93% (RSD 3.7%, n=16) for oxamyl oxime, and the concurrent recoveries averaged 97% (RSD 6.6%, n = 154) for oxamyl and 96% (RSD 7.1%, n=156) for oxamyl oxime. The limit of quantitation (LOQ) for oxamyl and oxamyl oxime residues in soil was 0.005 mg/kg for each analyte.

Soil samples were analysed for the degradate DMOA (IN-D2708) at TNO Nutrition and Food Research (The Netherlands) using an LC/MS method. The DMOA residues were extracted with an aqueous solvent under basic conditions (10 mM ammonium carbonate in water), cleaned up by SPE, and then analysed by LC/MS operating in the negative ion mode monitoring a single MS-SIM (m/z 116 for DMOA and 122 for deuterated DMOA). Quantitation was obtained using an internal calibration curve created with a d6-DMOA analytical standard. Samples of untreated soil were fortified with DMOA at 0.01–0.2 mg/kg for validation, with average method validation recoveries of 86–89% (RSD 2–6%, n = 16) and concurrent recoveries averaging 99% (RSD 7%, n=58). The LOQ for DMOA residues in soil was 0.01 mg/kg.

All treated samples collected up to 62 days after application were analysed for oxamyl, oxamyl oxime, and DMOA. All horizons from top soil to 90 cm (0–90 cm) were investigated for each sampling date.

Samples collected later than 62 days after application were not analysed since oxamyl, oxamyl oxime, and DMOA dissipated rapidly, and no residues above 0.005 mg/kg or 0.01 mg/kg for DMOA were detected in any specimen taken beyond 28 days after application.

No residues of oxamyl or the degradates oxamyl oxime and DMOA were detected in any of the control samples and the untreated samples taken before application.

The residue concentrations of oxamyl in the three treated cropped and non-cropped plots ranged from <0.005 to 1.6 mg/kg (dry soil) at the day of application and were distributed over the four upper soil horizons as tabulated below.

The corresponding concentrations of oxamyl oxime ranged from <0.005 to 0.19 mg/kg and were again distributed over the four upper horizons. The concentrations of DMOA in the same samples ranged from <0.01 to 0.02 mg/kg. The oxamyl residues declined rapidly within the first month. After approximately seven weeks (DALA 48), no residues of oxamyl could be determined in any soil sample except three samples where the residue concentration was just above the LOQ (0.005 mg/kg).

The degradates oxamyl oxime and DMOA (IN-D2708) also declined within this time period. No residues of the degradates above the LOQ could be determined in any soil sample collected 48 days after application or later.

Oxamyl and oxamyl oxime tend to migrate into deeper horizons. In few cases, residues just above the LOQ were detected in the fifth soil horizon at 7 DAT through 20 DAT. No residues above the LOQ were determined in the deepest horizon 70–90 cm. No lateral diffusion greater than 30 cm was observed.

Although soil samples were collected at six more sampling dates, the analysis was terminated when the experimental residue data indicated that the $DT_{90}$ had been reached prior to 60 days after application.

The residue concentrations indicate that the majority of the test item remained in the third and fourth horizon where the analyte dissipated quickly. Only minor quantities tended to migrate further.

For the kinetics evaluation, the analyte concentrations (mg/kg) were converted into mass per area as $\mu g/cm^2$, with the masses of the metabolites calculated as oxamyl equivalents. The results of the single plots were averaged, and the masses in the six horizons were summed for each sampling date. As there was little difference in the dissipation between the cropped and non-cropped plots, the data for the cropped and non-cropped plots were combined to calculate an overall half-life for the parent and two metabolites in soil. The first order residue concentrations in the soil averaged over all three treated plots yielded a half-life ($DT_{50}$) of 5.3 days for oxamyl, 5.7 days for oxamyl oxime (IN-A2213), and 3.2 days for DMOA (IN-D2708). Since soil residues were tracked up until one clean layer, the calculated half-lives are understood as true degradation half-lives, rather than dissipation half-lives.

The storage period between sampling in the field and extraction did not exceed 14.4 months for all samples analysed for oxamyl and oxamyl oxime. The maximum storage interval between the sampling and extraction date for the DMOA metabolite was 17.2 months. The freezer storage stability of the soil residues has been determined (refer to summary for DuPont-9342).

## I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: Oxamyl 10SL
   Lot/Batch #: D1410-417
   Purity: 100 g/L (nominal), 101.3 g a.s./L (measured)
   Description: Dark green liquid
   CAS #: None for the formulation; 23135-22-0 for oxamyl active substance
   Stability of the test compound: Stable for a minimum of 2 months when stored at 4 to 8°C in a refrigerator

2. Test Site

Test site description is detailed in Table . Soil samples collected to 90 cm depth were characterized and the soil characterization data are included in Table .

**Table 73 Test site description at the Italy site**

| | |
|---|---|
| Location: | Punta Secca, Sicily, Italy |
| Region: | Southern coast of Sicily |
| GPS Coordinates: | Not reported |
| Representative Crop Region: | Vegetables cultivated in greenhouses |
| Site Selection Criteria: | Typical greenhouse with flat and level soil. Soil allowing coring at least down to 90 cm |
| Weather Station: | Not applicable |
| Pretreatment Exclusion Criteria: | No other chemical of similar structure applied during the past 3 years. |
| Plot History, Crops Grown: | Cucumber (October 1997–March 1998); cucumber (October 1998–March 1999); tomato (March 1999–June 1999); cucumber (October 1999–April 2000). |
| Pesticides Used in Preceding 3 Years: | No oxamyl was allowed during the recent 3 years. Fenamiphos, cymoxanil, dinocap, cyproconazole, pemconazole, pyrifenox, imidacloprid, abamectin, pyridaben, endosulfan, dicloran, and methyl bromide (last $CH_3Br$ fumigation 29-Sep-99). |
| Cultivation Method: | The soil was cultivated with a rotary tiller and levelled with a rake before the plots were surveyed. Due to the sandy soil, the tilth was extremely fine with clods less than 1 cm diameter. |
| Location/Identification of Weather Station | Not applicable. Climate data recorded inside the greenhouse were reported (daily average temperatures and humidity) for the period of the field phase November 2000 until October 2001. |
| Distance of Weather Station from Test Site: | Not applicable |
| Depth to Ground Water Table: | Not applicable |

**Table 74 Soil properties at the Italy site**

| | **Soil depth (cm)** | | | | | |
|---|---|---|---|---|---|---|
| **Soil Property** | **0-10** | **10-25** | **25-40** | **40-55** | **55-70** | **70-90** |
| Sand % (0.063-2 mm)[a] | 87.9 | 89.3 | 89.9 | 84.4 | 84.5 | 86.5 |
| Silt % (0.002-0.063 mm)[a] | 6.2 | 5.2 | 4.8 | 7.8 | 6.2 | 6.9 |
| Clay % (<0.002 mm)[a] | 5.9 | 5.5 | 5.3 | 7.8 | 9.3 | 6.5 |
| pH (water) | N/A[b] | N/A | N/A | N/A | N/A | N/A |
| pH (0.01M $CaCl_2$) | 7.4 | 7.5 | 7.5 | 7.3 | 7.1 | 7.2 |
| % Total Organic Carbon (TOC)[c] | 0.7 | 0.5 | 0.2 | 0.7 | 0.5 | 0.3 |
| % Organic matter[d] | 1.2 | 0.8 | 0.4 | 1.1 | 0.8 | 0.6 |
| C.E.C [meq/100g][e] | 5.9 | 5.7 | 5.3 | 9.0 | 7.9 | 6.7 |
| Bulk density (g $cm^{-3}$) [f] | 2.07 | - | - | - | - | - |
| Water Holding Capacity (% of dry wt.) | 31.9 | 35.6 | 32.4 | 39.9 | 39.2 | 35.1 |
| Soil Classification[g] | Sand | Sand | Sand | Sand | Sand | Sand |
| Soil Series Name | N/A | N/A | N/A | N/A | N/A | N/A |

[a] Particle size distribution (%)

[b] Not applicable and/or not reported

[c] Organic carbon content [% or g/100 g of dry soil]

[d] TOC × 1.72 (%)

[e] Cation Exchange Capacity (C.E.C)

[f] The density at the original position in the field (horizon 0–12 cm), average of eight spots in the field taken into account

[g] Soil classification according to USDA system

## B. METHODS

### 1. Experimental design

The experimental details for the test substance application, application rate, application method, etc., are included in **Errore. L'origine riferimento non è stata trovata.**.

2. Soil sampling

Soil sampling intervals, sampling depths, and number of cores collected are listed in **Errore. L'origine riferimento non è stata trovata.**.

3. Description of analytical methods

Soil samples were analysed for oxamyl and oxamyl oxime (IN-A2213) residues by Institut Fresenius (Taunusstein, Germany) according to method DuPont-2392, summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 4, DuPont-40931 EU, modified (method details are included in the study report). The residues were extracted with acidified acetonitrile:methanol (80:20, v/v), concentrated, diluted with acidified water, filtered (0.45 µm PTFE), and determined by LC/MS/MS monitoring ion transitions m/z 237→72 for oxamyl and m/z 163→72 for oxamyl oxime using electrospray ionization. The concentrations (mg/kg) of oxamyl and its degradation product oxamyl oxime were quantitated using an external calibration curve of each analyte. The LOQ for oxamyl and oxamyl oxime residues in soil was 0.005 mg/kg each.

Soil samples were analysed separately for the degradate DMOA (IN-D2708) by TNO Nutrition and Food Research (AJ Zeist, The Netherlands) using an LC/MS method (method details are included in the study report). The DMOA residues were extracted with an aqueous solvent under basic conditions (10 mM ammonium carbonate in water), cleaned up by SPE, and then analysed by LC/MS operating in the negative ion mode monitoring a single MS-SIM (m/z 116 for DMOA and 122 for d6-DMOA). Quantitation was obtained using an internal calibration curve created with a d6-DMOA analytical standard. The LOQ for DMOA residues in soil was 0.01 mg/kg.

The concentrations (mg/kg) of oxamyl and its degradation products in treated field soil samples were calculated on a dry weight basis.

**Table 75 Experimental design, plot set up, and application details for the Italy site**

| Details | Punta Secca, Sicily, Italy (Greenhouse) |
|---|---|
| Duration of study | 62 days |
| Uncropped (bare) or cropped | Bare (maintained weed free) and Cropped (cucumber) |
| Controls used | Yes |
| Number of plot(s) | Three treated (replicates DA, DB, and DC) and one untreated control (DX), each divided into non-cropped and cropped areas. |
| Treated plot dimensions | 22.5 × 1.4 m (three each for the uncropped and cropped regimes, for a total of six treated subplots of this dimension) |
| Untreated control plot dimensions | 22.5 × 1.4 m (one each uncropped and cropped, for a total of two untreated control subplots of this dimension) |
| Distance between control plot and treated plot | 5.7 m |
| Distance between treated plots | Not specified |
| Application rate used (g a.s./ha) | 1.5 kg a.s./ha, nominal |
| Was the maximum label rate per ha used in study? | Yes |
| Application date(s) | 22-Nov-00 (cucumber plants at BBCH 14) |
| Application method | Ground-directed drip irrigation technique (chemigation), after transplanting (for cropped plots) |
| Type of spray equipment | Commercial drip irrigation equipment |
| Volume of spray solution applied/plot | Approximately 480 to 570 L *via* chemigation |
| Identification and volume of carrier (*e.g.*, water), if used | Water<br>(pH adjusted) |
| Monthly weather reports included (yes/no) | Yes |
| Pan evaporation data available? | No |
| Meteorological conditions during application | |
| Cloud cover (%) | Sunshine |
| Temperature (air) | 15–28°C |
| Relative humidity (%) | 45–81% |
| Wind speed | Not applicable |
| Sunlight (hr)<br>[time required for application] | Not reported |
| Supplemental irrigation and method | A permanent drip irrigation system was installed on the surface of the soil of each plot for regular irrigation with water. For the test substance application, a second drip line system was installed on the soil surface of the treated plots in parallel to the irrigation line. |
| Verification of application | Jars were placed under the last emitters at the end of each chemigation line (12 jars in total), and after application, the volume of water and test substance was determined in each jar. |
| Field spikes (transit stability samples) | None; Day 0 sample and application monitor analyses confirmed transit stability |
| Additional modules added to study: run-off, leaching, volatilization | None; irrigation was carefully controlled. Soil sampling to 90 cm (36 in.) to measure movement in soil |

**Table 76 Soil sampling details for the Italy site**

| Details | Punta Secca, Sicily, Italy (Greenhouse) |
|---|---|
| Method of sampling (random or systematic) | The selection of the subplot was random. Each corer was placed on the spot where an emitter was originally placed at the time of application. |
| Sampling intervals (days after treatment) | -1[a], 0[b], 1, 3, 5, 7, 10, 14, 20, 28/29, 48, 62, 90, 118, 149, 180, 218, 358 (The soil sampling was performed according to the Study Plan for a time period of 358 days until 15 November 2001; however, only the soil taken until 62 days after application was analysed since each analyte declined rapidly.) |
| Method of soil collection | The 0–10 cm segment was sampled using a metal cylinder with an inner diameter of 11.90 cm driven 10 cm into the soil, and the soil was then scooped out by hand using a ladle or spoon. The metal cylinder remained in place during collection of the lower depths to prevent treated soil from falling onto the sampling area and potentially contaminating the lower depths. Soil cores for the 10–90 cm depths were taken with a motor-driven (Humax® HS) coring system. |
| Sampling depth | Nominally to 90 cm depth |
| Number of cores collected per plot | 4 per replicate plot, 12 per time point total<br>(Five additional samples were taken one month after application from the designated subplot to investigate the potential of lateral diffusion of the test item. These samples were taken on a line perpendicular of the chemigation line. The sampling spots were 30 cm, 40 cm, and 60 cm apart from the corresponding emitter.) |
| Depth and diameter of segments | 0–10 cm (119 mm diameter)<br>10–25 cm (50 mm diameter)<br>25–40 cm (50 mm diameter)<br>40–55 cm (50 mm diameter)<br>55–70 cm (50 mm diameter)<br>70–90 cm (50 mm diameter) |
| Storage conditions | Frozen |
| Maximum storage length | 14.4 months (oxamyl and oxamyl oxime); 17.2 months (DMOA) |

[a] Control soil

[b] Just after application

## II. RESULTS AND DISCUSSION

### A. APPLICATION VERIFICATION

To determine the homogeneity of the application, the entire eluate of the last emitter at the end of each chemigation line was collected in a jar. The total number of jars was twelve. The volume in each jar was measured after the application, and the solution was stored deep-frozen thereafter. The content of oxamyl and oxamyl oxime was analysed, and the mass as the sum of oxamyl and the oxamyl equivalent of the oxime metabolite was determined. The average mass of the oxamyl equivalent was 26.5 mg/emitter (range 24.6–28.7, N = 12). Considering the 360 emitters per plot, the average output was 9.55 g/plot. Relative to the total plot area (63 $m^2$) the actual applied rate was 1516 g/ha, or 99.7% of the nominal application rate and 101% of the target application rate.

### B. RESIDUE DECLINE

Residues in mg/kg dry weight basis are listed in Table (cropped plot) and Table (uncropped plot).

The residue data reflect the degradation of oxamyl during two months starting from November 2000. Oxamyl was analysed together with the degradate oxamyl oxime in all six soil horizons 0–90 cm.

The residue concentrations of oxamyl in the three treated cropped and non-cropped plots ranged from <0.005 to 1.6 mg/kg (dry soil) at the day of application and was distributed over the four upper soil horizons as tabulated below. The corresponding concentrations of oxamyl oxime ranged from <0.005 to

0.19 mg/kg and were again distributed over the four upper horizons. The concentrations of DMOA in the same samples ranged from <0.01 to 0.02 mg/kg.

The oxamyl residues declined rapidly during the first twenty days. After approximately seven weeks (48 DAT), no residues of oxamyl the active substance of the test item could be determined in any soil sample except three samples where the residue concentration was just above the LOQ (0.005 mg/kg). The degradates oxamyl oxime and DMOA also declined within this time period. No residues of the degradates above the LOQ could be determined in any soil sample. Since these residues data indicated that the $DT_{90}$ of the test item and its metabolites had been reached prior to 60 days after application, no further samples were analysed.

Oxamyl and oxamyl oxime tended to migrate into deeper horizons. In few cases, residues just above the LOQ were detected in the fifth horizon at 7 through 20 DAT; however, no residues above the LOQ were determined in the deepest horizon 70–90 cm.

The residue concentrations indicate that the majority of the test item remained in the third and fourth horizon where the analytes dissipated quickly. Only minor quantities tended to migrate into the fifth horizon. At all sampling times, at least one residue free horizon was observed, indicating that oxamyl and the degradates did not migrate out of the sampling zone.

No residues of oxamyl or the degradate oxamyl oxime were found in any of the control samples and the untreated samples taken before application.

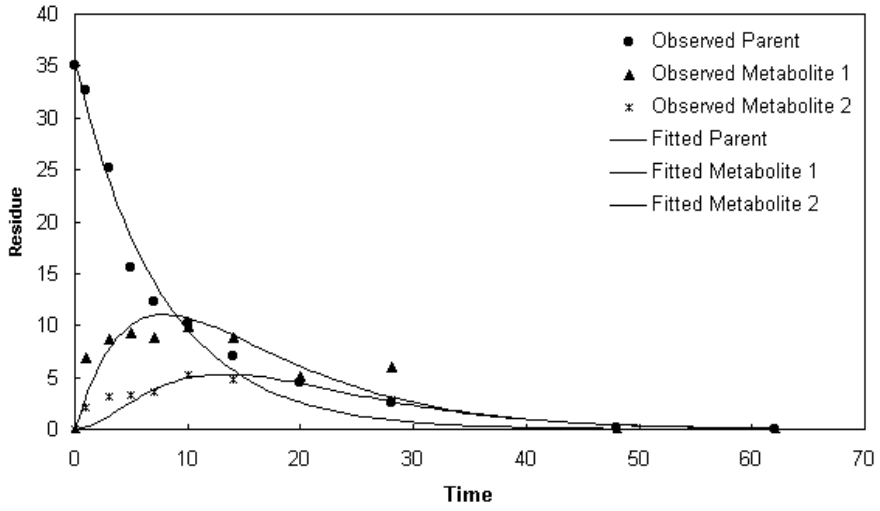
C. MASS BALANCE

Given that chemigation does not deliver a uniform application of the test material, the residue concentrations of each analyte (mg/kg) were converted into mass per area ($\mu g/cm^2$) and the masses of the metabolites were calculated and expressed as oxamyl equivalents. The results of the single plots were averaged, and the masses in the six horizons were summed for each sampling date. As there was little difference in the dissipation between the cropped and non-cropped plots, the data for the cropped and non-cropped plots were combined to calculate an overall half-life for the parent and two metabolites in soil. The resulting masses are tabulated in Table .

D. DISSIPATION KINETICS

The residue data for oxamyl and its degradates oxamyl oxime and DMOA in five horizons (0–70 cm) for the sampling period 0 through 62 days were used to calculate the first-order $DT_{50}$ and $DT_{90}$ values of the parent and the degradates. The first-order $DT_{50}$ and $DT_{90}$ values for the greenhouse in Punta Secca (Italy) are summarized in the following table and diagram (Figure 6) presented to show the dissipation curve derived from the average residue data of three plots (cropped and non-cropped combined).

**Figure 6 Decline of oxamyl at the Italy site**




Y-axis units are μg/cm$^2$
X-axis units are days
parent = oxamyl
Metabolite 1 = oxamyl oxime (IN-A2213)
Metabolite 2 = DMOA (IN-D2708)

| **Oxamyl** | Average[a] | Confidence Interval (95%) |
|---|---|---|
| $DT_{50}$ [d]: | 5.3 | 4.6–6.0 |
| $DT_{90}$ [d]: | 18 | 15–20 |
| $C_0$ [μg/cm$^2$][b]: | 35 | - |
| $r^{2c}$: | 0.97 | - |
| **Oxamyl oxime (IN-A2213)** | Average[a] | Confidence Interval (95%) |
| $DT_{50}$ [d]: | 5.7 | 3.2–8.2 |
| **DMOA (IN-D2708)** | Average[a] | Confidence Interval (95%) |
| $DT_{50}$ [d]: | 3.2 | 0.8–5.7 |

[a] The residue data of three cropped and three non-cropped plots were averaged prior to regression.

[b] $C_0$ is the calculated concentration just after application, and derived from the intercept of the ordinate.

[c] $r^2$ is the coefficient of determination for the sequential fit of the oxamyl, oxamyl oxime, and DMOA data.

**Table 77 Residues of oxamyl at each depth for the cropped plot (mg/kg, dry weight basis) for the Italy site**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **10** | **14** | **20** | **28** | **48** | **62** |
| Oxamyl (0–10 cm) | DA | 0.78 | 0.26 | 0.24 | 0.08 | 0.05 | 0.025 | 0.045 | 0.011 | 0.024 | <0.005 | 0.006 |
| | DB | 0.62 | 0.14 | 0.12 | 0.029 | 0.045 | 0.016 | 0.011 | 0.008 | 0.015 | <0.005 | <0.005 |
| | DC | 0.38 | 0.16 | 0.092 | 0.026 | 0.037 | 0.026 | 0.005 | <0.005 | 0.006 | 0.006 | <0.005 |
| | Avg.[a] | **0.59** | **0.19** | **0.15** | **0.05** | **0.04** | **0.02** | **0.02** | **0.01** | **0.02** | **<0.005** | **<0.005** |
| Oxamyl (10–25 cm) | DA | 1.3 | 0.27 | 0.31 | <0.005 | 0.071 | 0.027 | 0.063 | 0.006 | <0.005 | <0.005 | <0.005 |
| | DB | 0.87 | 0.39 | 0.18 | 0.024 | 0.056 | 0.005 | 0.008 | 0.045 | 0.016 | <0.005 | <0.005 |
| | DC | 0.97 | 0.22 | 0.089 | 0.028 | 0.025 | 0.016 | 0.017 | 0.006 | <0.005 | <0.005 | ND[b] |
| | Avg. | **1.05** | **0.29** | **0.19** | **0.02** | **0.05** | **0.02** | **0.03** | **0.02** | **0.01** | **<0.005** | **<0.005** |
| Oxamyl (25–40 cm) | DA | 1.2 | 0.7 | 0.64 | 0.17 | 0.39 | 0.14 | 0.16 | 0.049 | 0.023 | <0.005 | <0.005 |
| | DB | 1.4 | 0.82 | 0.51 | 0.34 | 0.26 | 0.052 | 0.12 | 0.11 | 0.046 | <0.005 | <0.005 |
| | DC | 0.82 | 0.65 | 0.33 | 0.34 | 0.27 | 0.124 | 0.23 | 0.069 | 0.024 | <0.005 | <0.005 |
| | Avg. | **1.14** | **0.72** | **0.49** | **0.28** | **0.31** | **0.11** | **0.17** | **0.08** | **0.03** | **<0.005** | **<0.005** |
| Oxamyl (40–55 cm) | DA | 0.008 | <0.005 | 0.11 | 0.21 | 0.11 | 0.2 | 0.067 | 0.092 | 0.052 | <0.005 | <0.005 |
| | DB | 0.015 | 0.15 | 0.21 | 0.11 | 0.23 | 0.31 | 0.1 | 0.029 | 0.075 | <0.005 | <0.005 |
| | DC | <0.005 | 0.32 | 0.41 | 0.37 | 0.15 | 0.12 | 0.084 | 0.11 | 0.06 | <0.005 | <0.005 |
| | Avg. | **0.009** | **0.16** | **0.24** | **0.23** | **0.16** | **0.21** | **0.08** | **0.08** | **0.06** | **<0.005** | **<0.005** |
| Oxamyl (55–70 cm) | DA | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | 0.021 | <0.005 | <0.005 | 0.02 | <0.005 | <0.005 |
| | DB | <0.005 | <0.005 | <0.005 | <0.005 | 0.007 | 0.14 | <0.005 | <0.005 | 0.038 | <0.005 | <0.005 |
| | DC | <0.005 | <0.005 | 0.02 | <0.005 | <0.005 | <0.005 | <0.005 | 0.021 | <0.005 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **0.01** | **<0.005** | **<0.005** | **0.05** | **<0.005** | **0.01** | **0.02** | **<0.005** | **<0.005** |
| Oxamyl (70–90 cm) | DA | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | ND | ND | <0.005 | <0.005 |
| | DB | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | ND | <0.005 | <0.005 | <0.005 |
| | DC | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | ND | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |

[a] Mean values calculated by the reviewer using data from Table 1 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[b] ND = Not detected

**Table 77 Residues of oxamyl at each depth for the cropped plot (mg/kg, dry weight basis) for the Italy site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **10** | **14** | **20** | **28** | **48** | **62** |
| Oxime (0–10 cm) | DA | 0.078 | 0.049 | 0.06 | 0.015 | 0.01 | 0.008 | 0.012 | <0.005 | 0.006 | <0.005 | <0.005 |
| | DB | 0.072 | 0.027 | 0.026 | 0.01 | 0.013 | 0.006 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DC | 0.073 | 0.028 | 0.023 | 0.007 | 0.006 | 0.007 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 |
| | Avg.[a] | **0.07** | **0.03** | **0.04** | **0.01** | **0.01** | **0.01** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (10–25 cm) | DA | 0.13 | 0.032 | 0.062 | <0.005 | 0.021 | 0.012 | 0.038 | 0.008 | <0.005 | <0.005 | ND[b] |
| | DB | 0.091 | 0.07 | 0.026 | <0.005 | 0.019 | <0.005 | 0.006 | 0.022 | 0.008 | <0.005 | <0.005 |
| | DC | 0.11 | 0.031 | 0.018 | <0.005 | <0.005 | <0.005 | 0.006 | <0.005 | <0.005 | <0.005 | <0.005 |
| | Avg. | **0.11** | **0.04** | **0.04** | **<0.005** | **0.01** | **0.01** | **0.02** | **0.01** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (25–40 cm) | DA | 0.18 | 0.12 | 0.23 | 0.087 | 0.28 | 0.15 | 0.26 | 0.1 | 0.035 | <0.005 | <0.005 |
| | DB | 0.19 | 0.17 | 0.12 | 0.17 | 0.14 | 0.014 | 0.1 | 0.079 | 0.048 | <0.005 | <0.005 |
| | DC | 0.12 | 0.051 | 0.1 | 0.081 | 0.12 | 0.071 | 0.12 | 0.051 | 0.015 | <0.005 | <0.005 |
| | Avg. | **0.16** | **0.11** | **0.15** | **0.11** | **0.18** | **0.08** | **0.16** | **0.08** | **0.03** | **<0.005** | **<0.005** |
| Oxime (40–55 cm) | DA | <0.005 | <0.005 | 0.033 | 0.096 | 0.074 | 0.17 | 0.12 | 0.088 | 0.114 | <0.005 | <0.005 |
| | DB | <0.005 | 0.027 | 0.045 | 0.051 | 0.11 | 0.13 | 0.1 | 0.018 | 0.094 | <0.005 | <0.005 |
| | DC | <0.005 | 0.046 | 0.11 | 0.097 | 0.05 | 0.062 | 0.05 | 0.079 | 0.036 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **0.03** | **0.06** | **0.08** | **0.08** | **0.12** | **0.09** | **0.06** | **0.08** | **<0.005** | **<0.005** |
| Oxime (55–70 cm) | DA | ND | <0.005 | <0.005 | <0.005 | <0.005 | 0.014 | <0.005 | <0.005 | 0.048 | <0.005 | <0.005 |
| | DB | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | 0.072 | <0.005 | <0.005 | 0.044 | <0.005 | <0.005 |
| | DC | <0.005 | <0.005 | 0.006 | <0.005 | <0.005 | <0.005 | <0.005 | 0.015 | <0.005 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.03** | **<0.005** | **0.01** | **0.03** | **<0.005** | **<0.005** |
| Oxime (70–90 cm) | DA | ND | ND | ND | <0.005 | ND | <0.005 | ND | ND | <0.005 | <0.005 | <0.005 |
| | DB | ND | ND | ND | ND | ND | <0.005 | ND | ND | <0.005 | <0.005 | <0.005 |
| | DC | ND | <0.005 | <0.005 | ND | ND | ND | ND | <0.005 | ND | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |

[a] Mean values calculated by the reviewer using data from Table 1 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[b] ND = Not detected

**Table 77 Residues of oxamyl at each depth for the cropped plot (mg/kg, dry weight basis) for the Italy site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **10** | **14** | **20** | **28** | **48** | **62** |
| DMOA (0–10 cm) | DA | 0.02 | 0.016 | 0.023 | <0.01 | ND[a] | ND | ND | ND | ND | ND | ND |
| | DB | 0.024 | 0.015 | 0.016 | ND | 0.01 | ND | ND | ND | ND | ND | ND |
| | DC | 0.016 | 0.01 | 0.012 | ND | ND | ND | ND | ND | ND | ND | ND |
| | Avg.[b] | **0.02** | **0.01** | **0.02** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** |
| DMOA (10–25 cm) | DA | 0.022 | 0.013 | 0.03 | ND | <0.01 | ND | ND | ND | ND | ND | ND |
| | DB | 0.019 | 0.024 | 0.025 | <0.01 | 0.015 | ND | ND | <0.01 | ND | ND | ND |
| | DC | 0.021 | 0.012 | 0.012 | <0.01 | ND | ND | ND | ND | ND | ND | ND |
| | Avg. | **0.02** | **0.02** | **0.02** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** |
| DMOA (25–40 cm) | DA | 0.012 | 0.023 | 0.029 | 0.026 | 0.037 | 0.043 | 0.017 | <0.01 | 0.012 | ND | ND |
| | DB | 0.014 | 0.014 | 0.032 | 0.037 | 0.056 | <0.01 | 0.037 | 0.036 | 0.013 | ND | ND |
| | DC | <0.01 | 0.02 | 0.019 | 0.04 | 0.028 | 0.048 | 0.073 | 0.021 | <0.01 | ND | ND |
| | Avg. | **0.01** | **0.02** | **0.03** | **0.03** | **0.04** | **0.03** | **0.04** | **0.02** | **0.01** | **<0.01** | **<0.01** |
| DMOA (40–55 cm) | DA | ND | ND | ND | 0.027 | 0.016 | 0.046 | 0.035 | 0.098 | 0.028 | ND | ND |
| | DB | ND | ND | <0.01 | 0.015 | 0.031 | 0.045 | 0.046 | 0.012 | 0.032 | ND | ND |
| | DC | ND | <0.01 | 0.018 | 0.044 | 0.034 | 0.075 | 0.041 | 0.058 | 0.015 | ND | ND |
| | Avg. | **<0.01** | **<0.01** | **<0.01** | **0.03** | **0.03** | **0.06** | **0.04** | **0.06** | **0.03** | **<0.01** | **<0.01** |
| DMOA (55–70 cm) | DA | ND | ND | ND | ND | ND | <0.01 | ND | ND | 0.018 | ND | ND |
| | DB | ND | ND | ND | ND | ND | 0.036 | ND | ND | 0.019 | <0.01 | <0.01 |
| | DC | ND | ND | ND | ND | ND | ND | ND | 0.015 | ND | ND | ND |
| | Avg. | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **0.02** | **<0.01** | **<0.01** | **0.01** | **<0.01** | **<0.01** |
| DMOA (70–90 cm) | DA | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| | DB | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| | DC | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| | Avg. | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** |

[a] ND = Not detected

[b] Mean values calculated by the reviewer using data from Table 1 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

**Table 78 Residues of oxamyl at each depth for the bareground (non-cropped) plot (mg/kg, dry weight basis) for the Italy site**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **10** | **14** | **20** | **28** | **48** | **62** |
| Oxamyl (0–10 cm) | DA | 0.44 | 0.18 | 0.098 | 0.047 | 0.045 | 0.016 | 0.043 | 0.019 | 0.017 | <0.005 | <0.005 |
| | DB | 0.5 | 0.13 | 0.14 | 0.072 | 0.052 | 0.047 | 0.019 | 0.007 | <0.005 | <0.005 | 0.006 |
| | DC | 0.32 | 0.44 | 0.11 | 0.023 | 0.018 | 0.017 | 0.016 | 0.024 | 0.005 | 0.008 | 0.006 |
| | Avg.[a] | **0.42** | **0.25** | **0.12** | **0.05** | **0.04** | **0.03** | **0.03** | **0.02** | **0.01** | **<0.005** | **<0.005** |
| Oxamyl (10–25 cm) | DA | 0.79 | 0.38 | 0.15 | 0.051 | 0.029 | 0.007 | 0.025 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DB | 0.85 | 0.32 | 0.28 | 0.18 | 0.058 | 0.085 | 0.026 | <0.005 | ND[b] | <0.005 | <0.005 |
| | DC | 0.55 | 0.47 | 0.15 | 0.027 | 0.022 | <0.005 | <0.005 | 0.01 | <0.005 | <0.005 | <0.005 |
| | Avg. | **0.73** | **0.39** | **0.19** | **0.09** | **0.04** | **0.03** | **0.02** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxamyl (25–40 cm) | DA | 1.5 | 1.1 | 0.6 | 0.43 | 0.11 | 0.087 | 0.17 | 0.036 | 0.011 | <0.005 | <0.005 |
| | DB | 1.6 | 0.82 | 0.62 | 0.38 | 0.2 | 0.19 | 0.089 | 0.027 | <0.005 | <0.005 | <0.005 |
| | DC | 1.1 | 0.64 | 0.46 | 0.25 | 0.28 | 0.12 | 0.092 | 0.084 | 0.01 | <0.005 | <0.005 |
| | Avg. | **1.40** | **0.85** | **0.56** | **0.35** | **0.20** | **0.13** | **0.12** | **0.05** | **0.01** | **<0.005** | **<0.005** |
| Oxamyl (40–55 cm) | DA | 0.007 | 0.034 | 0.33 | 0.17 | 0.13 | 0.23 | 0.14 | 0.13 | 0.06 | <0.005 | <0.005 |
| | DB | 0.016 | 0.34 | 0.12 | 0.16 | 0.26 | 0.22 | 0.094 | 0.092 | 0.037 | <0.005 | <0.005 |
| | DC | 0.006 | <0.005 | 0.28 | 0.4 | 0.022 | 0.25 | 0.061 | 0.084 | 0.052 | <0.005 | <0.005 |
| | Avg. | **0.01** | **0.13** | **0.24** | **0.24** | **0.14** | **0.23** | **0.10** | **0.10** | **0.05** | **<0.005** | **<0.005** |
| Oxamyl (55–70 cm) | DA | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | 0.033 | 0.025 | 0.02 | <0.005 | <0.005 | <0.005 |
| | DB | <0.005 | <0.005 | <0.005 | <0.005 | 0.006 | 0.01 | <0.005 | 0.019 | 0.018 | <0.005 | <0.005 |
| | DC | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | 0.085 | <0.005 | 0.005 | 0.012 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.04** | **0.01** | **0.01** | **0.01** | **<0.005** | **<0.005** |
| Oxamyl (70–90 cm) | DA | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | ND | 0.005 | <0.005 |
| | DB | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | ND | <0.005 | <0.005 | <0.005 | ND |
| | DC | <0.005 | <0.005 | <0.005 | <0.005 | ND | <0.005 | <0.005 | ND | <0.005 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |

[a] Mean values calculated by the reviewer using data from Table 1 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[b] ND = Not detected

**Table 78 Residues of oxamyl at each depth for the bareground (non-cropped) plot (mg/kg, dry weight basis) for the Italy site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **10** | **14** | **20** | **28** | **48** | **62** |
| Oxime (0–10 cm) | DA | 0.065 | 0.032 | 0.02 | 0.014 | 0.012 | <0.005 | 0.01 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DB | 0.082 | 0.024 | 0.019 | 0.026 | 0.017 | 0.025 | 0.01 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DC | 0.056 | 0.053 | 0.02 | 0.006 | <0.005 | 0.006 | <0.005 | 0.006 | <0.005 | <0.005 | <0.005 |
| | Avg.[a] | **0.07** | **0.04** | **0.02** | **0.02** | **0.01** | **0.01** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (10–25 cm) | DA | 0.098 | 0.066 | 0.019 | 0.011 | 0.008 | <0.005 | 0.006 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DB | 0.1 | 0.045 | 0.052 | 0.056 | 0.025 | 0.033 | 0.012 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DC | 0.053 | 0.040 | 0.024 | 0.008 | <0.005 | 0.014 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 |
| | Avg. | **0.08** | **0.05** | **0.03** | **0.03** | **0.01** | **0.02** | **0.01** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |
| Oxime (25–40 cm) | DA | 0.19 | 0.21 | 0.15 | 0.2 | 0.084 | 0.096 | 0.12 | 0.02 | 0.011 | <0.005 | <0.005 |
| | DB | 0.19 | 0.17 | 0.13 | 0.27 | 0.18 | 0.17 | 0.15 | 0.023 | 0.015 | <0.005 | <0.005 |
| | DC | 0.11 | 0.082 | 0.11 | 0.11 | 0.058 | 0.088 | 0.068 | 0.044 | 0.012 | <0.005 | <0.005 |
| | Avg. | **0.16** | **0.15** | **0.13** | **0.19** | **0.11** | **0.12** | **0.11** | **0.03** | **0.01** | **<0.005** | **<0.005** |
| Oxime (40–55 cm) | DA | <0.005 | <0.005 | 0.092 | 0.074 | 0.078 | 0.21 | 0.11 | 0.14 | 0.085 | <0.005 | <0.005 |
| | DB | <0.005 | 0.059 | 0.026 | 0.096 | 0.23 | 0.15 | 0.15 | 0.1 | 0.19 | <0.005 | <0.005 |
| | DC | <0.005 | <0.005 | 0.073 | 0.186 | 0.007 | 0.21 | 0.054 | 0.054 | 0.12 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **0.02** | **0.06** | **0.12** | **0.11** | **0.19** | **0.10** | **0.10** | **0.13** | **<0.005** | **<0.005** |
| Oxime (55–70 cm) | DA | <0.005 | ND[b] | <0.005 | <0.005 | <0.005 | 0.035 | 0.019 | 0.033 | 0.006 | <0.005 | <0.005 |
| | DB | <0.005 | <0.005 | <0.005 | <0.005 | 0.008 | 0.007 | <0.005 | 0.019 | 0.09 | <0.005 | <0.005 |
| | DC | ND | ND | <0.005 | <0.005 | <0.005 | 0.092 | <0.005 | <0.005 | 0.038 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **0.04** | **0.01** | **0.02** | **0.04** | **<0.005** | **<0.005** |
| Oxime (70–90 cm) | DA | ND | ND | <0.005 | ND | <0.005 | <0.005 | <0.005 | <0.005 | ND | <0.005 | <0.005 |
| | DB | <0.005 | ND | ND | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 | <0.005 |
| | DC | <0.005 | ND | <0.005 | <0.005 | ND | <0.005 | <0.005 | ND | <0.005 | <0.005 | <0.005 |
| | Avg. | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** | **<0.005** |

[a] Mean values calculated by the reviewer using data from Table 1 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[b] ND = Not detected

**Table 78 Residues of oxamyl at each depth for the bareground (non-cropped) plot (mg/kg, dry weight basis) for the Italy site (continued)**

| Analyte (soil depth) | Rep. | Days after treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **3** | **5** | **7** | **10** | **14** | **20** | **28** | **48** | **62** |
| DMOA (0–10 cm) | DA | 0.021 | 0.018 | 0.014 | <0.01 | <0.01 | ND[a] | <0.01 | <0.01 | <0.01 | ND | ND |
| | DB | 0.019 | 0.011 | 0.023 | 0.011 | <0.01 | <0.01 | ND | ND | ND | ND | ND |
| | DC | 0.014 | 0.021 | 0.011 | ND | ND | ND | ND | ND | ND | ND | ND |
| | Avg.[b] | **0.02** | **0.02** | **0.02** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** |
| DMOA (10–25 cm) | DA | 0.023 | 0.022 | 0.019 | <0.01 | <0.01 | ND | ND | ND | ND | ND | ND |
| | DB | 0.024 | 0.021 | 0.036 | 0.043 | 0.015 | 0.019 | ND | ND | ND | ND | ND |
| | DC | 0.016 | 0.023 | 0.014 | ND | ND | ND | ND | ND | ND | ND | ND |
| | Avg. | **0.02** | **0.02** | **0.02** | **0.02** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** |
| DMOA (25–40 cm) | DA | 0.014 | 0.017 | 0.038 | 0.033 | 0.026 | 0.032 | 0.026 | <0.01 | ND | ND | ND |
| | DB | 0.017 | 0.022 | 0.043 | 0.051 | 0.056 | 0.076 | 0.062 | 0.012 | ND | ND | ND |
| | DC | <0.01 | 0.019 | 0.02 | 0.026 | 0.061 | 0.045 | 0.022 | 0.025 | ND | ND | ND |
| | Avg. | **0.01** | **0.02** | **0.03** | **0.04** | **0.05** | **0.05** | **0.04** | **0.01** | **<0.01** | **<0.01** | **<0.01** |
| DMOA (40–55 cm) | DA | ND | ND | 0.018 | 0.014 | 0.027 | 0.051 | 0.059 | 0.06 | 0.048 | ND | ND |
| | DB | ND | <0.01 | <0.01 | 0.019 | 0.038 | 0.048 | 0.091 | 0.065 | 0.036 | ND | ND |
| | DC | ND | ND | 0.011 | 0.027 | ND | 0.069 | 0.042 | 0.069 | 0.035 | ND | ND |
| | Avg. | **<0.01** | **<0.01** | **0.01** | **0.02** | **0.02** | **0.06** | **0.06** | **0.06** | **0.04** | **<0.01** | **<0.01** |
| DMOA (55–70 cm) | DA | ND | ND | ND | ND | ND | 0.015 | 0.013 | 0.021 | 0.01 | ND | ND |
| | DB | ND | ND | ND | ND | ND | <0.01 | ND | 0.059 | 0.056 | ND | ND |
| | DC | ND | ND | ND | ND | ND | 0.025 | ND | 0.013 | 0.022 | ND | ND |
| | Avg. | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **0.02** | **<0.01** | **0.03** | **0.03** | **<0.01** | **<0.01** |
| DMOA (70–90 cm) | DA | ND | n.a.[c] | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| | DB | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| | DC | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| | Avg. | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** | **<0.01** |

[a] ND = Not detected

[b] Mean values calculated by the reviewer using data from Table 1 of the final report. For calculation purposes, residue results reported as non-quantifiable (for one or two individual replicates) were assigned values of 0.0025 mg/kg for oxamyl and oxamyl oxime and 0.005 mg/kg for DMOA, representing ½ the LOQ.

[c] n.a. = Not analysed.

**Table 79 Residues of oxamyl in soil from bare soil and cropped plots, combined and expressed in terms of mass per area for the Italy site**

| Time (DALA) | Oxamyl (µg/cm$^2$) | Oxamyl oxime[a] (IN-A2213) (µg/cm$^2$) | DMOA[a] (IN-D2708) (µg/cm$^2$) | Oxamyl (µg/cm$^2$) |
|---|---|---|---|---|
| 0 | 54.4 | 8.98 | 1.74 | [65.1][b] |
| 1 | 32.6 | 6.91 | 2.03 | 32.6 |
| 3 | 25.2 | 8.64 | 3.22 | 25.2 |
| 5 | 15.6 | 9.2 | 3.25 | 15.6 |
| 7 | 12.3 | 8.79 | 3.66 | 12.3 |
| 10 | 10.2 | 9.9 | 5.21 | 10.2 |
| 14 | 6.98 | 8.88 | 4.77 | 6.98 |
| 20 | 4.51 | 5.11 | 4.59 | 4.51 |
| 28 | 2.52 | 6.03 | 2.81 | 2.52 |
| 48 | 0.115 | 0 | 0 | 0.12 |
| 62 | 0.064 | 0 | 0 | 0.07 |

[a] Values given in oxamyl equivalents.

[b] The initial concentration includes oxamyl equivalents of the metabolites.

## III. CONCLUSIONS

A field soil dissipation study was conducted with Oxamyl 10SL insecticide and nematicide in a greenhouse at one site in Sicily, Italy. The greenhouse was located in a region that exhibited typical conditions of vegetable growing with respect to cultivation, soil and climate. The data compiled demonstrate the environmental fate of oxamyl and its major degradates oxamyl oxime (IN-A2213) and DMOA (IN-D2708) under these conditions.

Oxamyl 10SL (liquid formulation containing 10% of oxamyl as the active substance) was applied to the bare ground of each a cropped and non-cropped area each of three plots at an application rate of 15 L/ha anticipated for nematode control in greenhouse-cultivation. The rate corresponds to nominal 1.5 kg of oxamyl per hectare. The test item was applied by chemigation drip irrigation equipment and technique.

The climate conditions within the greenhouse were characterized by warm days, cool nights, and high humidity throughout the two months during which oxamyl degradation was observed. The plots were irrigated by drip irrigation frequently, according to local practice for cucumber cultivation. The cropped and the non-cropped units were irrigated equally.

Soil cores from the treated and control plots were collected randomly to a depth of 90 cm before application and at seventeen time points during one year following the treatment. The samples were analysed for residues of oxamyl and oxamyl oxime by LC-MS/MS and for IN-D2708 by a different method using LC-MS technique. The Limit of Quantitation (LOQ) was 0.005 mg/kg for oxamyl and oxamyl oxime and 0.01 mg/kg for DMOA.

No residues were detected in any sample taken before the application and in the soil from the untreated control plot. Oxamyl residue levels declined rapidly to concentrations below 10% of the original applied test item within one month. First-order dissipation curves were calculated for each analyte separately for the cropped and the non-cropped plots. Based on plot average soil concentrations, the calculated first-order $DT_{50}$ value was 5.3 days for oxamyl, 5.7 days for the degradate oxamyl oxime (IN-A2213), and 3.2 days for DMOA (IN-D2708). The corresponding $DT_{90}$ value of the parent compound was 18 days.

Oxamyl and its degradates leached to a depth of 55 cm, even on the day of chemigation. This can be explained by the sandy nature of the soil and the high volume of water that was applied to ensure an even application of the test item. During the next month of the study, the analytes were detected in addition in the fifth horizon 55–70 cm, but no analyte was determined above the Limit of Quantitation in the lowest horizon 70–90 cm, nor laterally away from the sampling zone at 30 cm or more. Since soil residues were tracked up until one clean layer, the calculated half-lives are understood as true degradation half-lives, rather than dissipation half-lives.

**Storage stability of oxamyl residues in soil**

The following two studies were conducted to demonstrate the storage stability of oxamyl, IN-A2213, and IN-D2708 residues in soils. The analytical method was that used in field dissipation studies.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.2.1/05**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.1.2.2.1/05** | **Report:** | Zietz, E. (2003); Determination of the storage stability of oxamyl (DPX-D1410) and its metabolite oxamyl oxime (IN-A2213) in soil samples from a field soil dissipation study conducted in the United Kingdom, season 2000<br><br>**DuPont Report No.:** DuPont-10099<br><br>**Guidelines:** EU 7032/VI/1995 Rev 5 (1997) App. H<br><br>**Deviations:** None<br><br>**Testing Facility:** Institut Fresenius Chemische und Biologische Laoratorien AG, Taunusstein, Germany<br><br>**Testing Facility Report No.:** IF-101/38560-00<br><br>**GLP:** Yes<br><br>**Certifying Authority:** Hessisches Ministerium fur Umwelt, Landlichen Raum und Verbraucherschutz |

**Executive summary:**

A field soil dissipation study was conducted with oxamyl in which the soil containing residues of the parent compound and its metabolite oxamyl oxime (IN-A2213) were stored frozen while awaiting analysis. In order to demonstrate that the residues in these studies did not change as a result of freezer storage, a storage stability study was conducted on representative soils which were fortified with oxamyl and soil metabolite oxamyl oxime and stored frozen under conditions similar to those used for the field and laboratory study samples. These fortified samples were analysed periodically over a 22 month period. This stability study was designed to cover the longest interval between sample collection and the analysis of these soils for the residues of oxamyl and soil degradate oxamyl oxime, which were stored frozen at ≤-18°C.

Soil representative of field sites chosen for the field dissipation study was selected for this study. The soil was obtained from the field dissipation study site in Spalding, England (summarised in Point B.8.1.2.2.1 in this document). Soil samples were fortified with oxamyl and oxamyl oxime at a level of 100 ppb for each analyte and stored at ≤-18°C. The samples were analysed at 0 days, 2.4, 2.8, 14.3, and 22.2 months over an approximately 22-month period.

Samples were analysed using the analytical method that had been used in the analysis of dissipation study samples, *viz.*, DuPont-2392, summarised in the Oxamyl EU Renewal Dossier, Document M-CA, Section 4, DuPont-40931 EU. This method has an LOQ of 5.0 ppb for each analyte. Residues of oxamyl and oxamyl oxime were extracted from soil with an 80:20 acetonitrile:methanol solution, acidified with 0.01% of formic acid, concentrated under a stream of nitrogen, appropriately diluted with 0.01% of formic acid in water and passed through a membrane filter, they were analysed by LC/MS/MS. Fresh fortified soil samples were analysed concurrently in each analysis set. The average recoveries in fresh fortified (FF) soil samples and the frozen aged fortified samples (AF) fortified for both soil types were statistically equivalent (summarised below) over the entire 22-month period.

**Table 80 Percent recoveries from field dissipation study in England**

| | Percent recoveries | |
|---|---|---|
| | **Oxamyl** | **Oxamyl oxime** |
| AVG FF[a] recoveries | 93 | 90 |
| FF RSD[a] | 9 | 4 |
| AVG AF[a] recoveries | 86 | 84 |
| AF RSD[a] | 6 | 5 |
| AVG normalised recoveries | 99 | 96 |
| Normalised RSD[a] | 10 | 5 |

[a] FF = Fresh fortification AF = Aged fortification RSD = Relative standard deviation

Statistical analysis showed that the averaged fresh fortification recoveries as well as the frozen fortification recoveries were in the 84 to 99% range, demonstrating that both oxamyl and the oxime (IN-A2213) are stable under frozen storage conditions for 22 months. There were no discernible trends of decreasing recoveries in the frozen stored samples over time.

It can be concluded from this study that the residues of oxamyl and its soil metabolite oxamyl oxime were found to be stable in soil for at least 22 months when stored at temperatures of ≤-18°C. No significant decline was observed during this period.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: Oxamyl technical
   Lot/Batch # and purity: D1410-376 99.9%
   A2213-11 100.0%
   Description: Oxamyl White powder
   Oxamyl oxime (IN-A2213) Solid white crystals
   CAS #: 23135-22-0

2. Test and reference substances

   The test substances used in this study also served as the reference standards during analysis because pure technical materials were used as the test and reference substances.

3. Test soils

   Untreated soil representative of the soil types in all field dissipation and laboratory degradation studies was used. This soil was collected from the untreated areas near the test plots used in the field dissipation study summarised in DuPont-3026, Point B.8.1.2.2.1 in this document. Silty loam from Spalding, England, was used in the field dissipation study. Soil characteristics for the uppermost depths, are included in Table .

**Table 81 Soil characteristics of the soils used for storage stability**

| | |
|---|---|
| % Sand (2 mm–50 μm)[a] | 23.4 |
| % Silt (50 μm–2 μm)[a] | 66.1 |
| % Clay (<2 μm)[a] | 10.6 |
| CEC (meq/100 g) | 1.6 |
| % Moisture 0 bar[b] | 42.1 |
| % Organic matter[c] | 2.2 |
| pH[d] | 7.33 |
| Soil classification[e] | Silty loam |

[a] Particle size
[b] Moisture capacity of air dried soil
[c] Walkley-Black method
[d] pH in water
[e] Soil classification according to USDA system

B. EXPERIMENTAL DESIGN

1. Experimental design

The soils were fortified with oxamyl or oxamyl oxime. All samples were fortified at approximately 100 ppb, which was 20 times the LOQ of both analytes.

Samples were analysed at 0 days, 2.4, 2.8, 14.3, and 22.2 months over approximately 22 months.

Soils were fortified with oxamyl or oxamyloxime and placed in frozen storage in glass jars in 20 g amounts.

Twenty gram portions of control soil were also weighed into glass jars for each soil type to be used as control and fresh fortifications for each analysis interval. Two untreated control samples were saved for use as controls and fresh fortification with each analysis set.

2. Description of analytical method

Samples were analysed for oxamyl and oxamyl oxime based on LC-MS/MS, using minor modifications to the method (Oxamyl EU Renewal Dossier, Document M-CA, Section 4, Point 4.1.2, DuPont-40931 EU). Oxamyl and its oxime degradate were extracted from approximately 20 g of fortified and stored soil samples into acetonitrile/methanol (80:20), which was acidified with 0.01 % of formic acid, using the Dionex Accelerated Solvent Extraction ASE® 200. The samples were split into two equal portions that were transferred into two ASE extraction cells and extracted separately. Thereafter, the corresponding extracts were combined. An aliquot of the soil extract was concentrated under a stream of nitrogen, diluted appropriately with 0.01 % of formic acid in water, and passed through a membrane filter. An aliquot of the filtered extract was injected into the liquid chromatograph and determined using a MS/MS detector. The daughter ion (m/z 71.70) of the ammonium (N~ +) adduct ion of oxamyl (m/z 237) and daughter ion (m/z 89.65) of the protonated oxamyl oxime ion (m/z 163) were determined using electrospray ionization.

## II. RESULTS AND DISCUSSION

Fresh fortification (FF) recoveries as well as the aged fortification recoveries are listed in Table  for both analytes. Oxamyl recoveries were in 77–92% range. Averaged recoveries, as well as the standard deviations, in fresh fortified samples as well as aged fortified samples were quite similar (freezer average 86% vs fresh average of 83%). Oxamyl oxime recoveries were in 72-87% range. Averaged recoveries, as well as the standard deviations, in fresh fortified samples as well as aged fortified samples were quite similar (freezer average 84% vs. fresh average of 90%). Recovery ranges for both compounds stored frozen for up to 22 month, are summarised briefly in the following table.

**Table 82 Percent recovery range, freezer stored samples for each analyte**

| Analyte | Overall average recovery (%) | Relative standard deviation (%) |
|---|---|---|
| Oxamyl | 86 | 6 |
| Oxamyl oxime (IN-A2213) | 84 | 4 |

All aged fortified samples through 22 months showed recoveries in the 70 to 120% range for all analytes as did the fresh fortified samples.

Individual recoveries in fresh fortified and frozen fortified samples were also compared after normalisation of AF recoveries using FF recoveries. Normalised recoveries in AF samples for each analyte are also included in Table . Normalised recoveries for both analytes were in the 92 to 101% range.

**Table 83 Recoveries of oxamyl and oxamyl oxime in fresh fortified and frozen soil samples**

| Storage interim (months) | Sample ID | Recovery (%) | | | | Normalised recovery[a] (%) | |
|---|---|---|---|---|---|---|---|
| | | Freezer stored | | Fresh | | | |
| | | Oxamyl | Oxamyl oxime | Oxamyl | Oxamyl oxime | Oxamyl | Oxamyl oxime |
| 0 | 100TG217646-A | — | — | 99 | — | 100 | — |
| | 100TG217646-B | — | — | 94 | — | | |
| | 100TG217646-C | — | — | 101 | — | | |
| 0 | 100TG217646-D | — | — | — | 92 | — | 100 |
| | 100TG217646-E | — | — | — | 92 | | |
| | 100TG217646-F | — | — | — | 92 | | |
| 0 | 100TG217646-A | — | — | 99 | — | b | b |
| | 100TG217646-B | — | — | 94 | — | | |
| | 100TG217646-C | — | — | 101 | — | | |
| 0 | 100TG217646-D | — | — | — | 92 | b | b |
| | 100TG217646-E | — | — | — | 92 | | |
| | 100TG217646-F | — | — | — | 92 | | |
| 2.4 | 101TG046680-11 | — | 86 | — | — | — | 92 |
| | 101TG046680-12 | — | 75 | — | — | | |
| | 101TG046680-23 | — | — | — | 87 | b | b |
| 2.8 | 101TG046680-3 | 88 | — | — | — | 94 | — |
| | 101TG046680-4 | 92 | — | — | — | | |
| | 101TG046680-25 | — | — | 96 | — | b | b |
| 14.3 | 101TG046680-07 | 90 | — | — | — | 110 | — |
| | 101TG046680-08 | 86 | — | — | — | | |
| | 101TG046680-15 | — | 87 | — | — | — | 94 |
| | 101TG046680-16 | — | 86 | — | — | | |
| | 101TG046680-30A | — | — | 80 | — | b | b |
| | 101TG046680-33A | — | — | — | 91 | b | b |
| 22.2 | 101TG046680-9 | 77 | — | — | — | 93 | — |
| | 101TG046680-10 | 82 | — | — | — | | |
| | 101TG046680-17 | — | 84 | — | — | — | 101 |
| | 101TG046680-18 | — | 84 | — | — | | |
| | 101TG046680-35 | — | — | 86 | — | b | b |
| | 101TG046680-36 | — | — | — | 83 | b | b |

[a] Normalised averages means that the recoveries determined within the fortified and stored samples were corrected by the recoveries of the freshly fortified samples.

[b] Normalisation not performed on fresh samples.

## III. CONCLUSION

Oxamyl (DPX-D1410) and its soil metabolite oxamyl oxime (IN-A2213) were found to be stable in soil for at least 22 months when stored at temperatures of ≤-18°C. No significant decline in soil residues was observed during this storage period.

(Zietz, E., 2004)

**Study submitted to the EU for the first time in this submission**

**B.8.1.2.2.1/06**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.1.2.2.1/02** | **Report:** | Mol, J.G.J. (2003); Freezer storage stability of oxamyl, IN-A2213 and IN-D2708 residues in soil samples from field soil dissipation studies conducted in Spain and Italy, season 2000<br><br>**DuPont Report No.:** DuPont-9342<br><br>**Guidelines:** Not given<br><br>**Deviations:** None<br><br>**Testing Facility:** TNO Nutrition and Food Research, Utrechtseweg, The Netherlands<br><br>**Testing Facility Report No.:** V4423<br><br>**GLP:** Yes<br><br>**Certifying Authority:** Inspectorate for Health Protection and Veterinary Public Health, Ministry of Health, Welfare and Sport (the Netherlands) |

**Executive summary:**

Two field soil dissipation studies were conducted with oxamyl in which the soils containing residues of the parent compound and its metabolites were stored frozen while awaiting analysis. In order to demonstrate that the residues in these studies did not change as a result of freezer storage, a storage stability study was conducted on two representative soils which were fortified with oxamyl and its metabolites IN-A2213 and IN-D2708 (oxamyl-oxime and DMOA) and stored frozen under conditions similar to those used for the field study samples. These fortified samples were analysed periodically over an 18 month period. This stability study was designed to cover the longest interval between sample collection and the analysis of these soils for the residues of oxamyl and soil degradates (oxamyl-oxime and DMOA), which were stored frozen at <-18°C.

Two soils, representative of field sites chosen for the field dissipation studies, were selected for this study. The soils were obtained from the field dissipation study sites in Spain (summarised in Point B.8.1.2.2.1 in this document) and Italy (summarised in Point B.8.1.2.2.1 in this document). Soil samples were fortified with oxamyl, oxamyl-oxime, and DMOA at a level of 100 ppb for each analyte and stored at approximately <-18°C. The samples were analysed at 0 days, and at 2, 5, 6, 11, 12, and 18 months over an 18-month period.

Oxamyl and oxamyl-oxime (IN-A2213) samples were analysed using the analytical method which had been used in the analysis of dissipation study samples, *viz*., DuPont-7191. This method has an LOQ of 5 ppb for each analyte. Residues were extracted from soil with a mixture of methanol, acetonitrile and formic acid and analysed by LC-MS/MS.

DMOA (IN-D2708) samples were analysed using the analytical method that had been used in the analysis of dissipation study samples, *viz*., DuPont-4800 and DuPont-4719. This method has an LOQ of 10 ppb for each analyte. Residues were extracted from soil with a mixture of methanol, water, glacial acetic acid, ammonium carbonate and ammonium acetate and analysed by LC-MS/MS.

Fresh fortified soil samples were analysed concurrently in each analysis set. The average recoveries in fresh fortified (FF) soil samples and the frozen aged fortified samples (AF) fortified for both soil types were statistically equivalent (summarised below) over the entire 18-month period.

**Table 40 Percent recoveries from field dissipation studies in Spain and Italy**

| | Percent recoveries | | |
|---|---|---|---|
| | **Oxamyl** | **Oxamyl-oxime** | **DMOA** |
| AVG FF[a] recoveries | 100 | 91 | 92 |
| FF SD[a] | 11 | 16 | 8 |
| AVG AF[a] recoveries | 96 | 82 | 89 |
| AF SD[a] | 15 | 13 | 6 |

[a] FF = Fresh fortification AF = Aged fortification SD = Standard deviation

Statistical analysis showed that the averaged fresh fortification recoveries as well as the frozen fortification recoveries were in the 80 to 100% range, demonstrating that both oxamyl and the oxime (IN-A2213) are stable under frozen storage conditions for 22 months. There were no discernible trends of decreasing recoveries in the frozen stored samples over time.

It can be concluded from this study that the residues of oxamyl and DMOA are stable in frozen soil for at least 18 months and that while residues of oxamyl-oxime were stable for at least 18 months in one of the two soils the recoveries obtained at 18 months for the other soil suggest a more limited stability of at least 12 months.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: Oxamyl technical

| | | |
|---|---|---|
| Lot/Batch # and purity: | D1410-376 | 99.9% |
| | A2213-11 | 100.0% |
| | D2708-6 | 99.6% |
| Description: | Oxamyl | White solid |
| | Oxamyl oxime | Solid crystals, white |
| | DMOA | Solid crystals, white |
| CAS #: | 23135-22-0 | |

2. Test and reference substances

The test substances used in this study also served as the reference standards during analysis because pure technical materials were used as the test and reference substances. In addition, d6-DMOA (BZQ22) served as an additional internal standard.

3. Test soils

Two soils representative of the soil types in all field dissipation and laboratory degradation studies were used. These soils were collected from sites used for field soil dissipation trials with oxamyl. Soil from Spain was used in the field dissipation study summarised in DuPont-4719, Point B.8.1.2.2.1 in this document. Soil from Italy was used for the dissipation study reported in DuPont-4800, Point B.8.1.2.2.1 in this document. Soil characteristics for the uppermost depths, are included in

.

Both soils had a pH of 7.4–7.79, and choice of slightly alkaline pH for both soils was made because it represented the worst case scenario for residue stability. Oxamyl and some metabolites degrade via hydrolytic reactions, which are assisted by alkaline pH, hence pH >7 would be the worst case for storage stability.

**Table 85 Soil characteristics of the soils used for storage stability**

| | Spain soil | | Italy soil | |
|---|---|---|---|---|
| | Soil depth (cm) | | | |
| **Soil property** | **0-10** | **10-25** | **0-10** | **10-25** |
| Sand % (0.063-2 mm)[a] | 71.7 | 67.7 | 87.9 | 89.3 |
| Silt % (0.002-0.063 mm)[a] | 20.0 | 22.1 | 6.2 | 5.2 |
| Clay % (<0.002 mm)[a] | 8.3 | 10.2 | 5.9 | 5.5 |
| pH (0.01M $CaCl_2$) | 7.79 | 7.72 | 7.4 | 7.5 |
| % Total Organic Carbon (TOC)[b] | 2.0 | 1.4 | 0.7 | 0.5 |
| % Organic matter[c] | 3.5 | 2.4 | 1.2 | 0.8 |
| C.E.C [meq/100g][d] | 10.0 | 7.1 | 5.9 | 5.7 |
| Bulk density (g $cm^{-3}$) [e] | 1.93 | - | 2.07 | - |
| Water Holding Capacity (% of dry wt.) | 43.1 | 38.4 | 31.9 | 35.6 |
| Soil Classification[f] | Medium loamy sand | | Sand | |

[a] Particle size distribution (%)
[b] Organic carbon content [% or g/100 g of dry soil]
[c] TOC × 1.72 (%)
[d] Cation Exchange Capacity (C.E.C)
[e] The density at the original position in the field (horizon 0–12 cm), average of eight spots in the field taken into account
[f] Soil classification according to USDA system

B. EXPERIMENTAL DESIGN

1. Experimental design

The soils were fortified with oxamyl, oxamyl oxime, and DMOA. All samples were fortified at 100 ppb, which was 20 times the LOQ of oxamyl and oxamyl-oxime and 10 times the LOQ of DMOA.

Samples were analysed at 0 days, 2, 4–6, 12, and 18 months over 18 months.

Soils were fortified with oxamyl, oxamyl-oxime, or DMOA and placed in frozen storage in glass jars in 20 g amounts.

Twenty gram portions of control soil were also weighed into glass jars for each soil type to be used as control and fresh fortifications for each analysis interval. Two untreated control samples were saved for use as controls and fresh fortification with each analysis set.

2. Description of analytical method

Samples were analysed for oxamyl and oxamyl-oxime based on HPLC with MS/MS detection, according to the procedures validated in the Oxamyl EU Renewal Dossier, Document M-CA, Section 4, Point 4.2.3, DuPont-40931 EU (DuPont-7191). Homogenized soil was extracted with a mixture of methanol/acetonitrile/formic acid. After preheating of the soil/solvent mixture to 50°C, the extraction was carried out on a mechanical shaker. After settling of suspended solids, an aliquot of the clear extract was evaporated to dryness and the residue reconstituted in mobile phase. Analysis was performed using reversed phase HPLC with MS/MS detection. The triple-quadrupole mass spectrometer was equipped with an electrospray interface operating in the positive ion mode. One precursor → product ion transition was monitored for each analyte.

Samples were analysed for DOMA based on HPLC with MS detection, according to the procedures validated in DuPont-4800 and DuPont-4719 (summarised in Point B.8.1.2.2.1 in this document). DMOA was extracted with an aqueous solvent under basic conditions. The extract was cleaned by SPE and then analysed by LC-MS.

## II. RESULTS AND DISCUSSION

Fresh fortification (FF) recoveries as well as the aged fortification recoveries are listed in **Errore. L'origine riferimento non è stata trovata.** for all three analytes. Recoveries in aged fortified (AF) samples stored under frozen conditions were within the range of recoveries observed for the analytical method.

**Table 86 Recoveries of oxamyl, oxamyl-oxime, and DMOA in fresh fortified and frozen soil samples**

| Storage interim (months) | Soil ID | Extraction date | Recovery (%) | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Freezer stored | | | Fresh | | |
| | | | Oxamyl | Oxamyl -oxime | DMOA | Oxamyl | Oxamyl -oxime | DMOA |
| 0 days | Spain | 23-Jun-01 | — | — | — | 96 | 89 | a |
| | | 23-Jun-01 | — | — | — | 99 | 107 | a |
| | | 23-Jun-01 | — | — | — | 110 | 102 | a |
| 2 | Spain | 28-Aug-01 | — | — | — | 111 | 110 | — |
| | | 28-Aug-01 | 108 | 99 | — | — | — | — |
| | | 28-Aug-01 | 118 | 111 | — | — | — | — |
| 6 | Spain | 13-Dec-01 | — | — | — | 125 | 116 | — |
| | | 13-Dec-01 | 103 | 95 | — | — | — | — |
| | | 13-Dec-01 | 120 | 96 | — | — | — | — |
| 12 | Spain | 26-Jun-02 | — | — | — | 89 | 95 | — |
| | | 26-Jun-02 | 81 | 74 | — | — | — | — |
| | | 26-Jun-02 | 82 | 76 | — | — | — | — |
| | Spain | 04-Jul-02 | — | — | — | — | — | 95 |
| | | 04-Jul-02 | — | — | 90 | — | — | — |
| | | 04-Jul-02 | — | — | 89 | — | — | — |
| 18 | Spain | 10-Dec-02 | — | — | — | 102 | 95 | — |
| | | 10-Dec-02 | 81 | 67 | — | — | — | — |
| | | 10-Dec-02 | 71 | 64 | — | — | — | — |
| | Spain | 12-Dec-02 | — | — | — | — | — | 84 |
| | | 12-Dec-02 | — | — | 89 | — | — | — |
| | | 12-Dec-02 | — | — | 79 | — | — | — |
| 0 days | Italy | 04-Feb-02 | — | — | — | 91 | 71 | 86 |
| | | 04-Feb-02 | — | — | — | 97 | 78 | 85 |
| | | 04-Feb-02 | — | — | — | 84 | 69 | 84 |
| 5 | Italy | 26-Jun-02 | — | — | — | 95 | 87 | — |
| | | 26-Jun-02 | 90 | 77 | — | — | — | — |
| | | 26-Jun-02 | 92 | 75 | — | — | — | — |
| 5 | Italy | 27-Jun-02 | — | — | — | — | — | 95 |
| | | 27-Jun-02 | — | — | 87 | — | — | — |
| | | 27-Jun-02 | — | — | 87 | — | — | — |
| 11 | Italy | 10-Dec-02 | — | — | — | — | — | 87 |
| | | 10-Dec-02 | — | — | 87 | — | — | — |
| | | 10-Dec-02 | — | — | 87 | — | — | — |
| 11 | Italy | 12-Dec-02 | — | — | — | 87 | 80 | — |
| | | 12-Dec-02 | 109 | 86 | — | — | — | — |
| | | 12-Dec-02 | 93 | 76 | — | — | — | — |
| 18 | Italy | 22-Jul-03 | — | — | — | 103 | 92 | 103 |
| | | 22-Jul-03 | 88 | 81 | 101 | — | — | — |
| | | 22-Jul-03 | 101 | 76 | 94 | — | — | — |

[a] Analysis method was not yet available at time of preparation of storage stability samples

The test substances were considered stable as long as recoveries were at least 70%. Oxamyl, oxamyl-oxime, and DMOA recoveries were in 70–116% range with the exception of the 18 month Spain oxamyl-oxime samples (see below). Averaged recoveries, as well as the standard deviations, in fresh fortified samples as well as aged fortified samples were quite similar. Recovery ranges for all compounds (in both soil types) stored frozen for up to 18 months, are summarised briefly in the following table.

**Table 87 Percent recovery range, freezer stored samples for each analyte**

| Analyte | Overall average recovery (%) | Relative standard deviation (%) |
|---|---|---|
| Oxamyl | 96 | 15 |
| Oxamyl oxime | 82 | 13 |
| DMOA | 89 | 6 |

It is clear from the data that the recoveries were within the specified range. With the following exception of the 18 month Spain oxamyl-oxime samples, all aged fortified samples through 18 months showed recoveries greater than 70% range for all test substances as did the fresh fortified samples.

In the Spain study (DuPont-4800), only 24 out of 389 samples (6%) were analysed for oxamyl oxime after 12 months of storage. These samples were analyzed at 14 months. All of these samples were taken 154 days after application. At this time point, oxamyl-oxime residues had declined to levels <1% of the peak values observed at day 5. Therefore, there was no impact on the field soil dissipation study.

## III. CONCLUSION

Oxamyl and DMOA are stable in the freezer in the two types of soil investigated for at least 18 months. Oxamyl-oxime was stable for at least 18 months in one of the two soils while the recoveries obtained at 18 months for the other soil suggest a more limited stability of at least 12 months.

(Mol, J. G. J., 2003)

**Summary of persistence endpoints derived from field dissipation studies with oxamyl**

**Study submitted to the EU for the first time in this submission.**

**B.8.1.2.2.1/07**

| | | |
|---|---|---|
| **Reference: CA 7.1.2.2.1/03** | **Report:** | Partsch, S., Zillgens, B. (2015); Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213) and DMOA (IN-D2708) from field soil dissipation studies<br>**DuPont Report No.:** DuPont-41859 EU, Supplement No. 1<br>**Guidelines:** Council Directive 91/414/EEC, AIR-3, Annexes II and III, Article 79 (2) of EU Regulations (EC) No. 1107/2009 (14 June 2011), EU Regulations (EC) No. 1136/2013 (02-03 Oct 2013)<br>**Deviations:** None<br>**Testing Facility:** Dr. Knoell Consult GmbH, Mannheim, Germany<br>**Testing Facility Report No.:** DuPont-41859 EU, Supplement No. 1<br>**GLP:** No<br>**Certifying Authority:** Not applicable |

**Executive summary:**

A summary of this modelling position paper can be found in the Oxamyl EU Renewal Dossier, Document M-CP, Section 9 (DuPont-40953 EU and DuPont-42129 EU). Residue data from soil dissipation studies (DuPont-2815 and DuPont-3026) were reevaluated to meet the requirements of the current FOCUS (2006, 2011, 2014) and EFSA (2014) guidelines on degradation kinetics. The present study was aimed at assessing the same two field dissipation studies based on the current guidance of the FOCUS workgroup (FOCUS 2006, 2011 and 2014) and EFSA (2014) on the kinetic evaluation of degradation studies to provide persistence endpoints for comparison against trigger values and modelling endpoints for use in exposure modelling tools. The normalization of the residue data to reference soil temperature and soil moisture conditions was conducted with updated weather data sets from available open sources. In the present study, normalization was performed by adjusting the individual day length as a function of fluctuating temperature and moisture. The following equation was used for this so called “time-step normalization”. It results in a value > 1 for warmer and wetter conditions and a value < 1 for cooler and drier conditions.

In both field dissipation studies the parent compound oxamyl was applied as a granule formulation and was incorporated into the soil immediately after application. Therefore, surface related processes like photodegradation and volatilization were considered not significant and the procedure proposed by EFSA (2014) for the evaluation of tailored DegT50 field studies was followed. Consequently, all persistence and modelling endpoints were determined based on current guidances of the FOCUS workgroup (FOCUS 2006, 2011 and 2014) and including those residue data that were measured before 10 mm of cumulative rainfall has occurred. The results of the kinetic evaluations of the active substance and its metabolites are summarised below. Two field dissipation studies for oxamyl were conducted at European trial sites in Ottersum in The Netherlands (DuPont-2815) and Spalding in the UK (DuPont-3026). In both field dissipation studies, the Oxamyl 10GR (granule, 10% oxamyl) was applied at 4 (NL) and 5.5 kg a.s./ha (UK) onto bare ground, and the granules were further incorporated into the topsoil. Persistence endpoints and modelling endpoints were then calculated for comparison against trigger values and for use in exposure modelling, respectively.

$$Daynorm = Day \times Q10[(Tact - Tref)/10] \times (Moistact/FC)0.7$$

Where:
Daynorm = Normalized Day Length (NDL)
Day = 1 d
Q10 = Standard Q10 factor of 2.58
Tact = Daily soil temperature at measured depth
Tref = Reference temperature, 20 °C
Moistact = Daily soil water content at measured depth
FC = Reference soil water content (field capacity)

Two bare field dissipation and two greenhouse dissipation studies with oxamyl were carried out in the Northern and Southern EU. The results of the field dissipation studies confirmed that oxamyl degrades rapidly in soil with persistence $DT_{50}$ values ranging from 0.7 to 9.5 days. Pathway kinetics was also performed to determine persistence $DT_{50}$ values for the major metabolites, IN-A2213 and IN-D2708. The FOCUS derived persistence endpoints from the field dissipation studies are presented in the following tables.

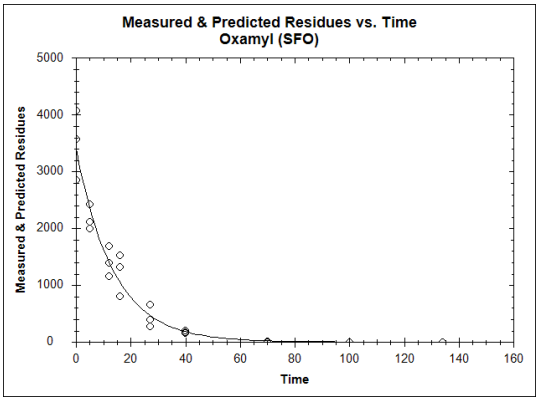
**Table 88 Summary of persistence and modelling endpoints for oxamyl**

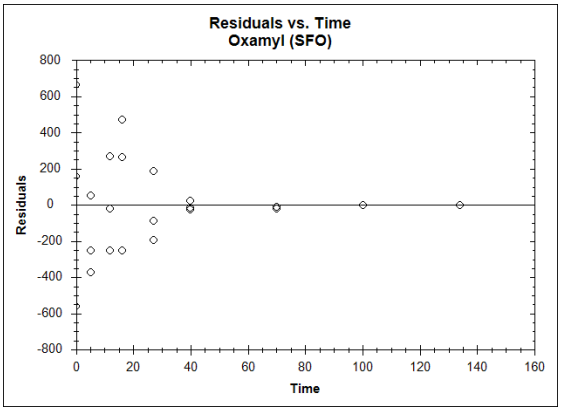
| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | $DT_{50norm}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|---|
| DuPont-2815 | Ottersum (NL) non-normalized data | 9.5 | 31.4 | 5.0 | 5.7 | SFO |
| DuPont-3026 | Spalding (UK) non-normalized data | 0.7 | 30.6 | 6.9 | 8.2 | DFOP |

The kinetics fittings for degradation of oxamyl in soil were derived from the respective best-fit model and are presented in figure below

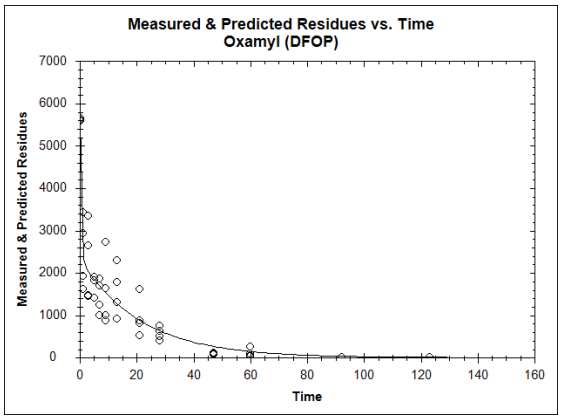
**Figure: The kinetics fittings for degradation of oxamyl in soil**

*Ottersum soil, non-normalized data*




*Spalding soil, non-normalized data*



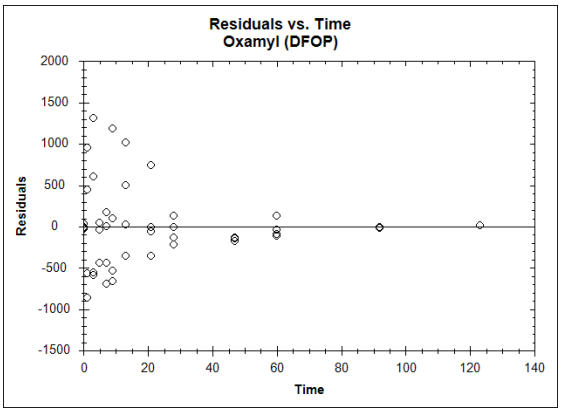




**Table 89 Summary of persistence and modelling endpoints for IN-A2213**

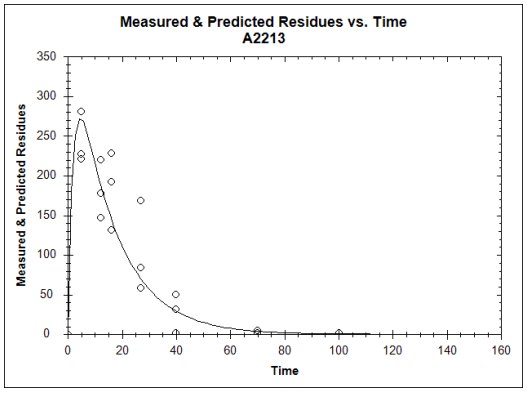
| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Formation fraction from oxamyl | $DT_{50norm}$ (days) | $\chi 2$ | Model |
|---|---|---|---|---|---|---|---|
| DuPont-2815 | Ottersum (NL) non-normalized data | 1.4 | 4.6 | 0.77 | 0.8 | 16.9 | SFO-SFO |
| DuPont-3026 | Spalding (UK) non-normalized data | 17.5 | 58.0 | Calculation not possible[a] | 8.8 | 13.0 | SFO[a] |

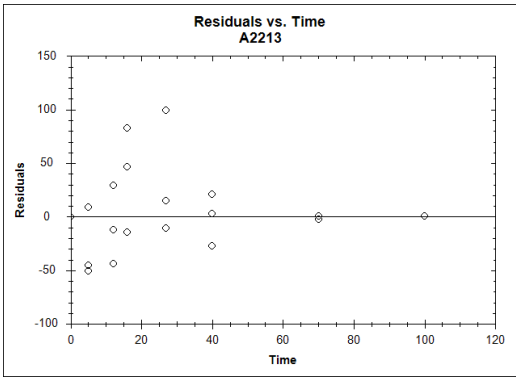
[a] Determined from decline fit

The kinetics fittings for degradation of oxamyl's metabolite, IN-A2213 for all seven soils were derived from the respective best-fit model and are presented in figure below

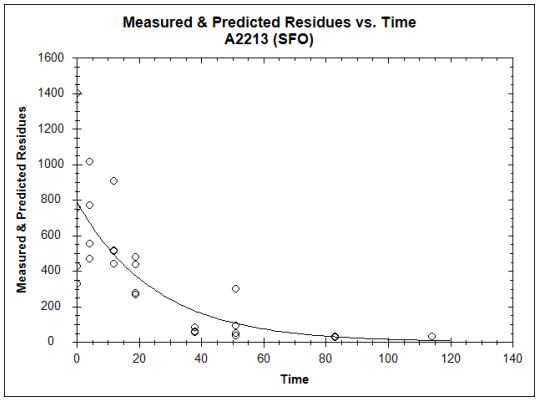
**Figure: The kinetics fittings for degradation of IN-A2213 in soil**

*Ottersum soil, non-normalized data*




*Spalding soil, non-normalized data*



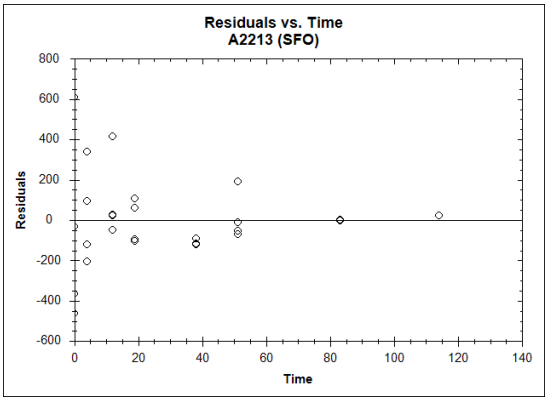



**Table 90 Summary of persistence and modelling endpoints for IN-D2708**

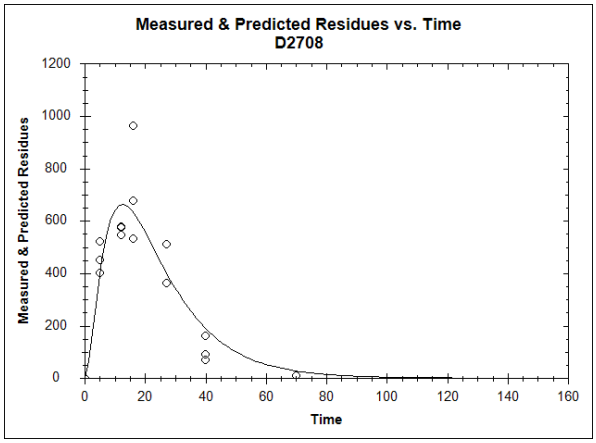
| Study | Soil/Condition | $DT_{50}$ (days) | $DT_{90}$ (days) | Formation fraction from IN-A2213 | $DT_{50norm}$ (days) | $\chi^2$ | Model |
|---|---|---|---|---|---|---|---|
| DuPont-2815 | Ottersum (NL), non-normalized data | 5.0 | 16.6 | 1 (fixed) | 2.9 | 11.5 | SFO-SFO |
| DuPont-3026 | Spalding (UK), non-normalized data | 9.1 | 30.1 | Calculation not possible[a] | 4.7 | 14.4 | SFO[a] |

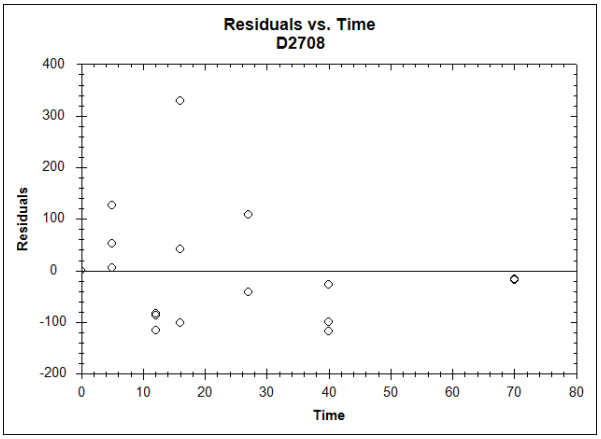
[a] Determined from decline fit

The kinetics fittings for degradation of oxamyl's metabolite, IN-D2708 in soil were derived from the respective best-fit model and are presented in figure below

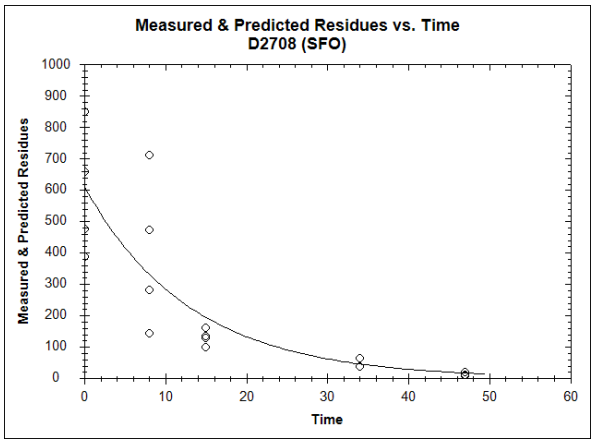
**Figure: The kinetics fittings for degradation of IN-D2708 in soil**

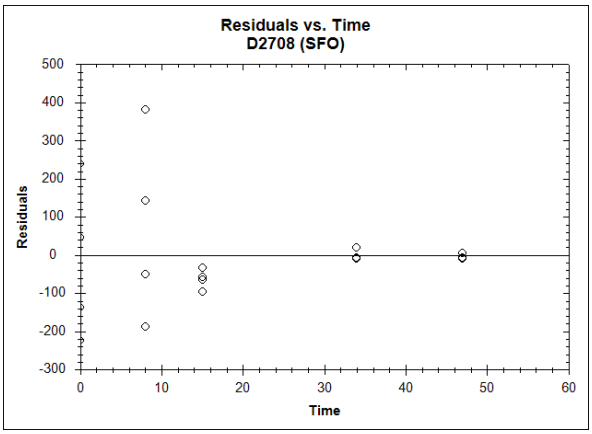
*Ottersum soil, non-normalized data*




*Spalding soil, non-normalized data*



A similar rapid degradation of oxamyl was observed in two greenhouse dissipation studies. In these studies, the $DT_{50}$ and $DT_{90}$ values for oxamyl ranged from 3.0–5.3 days and 12–18 days, respectively. The degradation pattern was also similar between the bare field trials and the greenhouse trials. The maximum occurrence of the major soil metabolites in field dissipation studies carried out with oxamyl are listed in Table .

**Table 91 Maximum occurrences of oxamyl metabolites in field dissipation studies (in parent equivalents)**

| Site | IN-A2213 | IN-D2708 |
|---|---|---|
| NL | 7.0 | 25.8 |
| UK | 25.5 | 16.0 |
| Greenhouse, Italy | 9.9 | 5.2 |
| Greenhouse, Spain | 26 | 4.9 |
| **Overall maximum** | **26** | **25.8** |

**RMS comments and conclusion**

This study is considered corrected to harmonize the derivation of degradation parameters from soil metabolism. Residue data of aerobic soil degradation field studies in two soils for oxamyl and its metabolites under field conditions (DuPont-2815 and DuPont-3026) were re-evaluated to derive persistence and modelling endpoints for oxamyl and its metabolites from field dissipation studies.
The kinetic assessments were conducted are in full compliance with the FOCUS kinetics guidance, however, since only two normalised field degradation endpoints are available for the parent and the metabolites, the geometric mean field DegT50 cannot be used as modelling endpoint for ground water and surface water risk assessment.

*B.8.1.2.2.2 Soil accumulation studies*

**Soil residue testing**

Soil residue studies are not required for oxamyl since the $DT_{50\ lab}$ is less than one third of the interval between application and harvest of potatoes (PHI = 90days).

**Soil accumulation testing**

Soil accumulation studies were not done with oxamyl since the $DT_{90}$ was much less than one year. The soil equilibrium concentration or soil $PEC_{soil}$ estimates reliably indicate no potential for accumulation after repeated applications in potatoes.

### B.8.1.3 Adsorption and desorption in soil

#### B.8.1.3.1 Adsorption and desorption

*B.8.1.3.1.1 Adsorption and desorption of the active substance*

**B.8.1.3.1.1/01**

| | | |
|---|---|---|
| **Reference:** -- | **Report:** | Ohm, M.B. (2001); Adsorption/desorption of $^{14}$C-oxamyl in five soils<br>**DuPont Report No.:** DuPont-3166, Revision No. 1<br>**Guidelines:** OECD 106 (1981), U.S. EPA 163-1 (1982) |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: HOTC 417
   Purity: Radiochemical purity - >95%

**Material and Methods:**

The adsorption/desorption of oxamyl (specific activity 18.65 µCi/mg, radiochemical purity >95%) was investigated in five soils (Table ). The soil types ranged from silty clay loam to loam, with three of US origin, one of German origin and one from The Netherlands. The pH ranged from 4.8-7.0, with four soils in range of 6.9-7.8, and organic matter content ranged from 0.4-4.4%.
Soils were pre-equilibrated with 0.01M calcium chloride prior to adsorption. Initial solution concentrations of 0.05, 0.10, 0.50, 1.00 and 5.00 µg/mL were prepared in 0.01M calcium chloride. Testing was conducted in duplicate with a soil/solution ratio of 1/1 w/v (10 g dry weight of soil/10 mL solution).
The soil/solution mixtures were equilibrated for 4 hours at 20-25°C. Two 4-hour desorption cycles were conducted following the adsorption cycle. Only soils at the highest solution concentration (5.00 µg/mL) that had an average soil adsorption exceeding 10% were desorbed. Desorption was accomplished by completely decanting the supernatant (after centrifugation) and replacing it with an equivalent volume of fresh 0.01M calcium chloride solution.
Linear adsorption coefficients (KD) were calculated from the mean ratios of CS to CW, where CS and CW represent the test substance concentration in soil and in the aqueous phase at equilibrium, respectively. Adsorption data were further analysed using the log form of the Freundlich equation [logCS = 1/n·(logCW) + logKF; KF = Freundlich coefficient].
Aqueous samples were analysed by LSC. Soil samples from the highest solution concentration (5.00 µg/mL) were analysed by combustion and LSC. The aqueous phase of one replicate of the lowest (0.05 µg/mL) and highest solution concentration (5.00 µg/mL) from each soil was analysed by reversed phase HPLC. The $^{14}C$-radioactivity mass balance was determined for samples at the highest solution concentration (5.00 µg/mL) by summing the amount in the adsorption and desorption phases and in the soil phase.

**Table 92 Characteristics of the soils used in the oxamyl adsorption/desorption studies**

| Characteristic | Commerce | Drummer #6 | Gross Umstadt | Mattapex #25 | Nijmegen |
|---|---|---|---|---|---|
| Soil origin | Greenville, MS, USA | Rochelle, IL, USA | Gross Umstadt, Germany | Newark, DE, USA | Nijmegen, The Netherlands |
| DuPont soil number | L1133-68 | L1133-74 | L1133-80 | L1133-72 | L1133-82 |
| pH | 7.0 | 4.8 | 7.8 | 6.9 | 7.0 |
| Phosphorous (mg/kg) | 125 | 90 | 63 | 6 | 310 |
| Potassium (mg/kg) | 204 | 219 | 487 | 51 | 227 |
| Magnesium (mg/kg) | 106 | 557 | 112 | 35 | 107 |
| Calcium (mg/kg) | 972 | 2434 | 3405 | 1713 | 1558 |
| Sodium (mg/kg) | <20 | <20 | 27 | <20 | <20 |
| Total nitrogen (mg/kg) | 397 | 2595 | 1497 | 2183 | 1297 |
| Organic matter (%) | 0.4 | 4.4 | 2.1 | 4.3 | 2.4 |
| Organic carbon (%) | 0.2 | 2.6 | 1.2 | 2.5 | 1.4 |
| Soluble salts (mmhos/cm) | 0.21 | 0.81 | 0.43 | 0.25. | 0.27 |
| CEC (meq/100 g) | 6.7 | 26.3 | 9.6 | 12.7 | 10.1 |
| Bulk density (g/cm$^3$) | 1.23 | 1.17 | 0.93 | 1.07 | 1.28 |
| Moisture (%) | 11.6 | 22.1 | 21.6 | 22.9 | 14.2 |
| Air-dried moisture (%) | 0.87 | 3.31 | 1.43 | 2.29 | 1.18 |
| Moisture-holding capacity (%) at 0 bar | 33.3 | 49.4 | 50.0 | 49.3 | 33.3 |
| Moisture-holding capacity (%) at 1/3 bar | 13.1 | 31.0 | 20.7 | 30.8 | 17.1 |
| Moisture-holding capacity (%) at 1 bar | 7.4 | 22.6 | 11.7 | 20.8 | 11.1 |
| Moisture-holding capacity (%) at 15 bar | 3.8 | 13.4 | 9.0 | 11.2 | 6.9 |
| Sand (%) | 32.8 | 8.4 | 5.6 | 10.4 | 45.2 |
| Silt (%) | 56.4 | 60.8 | 77.2 | 70.8 | 40.8 |
| Clay (%) | 10.8 | 30.8 | 17.2 | 10.8 | 14.0 |
| USDA texture | silt loam | silty | silt loam | silt loam | loam |

% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

**Findings:**

The time to equilibration of oxamyl between soil and solution was judged to be ~2 hours based on results from a preliminary experiment. An equilibration time of four hours was chosen to ensure equilibrium for the definitive adsorption experiments. Total recoveries of radioactivity (average of duplicates) ranged from 91.4% to 99.4% in all five soils.

The values for the adsorption coefficient, *KD*, were calculated for all five test soils at each of the five test solution concentrations. The *KD* values ranged from 0.09 mL/g in the Commerce soil to 0.44 mL/g in the Drummer #6 soil are listed in Table . The adsorption coefficients were corrected for the organic matter and organic carbon content for each test soil to calculate the soil sorption coefficients *Kom* and *Koc*, Koc values ranged from 8 to 39 mL/g (average = 17 mL/g).

**Table 93 Summary of the averaged linear soil adsorption coefficients for oxamyl**

| Soil | USDA soil texture | % Organic matter | $K_D$ (mL/g) | $K_{om}$ (mL/g) | $K_{oc}$ (mL/g) |
|---|---|---|---|---|---|
| Commerce | silt loam | 0.4 | 0.09 | 22 | 39 |
| Drummer #6 | silty clay loam | 4.4 | 0.44 | 10 | 17 |
| Gross Umstadt | silt loam | 2.1 | 0.11 | 5 | 9 |
| Mattapex #25 | silt loam | 4.3 | 0.19 | 4 | 8 |
| Nijmegen | loam | 2.4 | 0.14 | 6 | 10 |

The values for the Freundlich adsorption isotherm parameter are given in Table , *KF*, were derived from the linear form of the Freundlich equation and ranged from 0.05 to 0.41 mL/g, KFoc values ranging from 4 to 37 mL/g (average = 16 mL/g). The 1/n values ranged from 0.946 to 1.27 (average = 1.07)..

**Table 94 Summary of the Freundlich equation parameters for the oxamyl adsorption isotherms**

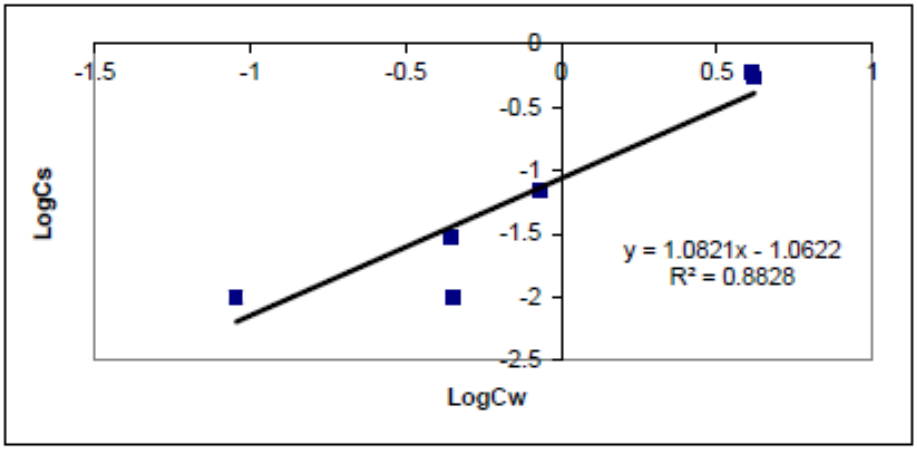
| Soil | USDA texture | % Organic matter | $K_F$ (mL/g) | 1/n | $K_{Fom}$ (mL/g) | $K_{Foc}$ (mL/g) |
|---|---|---|---|---|---|---|
| Commerce | silt loam | 0.4 | 0.09 | 1.08 | 22 | 37 |
| Drummer #6 | silty clay loam | 4.4 | 0.41 | 0.986 | 9 | 16 |
| Gross Umstadt | silt loam | 2.1 | 0.05 | 1.27 | 3 | 4 |
| Mattapex #25 | silt loam | 4.3 | 0.18 | 0.946 | 4 | 7 |

The Freundlich parameters for Nijmegen soil could not be reliably calculated due to negligible adsorption of oxamyl to this soil at several test concentrations.

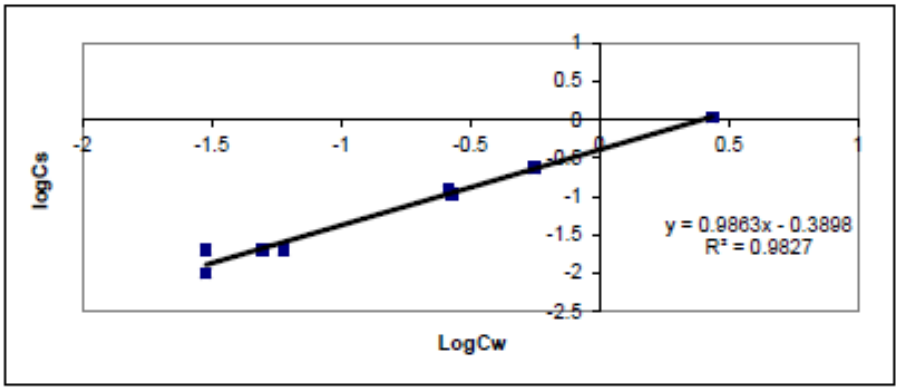
Plots of the adsorption isotherms of oxamyl for each soil are presented in Figure below.

**Figure Adsorption isotherms for the Commerce, Drummer and Mattapex soils treated with oxamyl**

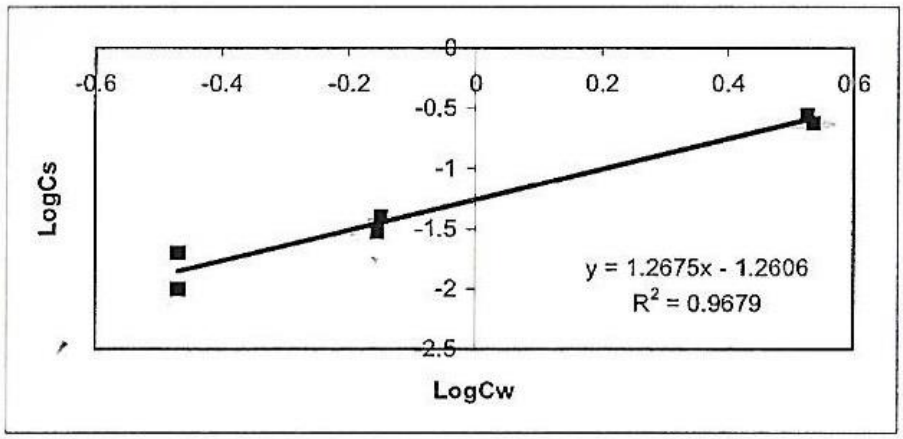
**A. Commerce Soil**



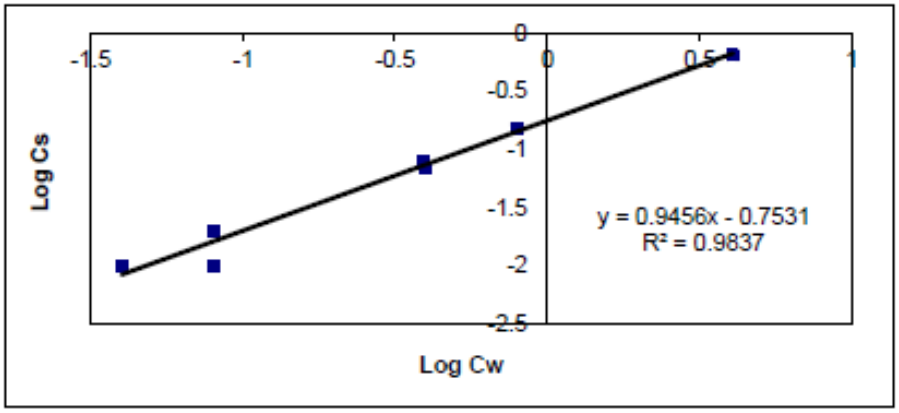

**B. Drummer #6 Soil**




**C. Gross-Umstadt Soil**



**D. Mattapex #25 Soil**




Oxamyl was moderately to readily desorbed from the three soils in which more than 10% of the applied material was originally adsorbed. The average percentage of the adsorbed oxamyl which was desorbed during two desorption intervals is listed in Table . A total of 36 to 71% of the adsorbed oxamyl was removed in the two desorption intervals.

**Table 95 Summary of the percentage of sorbed oxamyl desorbed during two desorption intervals**

| Soil | USDA soil texture | % Organic matter | % of adsorbed oxamyl desorbed during the 1st desorption | % of adsorbed oxamyl desorbed during the 2nd desorption | Total % of adsorbed oxamyl desorbed |
|---|---|---|---|---|---|
| Commerce | silt loam | 0.4 | 48.75 | 15.32 | 64.07 |
| Drummer #6 | silty clay loam | 4.4 | 48.25 | 22.79 | 71.04 |
| Mattapex #25 | silt loam | 4.3 | 19.62 | 16.87 | 36.49 |

Less than 10% of the applied oxamyl at the 5.00 µg/mL test concentration was adsorbed to Gross Umstadt and Nijmegen soils; so they were not used in the desorption phase of the study.

**Conclusions:**

Oxamyl is weakly adsorbed to soil. The average linear Koc and Freundlich KFoc values were 17 mL/g and 16 mL/g respectively. The average Freundlich 1/n value was 1.07. Sorption was reversible and the adsorbed oxamyl was moderately to readily desorbed from three test soils.

The adsorption and desorption of the active substance study DuPont-3166, Revision No. 1, originally submitted under EU Rev8 Point IIA 7.1.2 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines OECD 106 (1981) and U.S. EPA 163-1 (1982). A review of this study indicates that the experimental performance of the study fully meets the current guideline (OECD 106). The calculations of the Freundlich adsorption parameters presented in this study have been superseded by the re-analysis presented in DuPont-3166, Revision No. 2.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Oxamyl is weakly adsorbed to soil. The average linear Koc and Freundlich KFoc values were 17 mL/g and 16 mL/g respectively.

**Study submitted to the EU for the first time in this submission**

**B.8.1.3.1.1/02**

| | | |
|---|---|---|
| **Reference: CA 7.1.3.1.1./02** | **Report:** | Malin, J.N. (2015); Adsorption/desorption of $^{14}$C-oxamyl in five soils<br>**DuPont Report No.:** DuPont-3166, Revision No. 2<br>**Guidelines:** OECD 106 (1981), U.S. EPA 163-1 (1982)<br>**Deviations:** None<br>**Testing Facility:** DuPont Experimental Station, DuPont Stine-Haskell Research Center, Wilmington, Delaware, USA, Newark, Delaware, USA<br>**Testing Facility Report No.:** DuPont-3166, Revision No. 2<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Reason for Revision No. 2**

The following issues are addressed in this revision:

- Revised Freundlich sorption isotherm parameters for Gross-Umstadt soil are presented following re-analysis to include all available data.
- Freundlich sorption isotherm parameters for Nijmegen soil are presented following analysis of all available data.

Updated best-fit linear analyses using all replicate data for the Freundlich isotherms performed in each soil are presented in this revision. The experimental design, description, and results remain the same as those presented in the original report. This re-analysis resulted in the following Freundlich adsorption results. These revised results have been taken forward in all modelling assessments.

Freundlich sorption isotherm parameters for Nijmegen soil and (Revised Freundlich sorption isotherm parameters) for Gross-Umstadt soil are presented below.

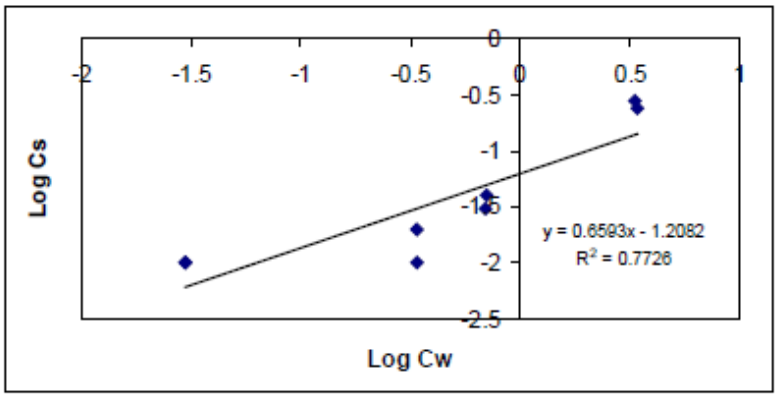
**Table 96 Summary of the Freundlich equation parameters for the oxamyl adsorption isotherms**

| Soil | USDA texture | % Organic matter | $K_F$ (mL/g) | 1/n | $K_{Fom}$ (mL/g) | $K_{Foc}$ (mL/g) |
|---|---|---|---|---|---|---|
| Gross Umstadt | silt loam | 2.1 | 0.06 | 0.66 | 3 | 5 |
| Nijmegen | loam | 2.4 | 0.12 | 0.78 | 5 | 9 |

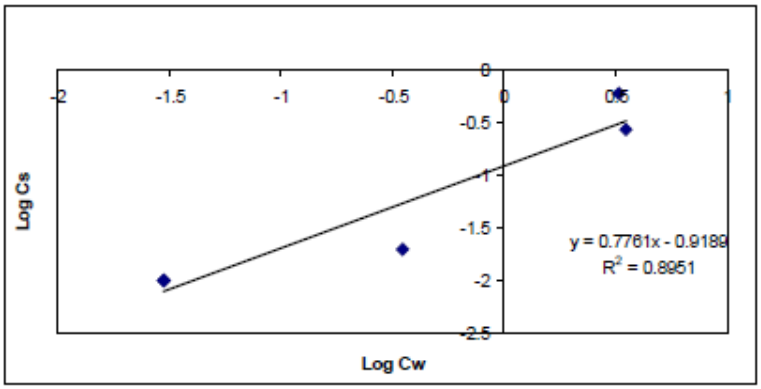
Plots of the adsorption isotherms of oxamyl for Gross-Umstadt and Nijmegen soil are presented in Figure below.

**Figure Adsorption isotherms for Gross-Umstadt and Nijmegen soil treated with oxamyl**

C. Gross-Umstadt Soil




E. Nijmegen Soil




Oxamyl was moderately to readily desorbed from the three soils in which more than 10% of the applied material

**Table 97 Summary of the updated Freundlich parameters for the oxamyl adsorption isotherms**

| Soil | USDA texture | % Organic matter | $K_F$ (mL/g) | $K_{FOM}$ (mL/g) | $K_{FOC}$ (mL/g) | 1/n |
|---|---|---|---|---|---|---|
| Commerce | Silt loam | 0.4 | 0.09 | 22 | 37 | 1.08 |
| Drummer #6 | Silty clay loam | 4.4 | 0.41 | 9 | 16 | 0.99 |
| Gross-Umstadt | Silt loam | 2.1 | 0.06 | 3 | 5 | 0.66 |
| Mattapex #25 | Silt loam | 4.3 | 0.18 | 4 | 7 | 0.95 |
| Nijmegen | Loam | 2.4 | 0.12 | 5 | 9 | 0.78 |

**RMS comments and conclusion**

Updated best-fit linear analyses using all replicate data for the Freundlich isotherms performed in each soil are those presented in the original report. These revised results have been taken forward in all modelling assessments.

**Study submitted to the EU for the first time in this submission.**

**B.8.1.3.1.1/03**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.1.3.1.1./01** | **Report:** | Allan, J. (2012); [$^{14}$C]DPX-D1410: Batch equilibrium (adsorption/desorption) in six soils<br>**DuPont Report No.:** DuPont-33692<br>**Guidelines:** OECD 106 (2000), OPPTS 835.1230 (2008)<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** 67481<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

The adsorption/desorption characteristics of oxamyl was studied in six soils (pH range of 5.1 to 7.4 using 1:2 soil:0.01 M $CaCl_2$, organic carbon range of 1.2 to 3.3%) from Europe and North America in a batch equilibrium experiment. The adsorption phase of the study was carried out by equilibrating fresh soil with oxamyl at 0.05, 0.10, 0.50, 1.0, and 5.0 µg/mL in the dark at 10 ± 2°C for 24 hr. The equilibrating solution used was 0.01 M $CaCl_2$, with a soil/solution ratio of 1:1. Desorption phase of the study was carried out at the highest concentration for an additional two cycles by replenishing the adsorption supernatant removed with 0.01 M $CaCl_2$ solution, re-suspending the soil pellets, and shaking the samples. The mean mass balance in the adsorption/desorption ranged 91.3 to 97.2% AR.

The mean isotherm adsorption $K_F$ values ranged from 0.0708 to 0.598 mL/g. Mean isotherm adsorption $K_{foc}$ values ranged from 4.1 to 23 mL/g. At the end of the desorption phase, between 35.3 and 51.0% of the adsorbed amount was desorbed. The desorption $K_d$ values from the first desorption step ranged from 0.165 to 1.29 mL/g. Desorption $K_d$ values after the second desorption step ranged 0.287 to 1.05 mL/g. Desorption $K_d$ and $K_{oc}$ values were higher than those obtained for adsorption.

There was little correlation between adsorption constants and soil pH. Determined $K_{oc}$ values indicate high potential for mobility.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabeled test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: D1410-222 (radiolabel at the central imine carbon)
   Radiochemical purity: 96.6%
   Specific activity: 22.8 µCi/mg
   Stability of test compound: The test material was stable in the aqueous phase under the test conditions

2. Soils:

The study was conducted with six different soil types (three European and three from the U.S.). These soils were collected fresh from the field. Fresh soils were 2 mm sieved prior to experimentation. A summary of the physical and chemical properties of the soils is provided in **Table** . The percent sand, silt, and clay are presented on the basis of the USDA classification.

**Table 98 Soil characteristics**

| Property | Nijmegen | Tama | Drummer |
|---|---|---|---|
| Origin | Gelderland Province The Netherlands | Stark County, Illinois, USA | Ogle County, Illinois, USA |
| Soil texture [a] | Sandy Loam | Silty Clay Loam | Clay Loam |
| % Sand (2000–50 μm) | 68 | 16 | 25 |
| % Silt (<50–2 μm) | 18 | 54 | 42 |
| % Clay (<2 μm) | 14 | 30 | 33 |
| pH [b] | 6.3 | 6.0 | 5.8 |
| Organic carbon (%) | 1.7 | 1.9 | 3.3 |
| CEC (meq/100 g) | 11.1 | 18.3 | 24.6 |
| Moisture at 1/3 atm (%) | 19.0 | 33.7 | 33.6 |
| Bulk density (g/cm$^3$) | 1.20 | 1.06 | 1.06 |

| Property | Sassafras | Gross-Umstadt | Nambsheim |
|---|---|---|---|
| Origin | Kent County Maryland, USA | Darmstadt, Hessen, Germany | Nambsheim, Alsace, France |
| Soil texture [a] | Loam | Loam | Sandy Loam |
| % Sand (2000–50 μm) | 50 | 36 | 64 |
| % Silt (<50–2 μm) | 40 | 50 | 30 |
| % Clay (<2 μm) | 10 | 14 | 6 |
| pH [b] | 5.1 | 6.7 | 7.4 |
| Organic carbon (%) | 1.5 | 1.2 | 1.7 |
| CEC (meq/100 g) | 8.3 | 11.3 | 10.6 |
| Moisture at 1/3 atm (%) | 16.3 | 15.8 | 12.3 |
| Bulk density (g/cm$^3$) | 1.14 | 1.15 | 1.11 |

[a] USDA soil classification system

[b] pH in 1:2 soil:0.01M $CaCl_2$ (aq)

B. STUDY DESIGN

1. Experimental conditions

Stock solutions of $^{14}$C-labeled oxamyl were prepared in 0.01 M $CaCl_2$ to give dose solution concentrations in the range of 0.0516, 0.102, 0.513, 1.02, and 5.23 μg/mL. The pH of adsorption supernatants in the definitive test samples ranged from 6.30 to 7.62 after addition of the test substance. The appropriate solution to soil ratio was determined in preliminary testing at 1:1. Portions of test solution (15 mL) were shaken at 10 ± 2°C with samples of test soil (15 g dry weight) for a 24-hour equilibration period in darkness. A control experiment was also performed to assess potential adsorption to glass test vessels. Following centrifugation (1430 × *g* for 10 minutes), the supernatant was decanted and triplicate aliquots were prepared for radioassay.

Following the adsorption phase, fresh 0.01 M aqueous $CaCl_2$ (15 mL) was added to samples from the highest concentration tested, equilibrated for 24 hours at 10 ± 2°C, solutions and soils separated, quantified, and subject to a further desorption phase. Soil extracts (the four soils that had $K_d$ values less than 0.3 mL/g by the indirect method of calculation) from the highest concentration tested were further extracted three times using 30 mL of 50:50 acetonitrile:water (v/v).

2. Description of analytical procedures

Radioactivity was determined by LSC of the aqueous supernatants. Samples of soil residues from the highest concentration samples after desorption were combusted in a biological oxidizer for mass balance. Results were corrected for the overall average oxidizer efficiency and expressed as dpm/g.

Aqueous supernatants at two concentrations (0.10 and 5.0 μg/mL) obtained after equilibration were analysed by reverse phase HPLC. The limit of detection (LOD) for oxamyl (0.10 μg/mL) was 0.28% AR ($2.82 \times 10^{-3}$ μg/mL). The limit of quantification (LOQ) oxamyl (0.10 μg/mL) was 1.1% AR ($1.11 \times 10^{-3}$ μg/mL). LOD and LOQ values for the oxamyl in the soil extracts were 0.025 % AR ($1.24 \times 10^{-3}$ μg/mL) and 0.098% AR ($4.89 \times 10^{-3}$ μg/mL).

## II. RESULTS AND DISCUSSION

### A. MASS BALANCE

Mass balances ranged from 90.8–98.0%, which are within the recommended guideline range of 90–110%.

### B. FINDINGS

Adsorption isotherm data were analysed using the log form of the Freundlich equation: log x/m = 1/n * log Ce + log$K_f$ or Linear distribution coefficients ($K_d$) were calculated from the mean ratios of x/m to Ce (Table ).

**Table 99 Adsorption and desorption constants of oxamyl in the soils**

| | | | Adsorption | | | | Desorption | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Soil** | **OC %** | **pH[a]** | **$K_F$** | **1/n** | **$r^2$** | **$K_{foc}$** | **$K_{d1}$** | **$K_{d2}$** | **$K_{oc1}$** | **$K_{oc2}$** | **$D_T$** |
| Nijmegan | 1.7 | 6.3 | 0.0708 | 0.9458 | 0.9988 | 4.1 | 0.165 | 0.303 | 9.5 | 17 | 35.3 |
| Tama | 1.9 | 6.0 | 0.442 | 0.9760 | 0.9989 | 23 | 1.29 | 0.517 | 67 | 27 | NA |
| Drummer | 3.3 | 5.8 | 0.598 | 0.9133 | 0.9997 | 18 | 0.986 | 1.05 | 30 | 32 | NA |
| Sassafras | 1.5 | 5.1 | 0.164 | 0.9512 | 0.9998 | 11 | 0.234 | 0.417 | 15 | 28 | 51.0 |
| G-Umstadt | 1.2 | 6.7 | 0.128 | 0.9301 | 0.9988 | 11 | 0.176 | 0.287 | 15 | 25 | 45.8 |
| Nambsheim | 1.7 | 7.4 | 0.141 | 0.9645 | 0.9980 | 8.4 | 0.229 | 0.420 | 14 | 25 | 36.0 |

[a] pH in 1:2 soil:0.01 M $CaCl_2$ (aq)

$K_d$ = Adsorption and desorption coefficients; $K_f$= Freundlich adsorption and desorption coefficients; 1/n = Slope of Freundlich adsorption/desorption isotherms; $K_{foc}$ = Coefficient adsorption per organic carbon ($K_f \times 100\%$ organic carbon); $D_T$ = Sum of two desorption steps

The Freundlich adsorption/desorption plots obtained showed good linearity, with slopes generally close to unity, indicating both adsorption and desorption was linearly proportional to soil concentration over the range tested. The Freundlich adsorption constants ranged from 0.0708–0.598. Adsorption $K_{foc}$ values were in the range of 4.1–23 for the six test soils showing that oxamyl was weakly bound to all six soils. The desorption constants were larger than those obtained for adsorption, with the first desorption constants in the range 0.165–1.29 and $K_{oc}$ values in the range 9.5–67 indicating that once adsorbed, oxamyl is not as readily desorbed. The % adsorbed and desorbed oxamyl at each concentration is provided in Table and Table , respectively.

**Table 100 Concentration of oxamyl in the solid and liquid phases at the end of adsorption equilibration period**

| Concentration on soil (µg a.s./mL) | Nijmegen | | | Tama | | | Drummer | | |
|---|---|---|---|---|---|---|---|---|---|
| | on soil[a] (µg a.s./g) | in solution (µg a.s./mL) | % adsorbed[b] | on soil (µg a.s./g) | in solution (µg a.s./mL) | % adsorbed | on soil (µg a.s./g) | in solution (µg a.s./mL) | % adsorbed |
| 0.050 | 0.00394 | 0.0472 | 7.7 | 0.0173 | 0.0339 | 33.7 | 0.0226 | 0.0285 | 44.2 |
| | 0.00403 | 0.0471 | 7.9 | 0.0156 | 0.0353 | 30.6 | 0.0225 | 0.0288 | 43.9 |
| 0.10 | 0.00811 | 0.0938 | 8.0 | 0.0312 | 0.0701 | 30.8 | 0.0455 | 0.0572 | 44.3 |
| | 0.00734 | 0.0941 | 7.2 | 0.0344 | 0.0669 | 33.9 | 0.0449 | 0.0570 | 44.1 |
| 0.50 | 0.0328 | 0.474 | 6.5 | 0.152 | 0.357 | 29.8 | 0.204 | 0.305 | 40.1 |
| | 0.0341 | 0.475 | 6.7 | 0.160 | 0.348 | 31.5 | 0.206 | 0.304 | 40.3 |
| 1.0 | 0.0673 | 0.952 | 6.6 | 0.322 | 0.689 | 31.9 | 0.389 | 0.627 | 38.3 |
| | 0.0653 | 0.951 | 6.4 | 0.313 | 0.701 | 30.9 | 0.385 | 0.626 | 38.0 |
| 5.0 | 0.298 | 4.85 | 5.8 | 1.58 | 3.61 | 30.4 | 1.79 | 3.37 | 34.7 |
| | 0.353 | 4.83 | 6.8 | 1.52 | 3.66 | 29.4 | 1.83 | 3.35 | 35.4 |
| Concentration on soil (µg a.s./mL) | Sassafras | | | Gross-Umstadt | | | Nambsheim | | |
| | on soil[a] (µg a.s./g) | in solution (µg a.s./mL) | % adsorbed[b] | on soil (µg a.s./g) | in solution (µg a.s./mL) | % adsorbed | on soil (µg a.s./g) | in solution (µg a.s./mL) | % adsorbed |
| 0.050 | 0.00820 | 0.0433 | 15.9 | 0.00721 | 0.0442 | 14.0 | 0.00615 | 0.0441 | 12.3 |
| | 0.00827 | 0.0432 | 16.1 | 0.00717 | 0.0441 | 14.0 | 0.00749 | 0.0442 | 14.5 |
| 0.10 | 0.0158 | 0.0856 | 15.6 | 0.0126 | 0.0889 | 12.4 | 0.0145 | 0.0873 | 14.3 |
| | 0.0160 | 0.0860 | 15.7 | 0.0132 | 0.0880 | 13.1 | 0.0128 | 0.0876 | 12.7 |
| 0.50 | 0.0762 | 0.438 | 14.8 | 0.0599 | 0.452 | 11.7 | 0.0730 | 0.443 | 14.1 |
| | 0.0720 | 0.435 | 14.2 | 0.0619 | 0.446 | 12.1 | 0.0629 | 0.447 | 12.3 |
| 1.0 | 0.142 | 0.883 | 13.9 | 0.115 | 0.905 | 11.3 | 0.123 | 0.897 | 12.1 |
| | 0.149 | 0.873 | 14.6 | 0.123 | 0.891 | 12.1 | 0.123 | 0.897 | 12.1 |
| 5.0 | 0.709 | 4.51 | 13.6 | 0.480 | 4.64 | 9.40 | 0.587 | 4.62 | 11.3 |
| | 0.666 | 4.54 | 12.8 | 0.578 | 4.63 | 11.1 | 0.638 | 4.58 | 12.2 |

[a] Measured by soil residue analysis (mass of test substance adsorbed to soil [µg]/ initial dry soil weight [g])

[b] % adsorbed as the % of the applied

**Table 101Concentration of oxamyl in the solid and liquid phases at the end of desorption (total of all desorption phases)**

| Concentration on soil (mg a.s./kg or µg a.s./mL) | Nijmegen | | | | | Sassafras | | | | | Gross-Umstadt | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | on soil (µg a.s./g) | | in solution (µg a.s./mL) | | | on soil (µg a.s./g) | | in solution (µg a.s./mL) | | | on soil (µg a.s./g) | | in solution (µg a.s./mL) | | |
| | desorption interval | | desorption interval | | | desorption interval | | desorption interval | | | desorption interval | | desorption interval | | |
| | 1 | 2 | 1 | 2 | % desorbed [a] | 1 | 2 | 1 | 2 | % desorbed | 1 | 2 | 1 | 2 | % desorbed |
| 5.0 | 0.349 | 0.291 | 1.88 | 0.845 | NA | 0.443 | 0.334 | 1.91 | 0.821 | 52.9 | 0.323 | 0.237 | 2.16 | 1.02 | 50.6 |
| | 0.285 | 0.229 | 1.98 | 0.875 | 35.3 | 0.442 | 0.339 | 1.87 | 0.794 | 49.1 | 0.435 | 0.341 | 2.16 | 1.00 | 41.1 |

| Concentration on soil (mg a.s./kg or µg a.s./mL) | Nambsheim | | | | |
|---|---|---|---|---|---|
| | on soil (µg a.s./g) | | in solution (µg a.s./mL) | | |
| | desorption interval | | desorption interval | | |
| | 1 | 2 | 1 | 2 | % desorbed |
| 5.0 | 0.438 | 0.370 | 2.02 | 0.940 | 37.0 |
| | 0.481 | 0.414 | 1.99 | 0.926 | 35.1 |

[a] Total percent of test substance desorbed after both desorption intervals.

NA Values for second replicate could not be calculated due to low adsorption.

Note: Desorption concentrations and percentages could not be determined for the Tama and Drummer soils due to low adsorption and experimental error, which resulted in values less than zero.

## III. CONCLUSION

The adsorption constants did not appear to correlate with the organic carbon content of the soils tested. $K_{foc}$ values ranged from 4.1 to 23. Adsorption of oxamyl also did not appear to be related to soil pH. On the basis of the results obtained it appears that oxamyl has a high potential soil mobility (ASTM classification system) (American Society for Testing and Materials, 1988 Annual Book of ASTM Standards, pp. 731–737, Designation: E 1195 -87, "Standard Test Method for Determining a Sorption Constant [$K_{oc}$] for an Organic Chemical in Soil and Sediments.")

(J. Allan, 2012)

The adsorption and desorption of the active substance study DuPont-3166, submitted for the first time in this submission and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guidelines OECD 106 (2000) and U.S. EPA OPPTS 835.1230 (2008). A review of this study indicates that the study fully meets the current guideline (OECD 106). The study is relied upon.

**RMS comments and conclusion**

The study is relied upon. On the basis of the results obtained it appears that oxamyl has a high potential soil mobility The adsorption constants ($K_{foc}$ values) ranged from 4.1 to 23.

*B.8.1.3.1.2 Adsorption and desorption of relevant metabolites, breakdown and reaction products*

**B.8.1.3.1.2/01**

| **Reference:** -- | **Report:** | Berg, D.S. (2000a); Adsorption/desorption of [$^{14}$C]IN-A2213 in five soils<br>**DuPont Report No.:** DuPont-3929, Revision No. 1<br>**Guidelines:** OECD 106 (1981), U.S. EPA 163-1 (1982) |
|---|---|---|

1. Test material: [$^{14}$C]IN-A2213
   Lot/Batch #: HOTC 563
   Purity: Radiochemical purity - >95%

**Material and Methods:**

The adsorption/desorption of [ $^{14}$C]-IN-A2213 (specific activity 25.2 µCi/mg, radiochemical purity >95%) was investigated in five soils (Table ). The soil types ranged from silt loam to loam, with three of US origin, one of German origin and one from The Netherlands. The pH ranged from 5.8-7.5 and organic matter content ranged from 0.8-6.3%.

Soils were pre-equilibrated with 0.01M calcium chloride prior to adsorption. Initial solution concentrations of 0.05, 0.1, 0.5, 1.0 and 5.0 µg/mL were prepared in 0.01M calcium chloride. Testing was conducted in duplicate with a soil/solution ratio of 1/1 w/v (10 g dry weight of soil/10 mL solution).

The soil/solution mixtures were equilibrated for 24 hours at 20 °C. Two 24-hour desorption cycles were conducted following the adsorption cycle. Only soils at the highest solution concentration (5.0 µg/mL) that had an average soil adsorption exceeding 10% were desorbed. Desorption was accomplished by completely decanting the supernatant (after centrifugation) and replacing it with an equivalent volume of fresh 0.01M calcium chloride solution.

Linear adsorption coefficients (KD) were calculated for the highest solution concentration samples (5.0 µg/mL) from the mean ratios of CS to CW, where CS and CW represent the test substance concentration in soil and in the aqueous phase at equilibrium, respectively. Adsorption data were further analysed using the log form of the Freundlich equation [logCS = 1/n·(logCW) + logKF, KF = Freundlich coefficient].

Aqueous samples were analysed by LSC. Soil samples from a preliminary study and the desorption study (both at 5.0 µg/mL) were analysed by combustion and LSC. The aqueous phase of the highest solution concentration (5.0 µg/mL) from each soil was analysed by reversed phase HPLC. The $^{14}$C-radioactivity mass balance was determined for preliminary study samples (5.0 µg/mL) by summing the amount in aqueous and soil phases after 24 hours equilibration.

**Table 102 Characteristics of the soils used in the IN-A2213 adsorption/desorption studies**

| Characteristic | Commerce | Drummer | Gross Umstadt | Mattapex | Nijmegen |
|---|---|---|---|---|---|
| Soil origin | Greenville, MS, USA | Rochelle, IL, USA | Gross Umstadt, Germany | Rock Hall, MD, USA | Nijmegen, The Netherlands |
| DuPont soil number | L1133-100 | L1133- | L1133-46 | L1133-72 | L1133-82 |
| pH | 5.8 | 5.8 | 7.5 | 6.9 | 7.0 |
| Phosphorous (mg/kg) | 69 | 42 | 76 | 6 | 310 |
| Potassium (mg/kg) | 159 | 277 | 534 | 51 | 227 |
| Magnesium (mg/kg) | 140 | 750 | 111 | 193 | 107 |
| Calcium (mg/kg) | 1000 | 2900 | 3140 | 1713 | 1558 |
| Sodium (mg/kg) | 70 | 32 | 39 | <20 | <20 |
| Total nitrogen (mg/kg) | 570 | 3190 | 1400 | 2183 | 1297 |
| Organic matter (%) | 0.8 | 6.3 | 1.9 | 4.3 | 2.4 |
| Organic carbon (%) | 0.5 | 3.7 | 1.1 | 2.5 | 1.4 |
| Soluble salt (mmhos/cm) | 0.18 | 0.61 | 0.90 | 0.25 | 0.27 |
| CEC (meq/100 g) | 9.0 | 26.6 | 10.3 | 12.7 | 10.1 |
| Bulk density ($g/cm^3$) | 1.21 | 1.02 | 1.23 | 1.07 | 1.28 |
| Moisture (%) | na | na | 17.7 | 22.9 | 15.2 |
| Moisture-holding capacity (%) at 0 bar | 45.8 | 72.3 | 45.3 | 49.3 | 33.3 |
| Moisture-holding capacity (%) at 1/3 bar | 10.8 | 31.3 | 25.6 | 30.8 | 17.1 |
| Moisture-holding capacity (%) at 1 bar | 7.7 | 26.5 | 15.5 | 20.8 | 11.1 |
| Moisture-holding capacity (%) at 15 bar | 5.4 | 20.4 | 8.8 | 11.2 | 6.9 |
| Sand (%) | 50 | 26 | 8.8 | 10.4 | 45.2 |
| Silt (%) | 40 | 52 | 74.4 | 70.8 | 40.8 |
| Clay (%) | 10 | 22 | 16.8 | 18.8 | 14.0 |
| USDA texture | loam | silt loam | silt loam | silt loam | loam |

na = data not available
% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

**Findings:**

The time to equilibration of IN-A2213 between soil and solution was approximately 3 to 8 hours based on results from a preliminary experiment. An equilibration time of 24 hours was chosen to ensure equilibrium for the definitive adsorption experiments. Total recoveries of radioactivity (single sample for Nijmegen, average of duplicates for other soils) ranged from 89.3-93.2% for the five soils.

IN-A2213 was poorly adsorbed to all soils. The linear adsorption coefficients for IN-A2213 are listed in Table . The KD values ranged from 0.051 to 0.20 mL/g and Koc values ranged from 4 to 11 mL/g (average = 7 mL/g). Freundlich sorption isotherm parameters are given in Table 41. The Freundlich adsorption constant, KF, ranged from 0.048 to 0.20 mL/g, with KFoc values ranging from 4 to 10 mL/g (average = 7 mL/g). The 1/n values ranged from 0.87 to 1.24 (average = 1.03). The Freundlich adsorption distribution coefficient (KF) increased with increasing percentage organic matter, indicating that organic matter content (or organic carbon) is a factor influencing the soil sorption of IN-A2213.

**Table 103 Summary of the linear soil adsorption coefficients for IN-A2213**

| Soil | USDA soil texture | % Organic matter | $K_D$ (mL/g) | $K_{om}$ (mL/g) | $K_{oc}$ (mL/g) |
|---|---|---|---|---|---|
| Commerce | loam | 0.8 | 0.051 | 6 | 10.9 |
| Drummer #6 | silt loam | 6.3 | 0.20 | 3 | 5.5 |
| Gross Umstadt | silt loam | 1.9 | 0.11 | 6 | 9.8 |
| Mattapex | silt loam | 4.3 | 0.11 | 2 | 4.3 |
| Nijmegen | loam | 2.4 | 0.066 | 3 | 4.8 |

Coefficients determined from the 5 ug/mL isotherm data (average of duplicates). Re-calculation of the average linear values using data reported for all isotherm concentrations gave similar results, with an average KD of approximately 0.092 mL/g and average Koc of 6 mL/g.

**Table 41 Summary of the Freundlich equation parameters for the IN-A2213 adsorption isotherms**

| Soil | USDA texture | % Organic matter | $K_F$ (mL/g) | 1/n | $K_{Fom}$ (mL/g) | $K_{Foc}$ (mL/g) |
|---|---|---|---|---|---|---|
| Commerce | loam | 0.8 | 0.048 | 1.24 | 5.97 | 10.31 |
| Drummer #6 | silt loam | 6.3 | 0.20 | 1.07 | 3.23 | 5.56 |
| Gross Umstadt | silt loam | 1.9 | 0.11 | 0.89 | 5.94 | 10.24 |
| Mattapex | silt loam | 4.3 | 0.12 | 0.87 | 2.77 | 4.77 |
| Nijmegen | loam | 2.4 | 0.052 | 1.06 | 2.17 | 3.74 |

IN-A2213 was readily desorbed from the one soil in which more than 10% of the applied material was originally adsorbed. The average percentage of the adsorbed IN-A2213 which was desorbed during two desorption intervals is listed in Table 42. A total of 70% of the adsorbed IN-A2213 was removed in the two desorption intervals.

**Table 42 Average percentage of sorbed IN-A2213 desorbed during two desorption intervals**

| Soil | USDA soil texture | % Organic matter | % of adsorbed IN-A2213 desorbed during the 1st desorption | % of adsorbed IN-A2213 desorbed during the 2nd desorption | Total % of adsorbed IN-A2213 desorbed |
|---|---|---|---|---|---|
| Drummer #6 | silt loam | 6.3 | 49.57 | 20.70* | 70.26 |

Less than 10% of the applied IN-A2213 at the 5.0 µg/mL test concentration was adsorbed to Commerce, Gross Umstadt, Mattapex, and Nijmegen soils; so they were not used in the desorption phase of the study.

* Reported as 41.03% of the total amount of IN-A2213 remaining in soil after the first desorption.

**Conclusions:**

IN-A2213 was weakly adsorbed to all the test soils. The adsorption coefficients showed a positive correlation to soil organic matter content. The average linear Koc and Freundlich KFoc values were both 7 mL/g. The average Freundlich 1/n value was 1.03. Sorption was reversible and the adsorbed IN-A2213 was readily desorbed from one test soil.

The adsorption and desorption of metabolites, breakdown and reaction products study DuPont-3929, Revision No. 1, originally submitted under EU Rev8 Point IIA 7.1.2 and conducted with test material [$^{14}$C]IN-A2213, was conducted under guidelines OECD 106 (1981) and U.S. EPA 163-1 (1982). A review of this study indicates that it fully meets the current guideline (OECD 106) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The substance IN-A2213 is classified as highly mobile due to its low Kd. The adsorption constants ($K_{foc}$ values) of IN-A2213 ranged from 3.7 to 10.3 The average Freundlich 1/n value was 1.03.

**B.8.1.3.1.2/02**

| Reference: | Report: | |
|---|---|---|
| -- | | Berg, D.S. (2000b); Adsorption/desorption of [$^{14}$C] IN-D2708 (a metabolite of oxamyl) in five soils<br>**DuPont Report No.:** DuPont-3930, Revision No. 1<br>**Guidelines:** OECD 106 (1981), U.S. EPA 163-1 (1982) |

1. Test material: [$^{14}$C]IN-D2708
   Lot/Batch #: HOTC 549
   Purity: Radiochemical purity - Not given

**Material and Methods:**

The adsorption/desorption of [ $^{14}$C]-IN-D2708 (specific activity 49.53 µCi/mg, radiochemical purity >95%) was investigated in the same five soil types used for IN-A2213 and oxamyl (Table 43).

The soils were pre-equilibrated with 0.01M calcium chloride prior to adsorption. Initial solution concentrations of 0.05, 0.1, 0.5, 1.0 and 5.0 µg/mL were prepared in 0.01M calcium chloride. Testing was conducted in duplicate with a soil/solution ratio of 1/1 w/v (10 g dry weight of soil/10 mL solution).

The soil/solution mixtures were equilibrated for 24 hours at 20 °C. Two 24-hour desorption cycles were conducted following the adsorption cycle. Only soils at the highest solution concentration (5.0 µg/mL), in which the average adsorption exceeded 5% in a preliminary experiment, were desorbed. Desorption was accomplished by completely decanting the supernatant (after centrifugation) and replacing it with an equivalent volume of fresh 0.01M calcium chloride solution.

Linear adsorption coefficients (KD) were calculated for the highest solution concentration samples (5.0 µg/mL) from the mean ratios of CS to CW, where CS and CW represent the test substance concentration in soil and in the aqueous phase at equilibrium, respectively. Adsorption data were further analysed using the log form of the Freundlich equation.

Aqueous samples were analysed by LSC. Soil samples from a preliminary study and the desorption study (both at 5.0 µg/mL) were analysed by combustion and LSC. The aqueous phase of the highest solution concentration (5.0 µg/mL) from each soil was analysed by reversed phase HPLC. The $^{14}$C-radioactivity mass balance was determined for preliminary study samples (5.0 µg/mL) by summing the amount in aqueous and soil phases after 24 hours equilibration.

**Table 43 Characteristics of the soils used in the IN-D2708 adsorption/desorption studies**

| Characteristic | Commerce | Drummer #6 | Gross Umstadt | Mattapex | Nijmegen |
|---|---|---|---|---|---|
| Soil origin | Greenville, MS, USA | Rochelle, IL, USA | Gross Umstadt, Germany | Rock Hall, MD, USA | Nijmegen, The Netherlands |
| DuPont soil number | L1133-100 | L1133-116 | L1133-46 | L1133-72 | L1133-82 |
| pH | 5.8 | 5.8 | 7.5 | 6.9 | 7.0 |
| Phosphorous (mg/kg) | 69 | 42 | 76 | 6 | 310 |
| Potassium (mg/kg) | 159 | 277 | 534 | 51 | 227 |
| Magnesium (mg/kg) | 140 | 750 | 111 | 193 | 107 |
| Calcium (mg/kg) | 1000 | 2900 | 3140 | 1713 | 1558 |
| Sodium (mg/kg) | 70 | 32 | 39 | <20 | <20 |
| Total nitrogen (mg/kg) | 570 | 3190 | 1400 | 2183 | 1297 |
| Organic matter (%) | 0.8 | 6.3 | 1.9 | 4.3 | 2.4 |
| Organic carbon (%) | 0.5 | 3.7 | 1.1 | 2.5 | 1.4 |
| Soluble salts (mmohos/cm) | 0.18 | 0.61 | 0.90 | 0.25 | 0.27 |
| CEC (meq/100 g) | 9.0 | 26.6 | 10.3 | 12.7 | 10.1 |
| Bulk density (g/cm$^3$) | 1.21 | 1.02 | 1.23 | 1.07 | 1.28 |
| Moisture (%) | na | na | 17.7 | 22.9 | 14.2 |
| Moisture-holding capacity (%) at 0 bar | 45.8 | 72.3 | 45.3 | 49.3 | 33.3 |
| Moisture-holding capacity (%) at 1/3 bar | 10.8 | 31.3 | 25.6 | 30.8 | 17.1 |
| Moisture-holding capacity (%) at 1 bar | 7.7 | 26.5 | 15.5 | 20.8 | 11.1 |
| Moisture-holding capacity (%) at 15 bar | 5.4 | 20.4 | 8.8 | 11.2 | 6.9 |
| Sand (%) | 50 | 26 | 8.8 | 10.4 | 45.2 |
| Silt (%) | 40 | 52 | 74.4 | 70.8 | 40.8 |
| Clay (%) | 10 | 22 | 16.8 | 18.8 | 14.0 |

| | | | | | |
|---|---|---|---|---|---|
| USDA texture | loam | silt loam | silt loam | silt loam | loam |

na = data not available
% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

**Findings:**

The time to equilibration of IN-D2708 between soil and solution was approximately 3 hours based on results from a preliminary experiment. An equilibration time of 24 hours was chosen to ensure equilibrium for the definitive adsorption experiments. Total recoveries of radioactivity (average of duplicates) ranged from 93.6% to 95.5% for the five soils.

The linear adsorption coefficients for IN-D2708 are listed in Table . The KD values ranged from 0.03 to 0.31 mL/g and Koc values ranged from 2 to 10 mL/g (average = 6 mL/g). Freundlich sorption isotherm parameters are given in Table . The adsorption constant, KF, ranged from 0.05 to 0.39 mL/g with KFoc values ranging from 6 to 14 (average = 10 mL/g). The 1/n values ranged from 0.532 to 0.762 (average = 0.668).

**Table 107 Summary of the averaged linear soil adsorption coefficients for IN-D2708**

| Soil | USDA soil texture | % Organic matter | $K_D$ (mL/g) | $K_{om}$ (mL/g) | $K_{oc}$ (mL/g) |
|---|---|---|---|---|---|
| Commerce | loam | 0.8 | 0.05 | 5.64 | 9.73 |
| Drummer #6 | silt loam | 6.3 | 0.31 | 4.97 | 8.57 |
| Gross Umstadt | silt loam | 1.9 | 0.07 | 3.94 | 6.79 |
| Mattapex | silt loam | 4.3 | 0.09 | 2.07 | 3.56 |
| Nijmegen | loam | 2.4 | 0.03 | 1.12 | 1.94 |

Coefficients determined from the 5 μg/mL isotherm data (average of duplicates). Re-calculation of the average linear values using data reported for all isotherm concentrations gave higher results, with an average $K_D$ of approximately 0.28 mL/g and average $K_{oc}$ of 17 mL/g.

**Table 108 Summary of the Freundlich equation parameters for the IN-D2708 adsorption isotherms**

| Soil | USDA texture | % Organic matter | $K_F$ (mL/g) | 1/n | $K_{Fom}$ (mL/g) | $K_{Foc}$ (mL/g) |
|---|---|---|---|---|---|---|
| Commerce | loam | 0.8 | 0.05 | 0.728 | 6.4 | 11.1 |
| Drummer #6 | silt loam | 6.3 | 0.39 | 0.762 | 6.2 | 10.7 |
| Gross Umstadt | silt loam | 1.9 | 0.15 | 0.532 | 8.1 | 13.9 |
| Mattapex | silt loam | 4.3 | 0.17 | 0.592 | 4.1 | 7.0 |
| Nijmegen | loam | 2.4 | 0.08 | 0.727 | 3.2 | 5.5 |

IN-D2708 was readily desorbed from the three soils in which more than 5% of the applied material was originally adsorbed in a preliminary experiment. The average percentage of the adsorbed IN-D2708 which was desorbed during two desorption intervals is listed in Table . A total of 70.9 to 82.1% of the adsorbed IN-D2708 was removed in the two desorption intervals.

**Table 109 Average percentage of sorbed IN-D2708 desorbed during two desorption intervals**

| Soil | USDA soil texture | % Organic matter | % of adsorbed IN-D2708 desorbed during the 1st desorption | % of adsorbed IN-D2708 desorbed during the 2nd desorption | Total % of adsorbed IN-D2708 desorbed |
|---|---|---|---|---|---|
| Drummer #6 | silt loam | 6.3 | 50.4 | 20.5 | 70.9 |
| Mattapex | silt loam | 4.3 | 57.5 | 19.6 | 77.1 |
| Gross Umstadt | silt loam | 1.9 | 59.9 | 22.2 | 82.1 |

Less than 5% of the applied IN-D2708 at the 5.0 µg/mL test concentration was adsorbed to Commerce and Nijmegen soils in a preliminary experiment; so they were not used in the desorption phase of the study.

The percentages of adsorbed IN-D2708 desorbed during each interval were re-calculated from the IN-D2708 mass data presented in the report, apart from the amount desorbed from Mattapex soil during the first interval.

**Conclusions:**

IN-D2708 was weakly adsorbed to all the test soils. The amount of soil organic matter appeared to be the main factor affecting the adsorption process, with the adsorption coefficients showing a positive correlation to soil organic matter content. The average linear Koc value was 6 mL/g and the average Freundlich KFoc value was 10 mL/g. The average Freundlich 1/n value was 0.67. Sorption was reversible, with adsorbed IN-D2708 being readily desorbed from the soils that were tested. The total amounts of adsorbed IN-D2708 that were desorbed were 70.9% for Drummer #6 soil, 77.1% for Mattepex soil and 82.1% for Gross Umstadt soil.

The adsorption and desorption of metabolites, breakdown and reaction products study DuPont-3930, Revision No. 1, originally submitted under EU Rev8 Point IIA 7.1.2 and conducted with test material [$^{14}$C]IN-D2708, was conducted under guidelines OECD 106 (1981) and U.S. EPA 163-1 (1982). A review of this study indicates that it fully meets the current guideline (OECD 106).

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The substance IN-D2708 is classified as highly mobile due to its low Kd. The adsorption constants ($K_{foc}$ values) of IN-D2708 ranged from 5.5 to 13.9 The average Freundlich 1/n value was 0.67.

**B.8.1.3.1.2/03**

| **Reference:** -- | **Report:** | Berg, D.S. (2000c); Adsorption/desorption of [$^{14}$C] IN-N0079 in five soils<br>**DuPont Report No.:** DuPont-3931<br>**Guidelines:** OECD 106 (1981), U.S. EPA 163-1 (1982) |
|---|---|---|

1. Test material: [$^{14}$C]IN-N0079
   Lot/Batch #: HOTC 548
   Purity: Radiochemical purity - >95%

**Material and Methods:**

The adsorption/desorption of [ $^{14}$C]-IN-N0079 (specific activity 45 µCi/mg, radiochemical purity >95%) was investigated in the same five soil types used for oxamyl and the other metabolites (Table ).

The soils were pre-equilibrated with 0.01M calcium chloride prior to adsorption. An initial solution concentration of 5.0 µg/mL was prepared in 0.01M calcium chloride. Testing was conducted in duplicate with a soil/solution ratio of 1/1 w/v (10 g dry weight of soil/10 mL solution). Due to instability of the test substance in the presence of soil, the soil/solution mixtures were equilibrated for one hour at 5 °C and only the 5.0 µg/mL concentration was tested. Desorption was not conducted due to the substance instability.

Linear adsorption coefficients (KD) were calculated from the mean ratios of CS to CW, where CS and CW represent the test substance concentration in soil and in the aqueous phase at equilibrium, respectively.

Aqueous samples were analysed by LSC and reversed phase HPLC. Soil samples were analysed by combustion and LSC. The 14C-radioactivity mass balance was determined by summing the amount in aqueous and soil phases after the 1-hour equilibration.

**Table 110Characteristics of the soils used in the IN-N0079 adsorption/desorption studies**

| Characteristic | Commerce | Drummer#6 | Gross Umstadt | Mattapex | Nijmegen |
|---|---|---|---|---|---|
| Soil origin | Greenville, MS, USA | Rochelle, IL, USA | Gross Umstadt, | Rock Hall, | Nijmegen, The |
| DuPont soil number | L1133-100 | L1133-116 | L1133-46 | L1133-72 | L1133-82 |
| pH | 5.8 | 5.8 | 7.5 | 6.9 | 7.0 |
| Phosphorous (mg/kg) | 69 | 42 | 76 | 6 | 310 |
| Potassium (mg/kg) | 159 | 277 | 534 | 51 | 227 |
| Magnesium (mg/kg) | 140 | 750 | 111 | 193 | 107 |
| Calcium (mg/kg) | 1000 | 2900 | 3140 | 1713 | 1558 |
| Sodium (mg/kg) | 70 | 32 | 39 | <20 | <20 |
| Total nitrogen (mg/kg) | 570 | 3190 | 1400 | 2183 | 1297 |
| Organic matter (%) | 0.8 | 6.3 | 1.9 | 4.3 | 2.4 |
| Organic carbon (%) | 0.5 | 3.7 | 1.1 | 2.5 | 1.4 |
| Soluble salts (mmohos/cm) | 0.18 | 0.61 | 0.90 | 0.25 | 0.27 |
| CEC (meq/100 g) | 9.0 | 26.6 | 10.3 | 12.7 | 10.1 |
| Bulk density ($g/cm^3$) | 1.21 | 1.02 | 1.23 | 1.07 | 1.28 |
| Moisture (%) | na | na | 17.7 | 22.9 | 14.2 |
| Moisture-holding capacity (%) at 0 bar | 45.8 | 72.3 | 45.3 | 49.3 | 33.3 |
| Moisture-holding capacity (%) at 1/3 bar | 10.8 | 31.3 | 25.6 | 30.8 | 17.1 |
| Moisture-holding capacity (%) at 1 bar | 7.7 | 26.5 | 15.5 | 20.8 | 11.1 |
| Moisture-holding capacity (%) at 15 bar | 5.4 | 20.4 | 8.8 | 11.2 | 6.9 |
| Sand (%) | 50 | 26 | 8.8 | 10.4 | 45.2 |
| Silt (%) | 40 | 52 | 74.4 | 70.8 | 40.8 |
| Clay (%) | 10 | 22 | 16.8 | 18.8 | 14.0 |
| USDA texture | loam | silt loam | silt loam | silt loam | loam |

na = data not available
% organic carbon = (% organic matter)/1.724, 1.724 = Van Bemmelen factor

**Findings:**

Due to the instability of IN-N0079 in the presence of soil observed in a 24-hour preliminary experiment, a 1-hour equilibration time was chosen. Samples were also equilibrated at 5 °C in order to reduce breakdown of IN-N0079. Total recoveries of radioactivity ranged from 93.4 to 99.7% for the five soils.

The linear adsorption coefficients for IN-N0079 are listed in Table . The KD values ranged from 0.03 to 0.31 mL/g and Koc values ranged from 2 to 25 (average = 8 mL/g).

IN-N0079 was unstable in the presence of soil. Meaningful desorption experiments could not be conducted under the test conditions.

**Table 111 Summary of the averaged linear soil adsorption coefficients for IN-N0079**

| Soil | USDA soil texture | % Organic matter | $K_D$ (mL/g) | $K_{om}$ (mL/g) | $K_{oc}$ (mL/g) |
|---|---|---|---|---|---|
| Commerce | loam | 0.8 | 0.12 | 14.7 | 25.41 |
| Drummer #6 | silt loam | 6.3 | 0.31 | 4.88 | 8.41 |
| Gross Umstadt | silt loam | 1.9 | 0.06 | 2.99 | 5.16 |
| Mattapex | silt loam | 4.3 | 0.04 | 0.89 | 1.53 |
| Nijmegen | loam | 2.4 | 0.03 | 1.28 | 2.20 |

**Conclusions:**

IN-N0079 was unstable under room temperature test conditions with a 24-hour equilibration time. IN-N0079 proved to have greater stability at reduced temperature and adsorption time, providing KD values which ranged from 0.03 to 0.31 mL/g, Kom values which ranged from 0.89 to 14.70 mL/g and Koc values which ranged from 1.53 to 25.41 mL/g. These values were obtained from a 1-hour, 5 °C adsorption equilibrium. IN-N0079 is classified as highly mobile due to its low KD value.

The adsorption and desorption of metabolites, breakdown and reaction products study DuPont-3931, originally submitted under EU Rev8 Point IIA 7.1.2 and conducted with test material [$^{14}$C]IN-N0079, was conducted under guidelines OECD 106 (1981) and U.S. EPA 163-1 (1982). A review of this study indicates that it fully meets the current guideline (OECD 106) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The substance IN-N0079 is classified as highly mobile due to its low Kd. The adsorption constants ($K_{oc}$ values) of IN-0079 ranged from 1.5 to 25.4

#### B.8.1.3.2 Aged sorption

It was not necessary to conduct and report such a study since reliable absorption coefficient values have been obtained for the main metabolites of oxamyl in the adsorption/desorption studies reported (Point B.8.1.3.1.2 in this document).

### B.8.1.4 Mobility studies

#### B.8.1.4.1 Column leaching studies

##### *B.8.1.4.1.1 Column leaching studies with the active substance*

Column leaching studies with oxamyl were not conducted since reliable absorption coefficient values have been obtained in adsorption/desorption studies reported (Point B.8.1.3.1.1 in this document).

##### *B.8.1.4.1.2 Column leaching studies with relevant metabolites, breakdown and reaction products*

Column leaching studies with soil degradation products IN-A2213, IN-D2708 and IN-N0079 were not conducted since reliable absorption coefficient values have been obtained in adsorption/desorption studies reported (Point B.8.1.3.1.2 in this document).

#### B.8.1.4.2 Lysimeter studies

A lysimeter study was not conducted with oxamyl. Data from the adsorption/desorption studies, the aerobic soil degradation studies, and field dissipation studies can be used to adequately define the leaching potential of the parent compound and its metabolites in soil. Though oxamyl is classified as a mobile compound, its rapid degradation limits its potential to persist in the soil column long enough to leach to groundwater. The lack of leaching potential of oxamyl in soil was demonstrated in the modelling done in support of the various proposed uses. The $PEC_{gw}$ modelling demonstrated low risk for oxamyl concentrations in groundwater to exceed 0.1 ppb. Field dissipation studies also sufficiently demonstrated the extent of movement of oxamyl and its major metabolites in the soil column. In the field dissipation trial in the Netherlands, there was no detectable oxamyl residue below 45 cm, no detectable IN-A2213 residue below 30 cm, and no detectable IN-D2708 residue below 15 cm. In the UK field trial, maximum average oxamyl and IN-A2213 residues were only observed at the quantitation limit (0.005 mg/kg) in the 75–90 cm depth segment while IN-D2708 was not observed below the 30–45 cm segment. Maximum average oxamyl and IN-A2213 levels in the 75–90 cm segment were low, at 0.0063 mg/kg and 0.0062 mg/kg, respectively. In addition, saturated zone soil degradation testing demonstrated

that oxamyl can degrade in the saturated zone under a variety of pH and reduction-oxidation conditions at 10°C (Point B.8.2.3 in this document).

#### B.8.1.4.3 Field leaching studies

A field leaching study was not conducted since the data from the adsorption/desorption studies and the aerobic soil degradation studies can be used to adequately define the leaching potential of oxamyl and its metabolites in soil.

#### B.8.1.4.4 Overall assessment: Mobility in soil

Data from the batch equilibrium adsorption studies show that oxamyl is weakly absorbed to soils with $K_{foc}$ values ranging from 4.1 to 37 mL/g. Despite the adsorption results, which suggest that oxamyl is mobile in soils, leaching to groundwater is not likely due to the rapid degradation rates of oxamyl in soil. This lack of leaching was confirmed by FOCUS groundwater modelling (Oxamyl EU Renewal Dossier, Document M-CP, Section 9 [DuPont-40953 EU and DuPont-42129 EU]), where the PEC groundwater for oxamyl did not exceed 0.1 µg/L in the majority of scenarios of the proposed GAP.

The batch equilibrium adsorption studies suggest that the major soil metabolites of oxamyl are also weakly adsorbed to soil. IN-A2213 had $K_{foc}$ values ranging from 3.7 to 10.3 mL/g, IN-D2708 had $K_{foc}$ values ranging from 5.7 to 13.6 mL/g, and IN-N0079 had $K_{foc}$ values ranging from 1.6 to 25.9 mL/g. However, just like oxamyl, IN-A2213, IN-D2708, and IN-N0079 all quickly degrade in soil, limiting their actual potential to migrate into groundwater at unacceptable levels.

Two field soil dissipation studies conducted in typical potato-producing regions of The Netherlands and the UK (England) support the mobility conclusions above. Both studies were conducted in non-cropped fields (bare ground conditions) in separate seasons. Sampling was conducted to 90 cm at both sites to assess the total mass of oxamyl, IN-A2213, and IN-D2708 in the extended soil profile over time. At the site in The Netherlands, where oxamyl was broadcast-incorporated at the maximum country use rate of 4.0 kg a.s./ha, oxamyl was not detected (LOD = 0.001 mg/kg) below 45 cm, IN-A2213 was not detected (LOD = 0.001 mg/kg) below 30 cm, and IN-D2708 was not detected (LOD = 0.005 mg/kg) below 15 cm. At the site in England, where oxamyl was broadcast-incorporated at the maximum use rate specified on the GAP, 5.5 kg a.s./ha, the maximum average oxamyl soil concentration in the deepest segment sampled, 75–90 cm, was just above the quantitation limit of 0.005 mg/kg (0.0096 mg/kg). The maximum average IN-A2213 levels observed in the 75-90 cm segment were also near the quantitation limit of 0.005 mg/kg (0.0096 mg/kg). IN-D2708 was not observed below the 30–45 cm segment, where the maximum concentration from any plot was equal to the quantitation limit of 0.01 mg/kg.

In addition, saturated zone soil degradation testing demonstrated that oxamyl can degrade in the saturated zone under a variety of pH and reduction-oxidation conditions at 10°C (Point B.8.2.3 in this document). This result further mitigates leaching risks for oxamyl because the compound will continue to degrade significantly even if it moves to lower saturated soil zones.

Actual ground water monitoring data from the UK Environment Agency further demonstrate that oxamyl is not likely to adversely impact ground water under actual use conditions. Data were obtained from the Environment Agency database for 1992 to 1997 (DuPont-3145, in Point B.8.5 in this document). These data are generally obtained in response to particular concerns or incidents and are not the results of a detailed random monitoring program. Therefore, the data do not present a comprehensive picture of oxamyl levels in ground water, but are likely to show the worst case situations. Two regions (Anglia and Welsh) were monitored for oxamyl. A total of 132 samples were analysed from the Anglia region, where significant oxamyl use is likely, and 27 from the Welsh region, where oxamyl use is unlikely. The method detection limit was <0.1 µg/L for all samples analysed except one from the Anglia region where the detection limit was reported as <0.25 µg/L. Oxamyl was not detected (<0.1 µg/L) in any of the samples from the Welsh region. Only three of 132 samples from the Anglia region were reported at levels exceeding 0.1 µg/L (0.18, 0.329, and 0.541 µg/L). It should be noted that these samples represent a single time point, not an annual average, which is likely to be lower than single time point samples collected during the growing season.

The overall conclusion is that oxamyl is potentially mobile, but the leaching potential is mitigated by rapid degradation in viable agricultural soil. Degradation of oxamyl in the saturated zone may also occur, further

limiting the role that leaching may have on entry or persistence of oxamyl in ground water. This conclusion is supported by the aerobic degradation studies, batch equilibrium sorption study, field soil dissipation studies, saturated zone degradation studies, and $PEC_{gw}$ modeling. The conclusions regarding the mobility of the major soil degradates of oxamyl, IN-A2213, and IN-D2708, are similar to that for oxamyl. Moreover, the mammalian toxicity (Oxamyl EU Renewal Dossier, Document M-CA, Section 5, DuPont-40932 EU, Points CA 5.8.1 and CA 5.8.2), ecotoxicity (Oxamyl EU Renewal Dossier, Document M-CA, Section 8, DuPont-40935 EU, Points CA 8.2.1, CA 8.2.2, CA 8.2.5.4, and CA 8.2.6), and insecticidal activity (Oxamyl EU Renewal Dossier, Document M-CA, Section 8, DuPont-40935 EU, Point CA 8.2.7) of IN-A2213 and IN-D2708 have been assessed. These degradates have been shown to have low to negligible ecotoxicity and toxicity and are not insecticidally active. Therefore, the major soil degradates should be considered non-relevant.

## B.8.2 Fate and behaviour in water and sediment

### B.8.2.1 Route and rate of degradation in aquatic systems (chemical and photochemical degradation)

#### B.8.2.1.1 Hydrolytic degradation

**Study submitted to the EU for the first time in this submission.**

**B.8.2.1.1/01**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.2.1.1/01** | **Report:** | Clark, B. (2014); Hydrolysis of [$^{14}$C]-DPX-D1410 (oxamyl) as a function of pH<br>**DuPont Report No.:** DuPont-39015<br>**Guidelines:** OPPTS 835.2120 (2008), SETAC (1995), OECD 111 (2004)<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** ABC 80582<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

Hydrolysis of radiolabeled oxamyl at 0.928–1.04 µg/mL was studied in the dark at 20, 25, and 30°C in sterile aqueous buffered solutions at pH 4, pH 7, and pH 9 for up to 30 days. Acetonitrile (<0.5%) was used as a co-solvents. Total recovery of radioactivity ranged from 97.8–100.7%.

In the preliminary experiment, oxamyl was shown to be stable at pH 4, so this pH was not run in the definitive experiment. At pH 7, the major transformation product detected was IN-A2213, with a maximum mean concentration of 90.0% AR, observed on the 14.78 day of incubation/at test termination. At pH 9, the major transformation product detected was IN-A2213, with a maximum mean concentration of 93.4% AR, observed on the 0.19 day of incubation/at test termination. The first-order $DT_{50}$ values of oxamyl were 21.1, 9.01, and 4.16 days at 20, 25, and 30°C for pH 7, respectively. The first-order $DT_{50}$ values of oxamyl were 0.200, 0.098, and 0.046 days at 20, 25, and 30°C for pH 9, respectively.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabeled test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: 1841000
   Radiochemical purity: 99.7%
   Specific activity: 75.6 μCi/mg
   Description: Solid
   Stability of test compound: Not determined
2. Stable-labeled test material: [$^{13}$C]oxamyl
   Lot/Batch #: MBBB0508V
   Radiochemical purity: 98%
   Description: Solid
   Stability of test compound: Not determined

4. Buffers:

0.1 M buffer solutions in HPLC grade water were prepared at pH 4, using acetate, pH 7 using phosphate, and pH 9 using borate.

B. STUDY DESIGN

1. Experimental conditions

Hydrolysis of radiolabeled oxamyl at 0.928–1.04 μg/mL was studied in the dark at 20, 25, and 30 °C in sterile aqueous buffered solutions at pH 4 (0.01M acetate), pH 7 (0.01M phosphate), and pH 9 (0.01M borate) for 30 days. The test solutions (1 mL) were sterilised by autoclaving and were placed in 50 to 200-mL glass flasks. Samples were analysed at the timepoints shown in Table through Table and the oxamyl residues were analysed by LSC and HPLC. Identification of the transformation products was done by HPLC/UV with in-line radiodetection. The LOD for oxamyl was 11 dpm. The LOQ for oxamyl was 43 dpm.

## II. RESULTS AND DISCUSSION

A. MASS BALANCE

Recovery of radiolabel ranged from 97.8–100.7% for the oxamyl label.

B. FINDINGS

Hydrolysis of oxamyl occurred over the pH range 7–9 and was most rapid at pH 9. Hydrolysis rate increased substantially at the higher temperature (30 °C). At test termination, the concentration of the parent compound decreased from 97.1% at Day 0 to 37.3%, 98.6 to 9.7%, and 97.1 t 8.0% of the applied radioactivity (AR) at 20, 25, and 30°C for pH 7, and from 97.0 to 29.8%, 94.5 to 11.6%, and 94.6 to 3.6% AR at 20, 25, and 30 °C for pH 9. The first-order $DT_{50}$ values of oxamyl were 21.1, 9.01, and 4.16 days at 20, 25, and 30 °C for pH 7, respectively. The first-order $DT_{50}$ values of oxamyl were 0.200, 0.098, 0.046 days at 20, 25, and 30 °C for pH 9, respectively.

**Table 112Hydrolytic half-lives and rate constants for oxamyl**

| Analyte | pH | Temp | Half-life (day) | k ($day^{-1}$) | $r^2$ | Method of calculation |
|---|---|---|---|---|---|---|
| Oxamyl | 4 | --- | STABLE | STABLE | --- | First-order |
| | 7 | 20 | 21.1 | 0.0328 | 0.9965 | |
| | | 25 | 9.01 | 0.0769 | 0.9984 | |
| | | 30 | 4.16 | 0.167 | 0.9986 | |
| | 9 | 20 | 0.200 | 3.46 | 0.9926 | |
| | | 25 | 0.098 | 7.09 | 0.9974 | |
| | | 30 | 0.046 | 15.1 | 0.9851 | |

At pH 7, the major transformation product detected was IN-A2213, with a maximum concentration of 90.0% AR, observed on the 14.78 day of incubation/at test termination (Table ). At pH 9, the major transformation product detected was IN-A2213, with a maximum concentration of 93.4% AR, observed on the 0.19 day of incubation/at test termination (Table ). Minor components detected represented <1% of the applied radioactivity individually.

**Table 113Hydrolysis of oxamyl at pH 7, 20°C (expressed as mean percentage of the applied radioactivity)**

| | Sampling times (days) | | | | | | |
|---|---|---|---|---|---|---|---|
| **Compound** | **0.00** | **1.72** | **2.77** | **7.74** | **14.76** | **23.78** | **29.67** |
| Oxamyl | 97.1 | 90.7 | 88.3 | 73.7 | 59.1 | 42.9 | 37.3 |
| IN-A2213 | 0.00 | 6.6 | 9.2 | 23.4 | 39.3 | 52.4 | 62.7 |
| Unidentified radioactivity, if any | 2.9 | 2.2 | 2.2 | 3.1 | 1.2 | 0.8 | 0.3 |
| Total % recovery | 100.0 | 99.6 | 99.7 | 100.2 | 99.6 | 96.1 | 100.4 |

**Table 114 Hydrolysis of oxamyl at pH 7, 25°C (expressed as mean percentage of the applied radioactivity)**

| | Sampling times (days) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Compound** | **0.00** | **2.03** | **2.99** | **7.05** | **14.80** | **19.89** | **24.07** | **29.99** |
| Oxamyl | 98.6 | 83.1 | 80.5 | 54.8 | 31.5 | 21.5 | 15.6 | 9.7 |
| IN-A2213 | 0.0 | 14.3 | 20.2 | 44.4 | 70.7 | 79.1 | 83.0 | 88.7 |
| Unidentified radioactivity, if any | 1.4 | 2.3 | 0.3 | 1.6 | 0.0 | 1.1 | 1.4 | 1.3 |
| Total % recovery | 100.0 | 99.7 | 101.1 | 100.8 | 102.1 | 101.7 | 100.0 | 99.7 |

**Table 115 Hydrolysis of oxamyl at pH 7, 30°C (expressed as mean percentage of the applied radioactivity)**

| | Sampling times (days) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Compound** | **0.00** | **0.84** | **1.72** | **2.91** | **4.02** | **6.93** | **9.76** | **14.78** |
| Oxamyl | 97.1 | 80.8 | 72.8 | 58.3 | 49.1 | 29.6 | 19.7 | 8.0 |
| IN-A2213 | 0.0 | 16.0 | 24.5 | 40.2 | 47.3 | 68.0 | 78.8 | 90.0 |
| Unidentified radioactivity, if any | 2.9 | 3.8 | 2.3 | 2.6 | 3.3 | 2.3 | 1.1 | 1.1 |
| Total % recovery | 100.0 | 100.5 | 99.6 | 101.0 | 99.7 | 100.0 | 99.5 | 99.1 |

**Table 116 Hydrolysis of oxamyl at pH 9, 20°C (expressed as mean percentage of the applied radioactivity)**

| | Sampling times (days) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Compound** | **0.00** | **0.05** | **0.12** | **0.17** | **0.21** | **0.25** | **0.29** | **0.34** |
| Oxamyl | 97.0 | 79.8 | 59.8 | 53.7 | 44.3 | 39.1 | 35.6 | 29.8 |
| IN-A2213 | 2.0 | 16.9 | 36.5 | 46.0 | 51.6 | 58.6 | 62.0 | 66.6 |
| Unidentified radioactivity, if any | 1.0 | 1.7 | 0.9 | 0.4 | 1.8 | 0.9 | 0.7 | 1.3 |
| Total % recovery | 100.0 | 98.5 | 97.2 | 100.1 | 97.6 | 98.6 | 98.2 | 97.7 |

**Table 117 Hydrolysis of oxamyl at pH 9, 25°C (expressed as mean percentage of the applied radioactivity)**

| Compound | Sampling times (days) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | **0.00** | **0.04** | **0.08** | **0.13** | **0.17** | **0.21** | **0.25** | **0.30** |
| Oxamyl | 94.5 | 71.9 | 56.2 | 41.8 | 30.7 | 23.0 | 16.2 | 11.6 |
| IN-A2213 | 3.3 | 27.5 | 43.2 | 57.6 | 66.8 | 75.9 | 82.9 | 87.6 |
| Unidentified radioactivity, if any | 2.2 | 1.9 | 1.7 | 0.9 | 1.2 | 1.1 | 0.9 | 1.3 |
| Total % recovery | 100.0 | 101.3 | 101.1 | 100.3 | 98.7 | 100.0 | 100.0 | 100.5 |

**Table 118 Hydrolysis of oxamyl at pH 9, 30°C (expressed as mean percentage of the applied radioactivity)**

| Compound | Sampling times (days) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | **0.00** | **0.04** | **0.07** | **0.10** | **0.13** | **0.16** | **0.19** | **0.22** |
| Oxamyl | 94.6 | 58.9 | 35.0 | 19.9 | 12.1 | 7.7 | 5.8 | 3.6 |
| IN-A2213 | 3.9 | 38.0 | 62.4 | 77.9 | 81.2 | 89.9 | 93.4 | 93.1 |
| Unidentified radioactivity, if any | 1.5 | 1.0 | 1.2 | 1.2 | 0.9 | 0.7 | 0.6 | 0.8 |
| Total % recovery | 100.0 | 97.8 | 98.5 | 99.0 | 94.2 | 98.2 | 99.8 | 97.5 |

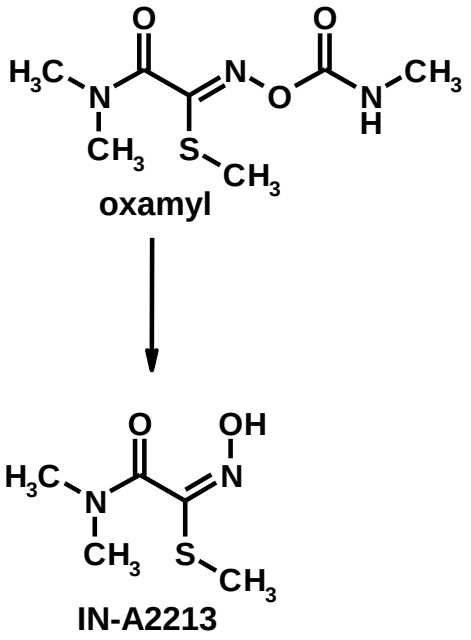
## III. CONCLUSION

The hydrolysis of oxamyl in sterile buffer solutions was temperature and pH dependent, and followed first order reaction kinetics. Oxamyl degraded most rapidly at pH 9, followed by pH 7. Oxamyl was shown to be hydrolytically stable at acidic pH 4. The rate of degradation was temperature dependent and increased with increasing temperature. First-order $DT_{50}$ values are 21.1, 9.01, and 4.16 days at 20, 25, and 30°C for pH 7, respectively and 0.200, 0.098, 0.046 days at 20, 25, and 30°C for pH 9, respectively. Hydrolysis products measured at greater than 10% of the applied were IN-A2213.

A proposed hydrolytic degradation pathway is outlined in Figure 7.

(Clark, B., 2014)

**Figure 7 Proposed degradation pathway of oxamyl (DPX-D1410) under hydrolytic conditions**



The hydrolytic degradation study DuPont-39015, submitted for the first time in this submission and conducted with test material [$^{14}$C]IN-D2708, was conducted under guidelines OPPTS 835.2120 (2008), SETAC (1995), OECD 111 (2004). A review of this study indicates that it fully meets the current guideline (OECD 111) and is relied upon.

**RMS comments and conclusion**

This study demonstrated the hydrolysis of oxamyl in buffer solutions was temperature and pH dependent.
Oxamyl was shown to be hydrolytically stable at acidic pH 4. Oxamyl degraded most rapidly at pH 9, followed by pH 7.
The rate of degradation was temperature dependent and increased with increasing temperature. Only one major metabolite (IN-A2213) was observed in this study

**B.8.2.1.1/02**

| **Reference:** -- | **Report:** | Lee, D.Y., Berg, D.S. (2001); Hydrolysis of IN-A2213 in buffer solutions of pH 4, 7, and 9<br>**DuPont Report No.:** DuPont-4024<br>**Guidelines:** SETAC Europe (1995), U.S. EPA 161-1 (1982) |
|---|---|---|

1. Test material: [$^{14}$C]IN-A2213
   Lot/Batch #: HOTC 563
   Purity: Radiochemical purity - >96%

**Materials and Methods:**

The hydrolytic stability of IN-A2213 was studied in aqueous solution buffered at pH 4, 7, and 9. [ $^{14}$C]-IN-A2213 (specific activity 25.2 µCi/mg, radiochemical purity >96%) was used as the test substance. Solutions containing IN-A2213 were prepared at nominal concentrations of 1.7 mg/L and were incubated for up to 34 days at 20 ±1 °C under sterile conditions in the dark.

Aliquots were removed from each test system with a pre-sterilised pipette under aseptic conditions into glass HPLC auto-injector vials for immediate analysis. Samples for all three pH values were taken immediately after treatment (day 0) and at designated sampling intervals thereafter, with testing being done in duplicate. The aqueous samples were analysed by LSC and reversed phase HPLC with radiochemical detection. Degradation products were identified by co-chromatography with authentic standards.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Findings:**

Material balance of radioactivity ranged from 99% to 106% of the applied in individual replicates. Average values for duplicates ranged from 100% to 104%. Sterility tests conducted at day 0 and later confirmed that all test systems remained sterile throughout the study.

IN-A2213 was stable at each pH. The composition of the radioactivity in buffer solutions at each pH is given in Table .

The DT50 values obtained for IN-A2213 are shown in Table . Half-life estimates at each pH were extrapolated well beyond the length of the study, demonstrating stability.

**Table 119 Percent composition of radioactivity in pH 4, pH 7 and pH 9 buffers at 20 °C**

| Day (replicate) | pH 4 | | pH 7 | | pH 9 | |
|---|---|---|---|---|---|---|
| | IN-A2213 | Unknowns | IN-A2213 | Unknowns | IN-A2213 | Unknowns |
| 0 (A) | 98.14 | 1.86 | 93.93 | 6.07 | 100.0 | 0.00 |
| 0 (B) | 96.77 | 3.23 | 96.72 | 3.28 | 100.0 | 0.00 |
| 1 (A) | 95.01 | 4.99 | 97.50 | 2.50 | 98.77 | 1.23 |
| 1 (B) | 95.85 | 4.15 | 98.99 | 1.01 | 99.21 | 0.79 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 3 (A) | 96.50 | 3.50 | 93.33 | 6.67 | 98.12 | 1.88 |
| 3 (B) | 74.23* | 25.77 | 96.70 | 3.30 | 96.68 | 3.32 |
| 7 (A) | 99.50 | 0.50 | 97.26 | 2.74 | 97.29 | 2.71 |
| 7 (B) | 96.29 | 3.71 | 94.71 | 5.29 | 96.96 | 3.04 |
| 14 (A) | 96.52 | 3.48 | 95.73 | 4.27 | 97.94 | 2.06 |
| 14 (B) | 97.21 | 2.79 | 98.83 | 1.17 | 97.94 | 2.06 |
| 21 (A) | 98.90 | 1.10 | 98.72 | 1.28 | 96.26 | 3.74 |
| 21 (B) | 97.60 | 2.40 | 97.84 | 2.16 | 97.46 | 2.54 |
| 34 (A) | 98.94 | 1.06 | 99.23 | 0.77 | 90.74 | 9.26 |
| 34 (B) | 97.05 | 2.95 | 98.32 | 1.68 | 96.45 | 3.55 |

* Value considered suspect and not used in further data analysis.

**Table 120Hydrolytic half-lives and rate constants for IN-A2213 at 20 °C**

| pH | First order $DT_{50}$ (day) | Rate ($day^{-1}$) | Method of calculation |
|---|---|---|---|
| 4 | 1386 (stable) | 0.0005 | linear simple first order |
| 7 | 770 (stable) | 0.0009 | linear simple first order |
| 9 | 462 (stable) | 0.0015 | linear simple first order |

The half-life estimates are extrapolated well beyond the length of the study. While quantitatively unreliable, the estimates demonstrate that IN-A2213 is stable under the conditions and length of the test.

**Conclusions:**

IN-A2213 is hydrolytically stable at pH 4, 7 and 9 for greater than 34 days at 20 ±1 °C.

The hydrolytic degradation study DuPont-4024, originally submitted under EU Rev8 Point IIA 7.2.1.1 and conducted with test material [$^{14}$C]IN-A2213, was conducted under guidelines SETAC Europe (1995) and U.S. EPA 161-1 (1982). A review of this study indicates that it fully meets the current guideline (OECD 111) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The substance IN-A2213 is considered stable at all pH's tested. The hydrolysis half-lives at pH 4, 7, and 9 were 1386 days, 770 days, and 462 days respectively.

**B.8.2.1.1/03**

| **Reference:** -- | **Report:** | Lee, D.Y., Berg, D.S. (2001); Hydrolysis of IN-D2708 in buffer solutions of pH 4, 7, and 9<br>**DuPont Report No.:** DuPont-4388<br>**Guidelines:** U.S. EPA 161-1 (1982), SETAC Europe (1995) |
|---|---|---|

1. Test material: [$^{14}$C]IN-D2708
   Lot/Batch #: HOTC 549
   Purity: Radiochemical purity - >98%

**Materials and Methods:**

The hydrolytic stability of IN-D2708 was studied in sterile aqueous solutions buffered at pH 4, 7, and 9. [ $^{14}$C]-IN-D2708 (specific activity 45.53 µCi/mg, radiochemical purity >98%) was used as the test substance. Solutions containing IN-D2708 were prepared at nominal concentrations of 1.0 mg/L and were incubated for up to 30 days at 20 ±1 °C under sterile conditions in the dark.

Aliquots were removed from each test system with a pre-sterilised pipette under aseptic conditions into glass HPLC auto-injector vials for immediate analysis. Samples for all three pH values were taken immediately after treatment (day 0) and at designated sampling intervals thereafter, with testing being done in duplicate. The aqueous samples were analysed by LSC and reversed phase HPLC with radiochemical detection. Degradation products were identified by co-chromatography with authentic standards.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Findings:**

Material balance of radioactivity ranged from 96% to 106% of applied radiolabel in individual replicates. Average values for duplicates ranged from 97% to 106%. Sterility tests conducted at day 0 and later confirmed that all test systems remained sterile throughout the study.

IN-D2708 was stable at each pH. Trace levels of IN-T2921, typically <1.6%, were reported in some samples, but there was no clear evidence that this was due to a reaction involving IN- D2708. The composition of the radioactivity in buffer solutions at each pH is given in Table .

The DT50 values obtained for IN-D2708 are shown in Table . Half-life estimates at each pH were extrapolated well beyond the length of the study, demonstrating stability.

**Table 121 Percent composition of radioactivity in pH 4, pH 7 and pH 9 buffers at 20 °C**

| Day (rep) | pH 4 | | | pH 7 | | | pH 9 | | |
|---|---|---|---|---|---|---|---|---|---|
| | IN-D2708 | IN-T2921 | Others | IN-D2708 | IN-T2921 | Others | IN-D2708 | IN-T2921 | Others |
| 0 (A) | 96.36 | 0.00 | 3.64 | 97.96 | 0.00 | 2.04 | 96.03 | 0.00 | 3.97 |
| 0 (B) | 96.71 | 0.00 | 3.29 | 94.95 | 0.00 | 5.05 | 94.08 | 0.00 | 5.92 |
| 1 (A) | 96.74 | 1.09 | 2.17 | 95.75 | 0.92 | 3.33 | 97.54 | 0.00 | 2.46 |
| 1 (B) | 98.66 | 0.00 | 1.34 | 98.52 | 0.00 | 1.48 | 98.65 | 0.00 | 1.35 |
| 3 (A) | 94.45 | 0.00 | 5.55 | 98.03 | 0.00 | 1.97 | 96.44 | 1.52 | 2.04 |
| 3 (B) | 98.11 | 0.00 | 1.89 | 95.86 | 0.00 | 4.14 | 96.11 | 1.13 | 2.76 |
| 7 (A) | 96.16 | 0.00 | 3.84 | 93.72 | 0.00 | 6.28 | 97.00 | 0.00 | 3.00 |
| 7 (B) | 91.64 | 0.00 | 8.36 | 90.14 | 0.00 | 9.86 | 89.95 | 6.94 | 3.11 |
| 14 (A) | 92.52 | 0.00 | 7.48 | 90.50 | 0.00 | 9.50 | 92.27 | 0.00 | 7.73 |
| 14 (B) | 89.67 | 0.00 | 10.33 | 93.10 | 0.00 | 6.90 | 93.30 | 0.00 | 6.70 |
| 21 (A) | 96.53 | 0.00 | 3.47 | 97.56 | 0.00 | 2.44 | 98.97 | 0.00 | 1.03 |
| 21 (B) | 97.72 | 0.00 | 2.28 | 90.97 | 0.00 | 9.03 | 93.29 | 0.00 | 6.71 |
| 30 (A) | 85.61 | 0.00 | 14.39 | 94.99 | 0.00 | 5.01 | 97.47 | 0.00 | 2.53 |
| 30 (B) | 95.04 | 0.00 | 4.96 | 95.59 | 1.27 | 3.14 | 95.12 | 1.26 | 3.62 |

No single component in the radioactivity designated as 'Others' was ≥10% of the applied radioactivity.

**Table 122 Hydrolytic half-lives and rate constants for IN-D2708 at 20 °C**

| pH | First order $DT_{50}$ (day) | Rate ($day^{-1}$) | Method of calculation |
|---|---|---|---|
| 4 | 386 (stable) | 0.0018 | linear simple first order |
| 7 | 981 (stable) | 0.0007 | linear simple first order |
| 9 | 8556 (stable) | 0.00008 | linear simple first order |

The half-life estimates are extrapolated well beyond the length of the study. While quantitatively unreliable, the estimates demonstrate that IN-D2708 is stable under the conditions and length of the test.

**Conclusions:**

IN-D2708 is stable to hydrolysis in sterile aqueous buffer solutions of pH 4, 7 and 9 for greater than 30 days at 20 ±1 °C.

The hydrolytic degradation study DuPont-4388, originally submitted under EU Rev8 Point IIA 7.2.1.1 and conducted with test material [$^{14}$C]IN-D2708, was conducted under guidelines U.S. EPA 161-1 (1982) and SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (OECD 111) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The substance IN-D2708 is considered stable at all pH's tested. The estimated hydrolysis half-lives at pH 4, 7, and 9 were more longer than the duration of the study, 386 days, 981 days, and 8556 days respectively.

**B.8.2.1.1/04**

| **Reference:** -- | **Report:** | Lee, D.Y. (2001); Hydrolysis of IN-N0079 in buffer solutions of pH 4, 7, and 9<br>**DuPont Report No.:** DuPont-4389, Revision No. 1<br>**Guidelines:** SETAC Europe (1995) |
|---|---|---|

1. Test material: [$^{14}$C]IN-N0079
   Lot/Batch #: HOTC 548
   Purity: Radiochemical purity - >98%

**Material and Methods:**

The hydrolytic stability of IN-N0079 was studied in aqueous solutions buffered at pH 4, 7, and 9. [ $^{14}$C] IN-N0079 (specific activity 45 µCi/mg, radiochemical purity >98%) was used as the test substance. Solutions containing IN-N0079 were prepared at nominal concentrations of 4.5 mg/L and were incubated for up to 30 days at 20 ±1 °C under sterile conditions in the dark.

Approximately 0.5 mL aliquots were removed from each test system with a pre-sterilised pipette under aseptic conditions into glass HPLC auto-injector vials for immediate analysis. Samples for all three pH values were taken immediately after treatment (day 0) and at designated sampling intervals thereafter. Sampling intervals for pH 4 and 7 were day 0 (i.e. immediately after treatment) and days 1, 3, 7, 14, 21 and 30. Sampling intervals for pH 9 were day 0 (i.e. immediately after treatment) and days 0.04 (1 hour), 0.08 (2 hour), 0.29 (7 hour), 0.5 (12 hour), 1, 2, 5, 8 and 12. Testing was done in duplicate. The aqueous samples were analysed by LSC and reversed phase HPLC with radiochemical detection. Degradation products were identified by co-chromatography with authentic standards.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Findings:**

Recovery of IN-N0079 ranged from 80% to 102% in individual replicates. Average values for duplicates ranged from 89% to 102%. Sterility tests conducted at 0 day and at the end of the study confirmed that all test systems remained sterile throughout the study.

IN-N0079 was hydrolytically stable at pH 4, with only low levels (typically <3%) of IN- D2708, IN-T2921 and others observed (Table ). At pH 7, up to 14% of the radioactivity was composed of IN-D2708, with IN-T2921 reaching 1% (Table ). At pH 9, IN-D2078 levels reached a maximum of 79%, with IN-T2921 reaching 19% by the end of the study (Table ).

At 20 ±1 °C, IN-N0079 was hydrolytically stable at pH 4 and no degradation products were observed above approximately 3%. At pH 9, IN-T2921 was formed readily from IN-N0079 and further degraded to IN-D2708; at pH 7 it was only formed to a lesser degree.

The DT50 values for the parent molecule are shown in Table . The half-life estimate at pH 4 was extrapolated well beyond the length of the study, demonstrating stability at this pH. DT50 values for degradation products of IN-N0079 were not calculated, as levels were generally increasing towards the end of the study.

**Table 123 Percent composition of radioactivity in pH 4 buffer at 20 °C**

| Day (replicate) | IN-N0079 | IN-T2921 | IN-D2708 | Unknowns | Total |
|---|---|---|---|---|---|
| 0 (A) | 97.37 | <LOD | <LOD | 2.63 | 100 |
| 0 (B) | 96.05 | 0.20 | <LOD | 3.75 | 100 |
| 1 (A) | 95.85 | 0.54 | 0.80 | 2.81 | 100 |
| 1 (B) | 97.24 | <LOD | 0.38 | 2.38 | 100 |
| 3 (A) | 98.88 | <LOD | <LOD | 1.12 | 100 |
| 3 (B) | 97.44 | <LOD | <LOD | 2.56 | 100 |
| 7 (A) | 96.51 | <LOD | 0.38 | 3.11 | 100 |
| 7 (B) | 96.45 | 0.36 | 0.28 | 2.91 | 100 |
| 14 (A) | 98.22 | <LOD | <LOD | 1.78 | 100 |
| 14 (B) | 97.61 | 0.27 | 0.47 | 1.65 | 100 |
| 21 (A) | 98.04 | <LOD | 0.40 | 1.56 | 100 |
| 21 (B) | 98.19 | 0.30 | 0.53 | 0.98 | 100 |
| 30 (A) | 99.16 | <LOD | <LOD | 0.84 | 100 |
| 30 (B) | 83.75* | <LOD | <LOD | 16.25 | 100 |

LOD = limit of detection (which was approximately 0.4% of the applied radioactivity for the radiochemical detection system used)

* This value was not used in the regression analysis for $DT_{50}$ determination.

**Table 124 Percent composition of radioactivity in pH 7 buffer at 20 °C**

| Day (replicate) | IN-N0079 | IN-T2921 | IN-D2708 | Unknowns | Total |
|---|---|---|---|---|---|
| 0 (A) | 98.00 | <LOD | 0.49 | 1.51 | 100 |
| 0 (B) | 98.18 | <LOD | <LOD | 1.82 | 100 |
| 1 (A) | 98.17 | <LOD | 0.67 | 1.16 | 100 |
| 1 (B) | 97.24 | <LOD | 0.38 | 2.38 | 100 |
| 3 (A) | 98.25 | <LOD | 1.16 | 0.59 | 100 |
| 3 (B) | 96.63 | 0.54 | <LOD | 2.83 | 100 |
| 7 (A) | 92.31 | 1.41 | <LOD | 6.28 | 100 |
| 7 (B) | 95.40 | <LOD | <LOD | 4.60 | 100 |
| 14 (A) | 93.27 | 0.59 | 0.79 | 5.35 | 100 |
| 14 (B) | 90.75 | 0.93 | 1.47 | 6.85 | 100 |
| 21 (A) | 87.95 | 0.37 | 10.75 | 0.93 | 100 |
| 21 (B) | 87.36 | 0.33 | 10.35 | 1.96 | 100 |
| 30 (A) | 84.88 | 0.32 | 14.17 | 0.63 | 100 |
| 30 (B) | 84.49 | 0.55 | 13.81 | 1.15 | 100 |

LOD = limit of detection (which was approximately 0.4% of the applied radioactivity for the radiochemical detection system used)

**Table 125 Percent composition of radioactivity in pH 9 buffer at 20 °C**

| Day (replicate) | IN-N0079 | IN-T2921 | IN-D2708 | Unknowns | Total |
|---|---|---|---|---|---|
| 0 (A) | 95.04 | <LOD | 0.81 | 4.15 | 100 |
| 0 (B) | 99.04 | <LOD | 0.56 | 0.40 | 100 |
| 0.042 (A) | 97.62 | <LOD | 1.18 | 1.20 | 100 |
| 0.042 (B) | 96.45 | <LOD | 1.71 | 1.84 | 100 |
| 0.083 (A) | 94.03 | 0.50 | 2.77 | 2.70 | 100 |
| 0.083 (B) | 94.23 | 0.79 | 4.45 | 0.53 | 100 |
| 0.29 (A) | 88.69 | 1.71 | 6.95 | 2.65 | 100 |
| 0.29 (B) | 89.24 | 1.91 | 7.65 | 1.20 | 100 |
| 0.50 (A) | 83.61 | 0.55 | 13.90 | 1.94 | 100 |
| 0.50 (B) | 84.28 | 2.94 | 10.56 | 2.22 | 100 |
| 1 (A) | 72.65 | 5.60 | 20.54 | 1.21 | 100 |
| 1 (B) | 73.64 | 4.59 | 20.65 | 1.12 | 100 |

| | | | | | |
|---|---|---|---|---|---|
| 2 (A) | 54.42 | 9.20 | 35.38 | 1.00 | 100 |
| 2 (B) | 54.70 | 8.85 | 35.03 | 1.42 | 100 |
| 5 (A) | 24.38 | 14.99 | 59.57 | 1.06 | 100 |
| 5 (B) | 24.87 | 15.07 | 58.50 | 1.56 | 100 |
| 8 (A) | 10.87 | 17.24 | 70.28 | 1.61 | 100 |
| 8 (B) | 11.00 | 16.80 | 70.37 | 1.83 | 100 |
| 12 (B) | 2.19 | 18.76 | 78.73 | 0.32 | 100 |
| 12 (B) | 6.50 | 13.07 | 75.67 | 4.76 | 100 |

LOD = limit of detection (which was approximately 0.4% of the applied radioactivity for the radiochemical detection system used)

**Table 126 Hydrolytic half-lives and rate constants for IN-N0079 at 20 °C**

| pH | First order $DT_{50}$ (day) | Rate ($day^{-1}$) | $r^2$ | Method of calculation |
|---|---|---|---|---|
| 4 | 990 (stable) | 0.0007 | 0.43 | linear simple first order |
| 7 | 136 | 0.0051 | 0.96 | linear simple first order |
| 9 | 2.6 | 0.2709 | 0.97 | linear simple first order |

The half-life estimate at pH 4 is extrapolated well beyond the length of the study. While quantitatively unreliable, the estimate demonstrates that IN-N0079 is stable under the conditions and length of the test at this pH.

**Conclusions:**

Hydrolysis is a major degradation mechanism for IN-N0079 in aqueous systems. The rate of degradation is pH-dependent and follows first-order kinetics. Alkaline conditions resulted in the most rapid degradation rate, with an average half-life of 3.0 days in the pH 9 test system. Less degradation was seen in pH 7 test systems, resulting in an average half-life of 136 days. IN-N0079 can be considered stable at pH 4, with a half-life estimated to be 990 days.

The hydrolytic degradation study DuPont-4389, Revision No. 1, originally submitted under EU Rev8 Point IIA 7.2.1.1 and conducted with test material [$^{14}$C]IN-N0079, was conducted under guideline SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (OECD 111) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The hydrolysis of IN-N0079 was pH dependent and followed first-order kinetics. The substance IN-N0079 degraded very rapidly at ph 9, with an average half-life of 2.6 days. Degradation was much slower at oH 7, with average half-life of 136 days. IN-N0079 can be considered stable at pH 4, with a calculated half-life of 990 days.

**B.8.2.1.1/05**

| **Reference:** -- | **Report:** | Van-Nguyen, A., Theilacker, W.M. (2001); Hydrolysis of IN-T2921 in buffer solutions of pH 4, 7, and 9<br>**DuPont Report No.:** DuPont-4390<br>**Guidelines:** U.S. EPA 161-1 (1982), OECD 111 (1981), SETAC Europe (1995) |
|---|---|---|

1. Test material: IN-T2921 technical metabolite
   Lot/Batch #: T2991-1
   Purity: 97.5%

**Material and Methods:**

The hydrolytic stability of IN-T2921 was studied in aqueous solutions buffered at pH 4, 7, and 9. Non-radiolabelled IN-T2921 standard (IN-T2921-1, 97.5% purity) was used as the test substance. Solutions containing IN-T2921 were prepared at nominal concentrations of 4 mg/L and were incubated for up to 30 days at 20 ±1 °C under sterile conditions in the dark.

Samples were taken immediately after the test substance was applied and at the following sampling schedules: day 0, 1, 3, 7, 10, 14, 21 and 30. On each sampling day, duplicate samples were removed from the system for analyses. If sample analysis by HPLC could not proceed immediately, the samples were stored at approximately -20 °C. The aqueous samples were analyzed by reversed phase HPLC with UV detection.

The analytical methods and calibration standard solutions used in the study were validated at each sampling point. At day of sampling or HPLC analysis, dose check or fortified solutions were prepared in selected pH buffer solutions at concentrations of 0.1 µg/mL (0.25 x LOQ), 1.0 µg/mL (~2.5 x LOQ) and 5.0 µg/mL (~10 x LOQ) and analysed by HPLC. Average recoveries of the test substance from the dose check were in the range of 70-120% with a relative standard deviation (RSD) of ≤20%. [The limit of quantitation (LOQ) is defined as the concentration where the peak signal is at least 10 times that of the background noise.]

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Findings:**

The solution concentration of non-radiolabelled IN-T2921 ranged from 85% to 101% of the initial concentration in individual replicates (re-calculated from the study data and based on an initial solution concentration of 4 mg/L). All concentration values with the exception of one replicate at pH 9, day 3 (3.40 mg/L, 85% of initial concentration) were 94% of initial values. Concentrations of IN-T2921 over time at each pH are presented in Table . No microbial growth was evident in the day-0 or day-30 samples. These tests confirmed that sterility was maintained for the duration of the hydrolysis study.

At 20 ±1 °C, IN-T2921 was hydrolytically stable at pH 4, 7 and 9. No degradation products were observed with the analytical method used. The DT50 values for IN-T2921 are shown in Table . Half-life estimates at each pH were extrapolated well beyond the length of the study, demonstrating stability.

**Table 127 Concentration (mg/L) of IN-T2921 in sterile buffers of pH 4, 7 and 9 at 20 °C**

| Sampling day | pH 4 | pH 7 | pH 9 |
|---|---|---|---|
| 0 | 3.79 | 3.94 | 3.94 |
| 0 | 3.88 | 3.98 | 3.99 |
| 1 | 3.90 | 3.96 | 4.00 |
| 1 | 3.94 | 3.98 | 3.99 |
| 3 | 3.95 | 3.97 | 3.40* |
| 3 | 3.92 | 4.01 | 4.02* |
| 7 | 3.86 | 3.96 | 3.93 |
| 7 | 3.90 | 3.97 | 3.93 |
| 10 | 3.90 | 3.94 | 3.92 |
| 10 | 3.91 | 3.99 | 3.90 |
| 14 | 3.89 | 3.95 | 3.86 |
| 14 | 3.90 | 3.92 | 3.97 |
| 21 | 3.90 | 3.94 | 3.83 |
| 21 | 3.89 | 3.85 | 3.80 |
| 30 | 3.87 | 3.94 | 3.74 |
| 30 | 3.89 | 3.92 | 3.74 |

Initial solution concentration was 4 mg/L in all samples.

* These values were considered suspect due to the large variation between replicates, and were not used in further data analysis.

**Table 128 Hydrolytic half-lives and rate constants for IN-T2921 at 20 °C**

| pH | Half-life (day) | Rate ($day^{-1}$) | Method of calculation |
|---|---|---|---|
| 4 | >1000 (stable) | <0.0001 | linear simple first order |
| 7 | >1000 (stable) | 0.000552 | linear simple first order |
| 9 | 337 (stable) | 0.00206 | linear simple first order |

The half-life estimate at each pH is extrapolated well beyond the length of the study. While quantitatively unreliable, these estimates demonstrate that IN-T2921 is stable under the conditions and length of the test.

**Conclusions:**

The hydrolysis of IN-T2921 at pH 4, 7 and 9 was studied for up to 30 days at 20 °C. IN-T2921 was stable in all cases. No degradation products were observed.

The hydrolytic degradation study DuPont-4390, originally submitted under EU Rev8 Point IIA 7.2.1.1 and conducted with test material IN-T2921 technical metabolite, was conducted under guidelines U.S. EPA 161-1 (1982), OECD 111 (1981), and SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (OECD 111) and it is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The substance IN-T2921 is considered stable at all pH's tested. The estimated hydrolysis half-lives at pH 4, 7, and 9 were more longer than the duration of the study, >1000 days, >1000 days, and 337 days respectively.

#### B.8.2.1.2 Direct photochemical degradation

In accordance with CA 2.8, new tests involving the photolysis of oxamyl at 25 °C using [1-$^{14}$C]-labeled oxamyl in sterile buffer solutions at pH 5 were submitted (DuPont-38008, Revision No. 1, summarized in the Oxamyl EU Renewal Dossier, Document M-CA, Section 2, DuPont-40929 EU). In this photolysis study conducted in accordance with the requirements of CA 2.8, the quantum yield of direct phototransformation was determined to be zero because oxamyl does not absorb light at or above 290 nm (DuPont-38008, Revision No. 1, summarized in the Oxamyl EU Renewal Dossier, Document M-CA, Section 2, DuPont-40929 EU).

**Study submitted to the EU for the first time in this submission**

**B.8.2.1.2/01**

| | | |
|---|---|---|
| **Reference:** **CA 7.2.1.2/01** | **Report:** | Hall, L. (2014); Aqueous photolysis of [$^{14}$C] DPX-D1410 (oxamyl)<br>**DuPont Report No.:** DuPont-38008 Revision No. 1<br>**Guidelines:** OECD 316, US EPA OPPTS 835.2240<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc., Columbia, Missouri, USA<br>**Testing Facility Report No.:** 81380<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

The aqueous phototransformation of radiolabeled [1-$^{14}$C] DPX-D1410 ([1-$^{14}$C]oxamyl) was studied at 25°C in sterile aqueous acetate buffer solution at pH 5 at an initial concentration of 1.10 mg/L under artificial irradiation (a xenon lamp and a Q Sun Daylight Q filter with a nominal 295 nm UV cut-off were used) for 10 days. Material balance was 100 ± 1.3% (range = 97.3 to 102.3%) and 99.9 ± 0.5% (range = 99.4 to 100.9%) of the applied amount in the irradiated and dark (non-irradiated) samples, respectively.

In the irradiated samples, the parent compound decreased from 96.7% of the applied amount at Day 0 to 14.8% of the applied amount at the end of irradiation. The only major transformation product (>5%) detected was IN-N0079, which comprised a maximum concentration of 67.6% of the applied amount on the tenth day of the test. IN-A2213 was observed as a minor transformation product and comprised a maximum of 1.8% of the applied amount on the seventh day. Volatile radioactivity was not observed in the current study.

The first-order half-lives for oxamyl in the irradiated and dark samples were 3.5 days and 530 days, respectively.

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabeled test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: 1841000/ 111754521
   Radiochemical purity: 96.0%
   Specific activity: 75.6 μCi/mg
   Description: Solid
   Stability of test compound: Not determined

2. Buffers:

   0.01 M buffer solutions in HPLC grade water were prepared at pH 5 using acetic acid and sodium hydroxide.

B. STUDY DESIGN

1. Experimental conditions

   The aqueous phototransformation of radiolabeled oxamyl was studied at 25°C in pH 5 sterile aqueous acetate buffer solution at an initial concentration of 1.10 mg a.s./L. The study was conducted under artificial irradiation (a xenon lamp and a Q Sun Daylight Q filter with a nominal 295 nm UV cut-off were used) for 10 days. Acetonitrile (<0.2%) was used as a co-solvent. Additional samples were used as dark controls that were maintained at 25°C. Test vessels were not connected to traps for the collection of $CO_2$ and organic volatiles.

2. Description of analytical procedures

   Samples were analysed at 0, 1, 2, 3, 4, 7, and 10 days after treatment. The aqueous samples were analysed by direct injection onto HPLC with on-line radiodetection. Identification of the transformation products was performed by HPLC chromatography and GC/MS. The LOD for oxamyl and related metabolites was 0.1% of the applied radioactivity (ca. 0.001 μg/mL). The LOQ for oxamyl and related metabolites was 0.3% of the applied radioactivity (ca. 0.003 μg/mL).

## II. RESULTS AND DISCUSSION

A. MASS BALANCE

Material balance was 100 ± 1.3% (range = 97.3 to 102.3%) and 99.9 ± 0.5% (range = 99.4 to 100.9%) of the applied amount in the irradiated and dark (non-irradiated) samples, respectively.

B. FINDINGS

In the irradiated samples, the parent compound decreased from 96.7% of the applied amount at Day 0 to 14.8% of the applied at the end of irradiation (Table ). The only major transformation product (>5%) detected was IN-N0079, which comprised a maximum concentration of 67.6% of the applied amount on the tenth day of the test. IN-A2213 was observed as a minor transformation product and comprised a maximum of 1.8% of the applied amount on the seventh day.

In the dark samples, the parent compound decreased from 96.1% of the applied amount at Day 0 to 94.6% of the applied at the end of the experimental phase (Table ). No major transformation products (>5% AR) were detected. A minor transformation product was tentatively identified as IN-N0079, which formed a

maximum concentration of 1.6% of the applied amount. Volatile radioactivity was not observed in the current study.

**Table 129 Phototransformation of oxamyl, expressed as percentage of applied radioactivity (mean ± s.d.) under aqueous photolytic conditions**

| Compound | | Sampling times [days] | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **2** | **3** | **4** | **7** | **10** |
| Oxamyl | irradiated | 96.7 ± 0.0 | 79.2 ± 1.6 | 64.9 ± 1.4 | 53.5 ± 4.9 | 43.7 ± 2.0 | 24.6 ± 0.4 | 14.8 ± 0.8 |
| | dark | 96.1 ± 0.0 | 96.2 ± 0.6 | 96.0 ± 0.1 | 94.8 ± 0.8 | 94.9 ± 0.5 | 95.9 ± 0.3 | 94.6 ± 0.8 |
| IN-A2213 | irradiated | 0.0 ± 0.0 | 1.0 ± 0.2 | 1.2 ± 0.4 | 1.2 ± 0.2 | 1.3 ± 0.1 | 1.8 ± 0.5 | 1.5 ± 0.1 |
| | dark | 0.2 ± 0.1 | 0.3 ± 0.0 | 0.4 ± 0.1 | 0.5 ± 0.1 | 0.6 ± 0.0 | 0.8 ± 0.0 | 0.7 ± 0.1 |
| IN-N0079 | irradiated | 1.3 ± 0.0 | 14.8 ± 1.8 | 26.9 ± 1.3 | 34.2 ± 3.4 | 44.2 ± 0.8 | 62.3 ± 0.2 | 67.6 ± 0.8 |
| | dark | 0.9 ± 0.7 | 1.0 ± 0.9 | 1.6 ± 0.2 | 1.2 ± 0.4 | 1.3 ± 0.1 | 0.7 ± 0.3 | 0.7 ± 1.0 |
| Unidentified radioactivity[a] | irradiated | 2.0 ± 0.1 | 5.7 ± 0.2 | 7.9 ± 0.2 | 11.0 ± 0.0 | 10.2 ± 0.9 | 12.4 ± 0.6 | 13.8 ± 0.9 |
| | dark | 2.8 ± 0.7 | 2.7 ± 0.9 | 2.1 ± 0.1 | 2.9 ± 0.1 | 2.7 ± 0.6 | 3.2 ± 0.8 | 3.5 ± 1.6 |
| $CO_2$ | irradiated | NA[b] | NA | NA | NA | NA | NA | NA |
| | dark | NA | NA | NA | NA | NA | NA | NA |
| Total volatile organic | irradiated | NA | NA | NA | NA | NA | NA | NA |
| | dark | NA | NA | NA | NA | NA | NA | NA |
| Total % recovery | irradiated | 100.0 ± 0.2 | 100.7 ± 0.2 | 100.9 ± 0.2 | 99.9 ± 1.8 | 99.5 ± 0.4 | 101.1 ± 1.6 | 97.8 ± 0.7 |
| | dark | 100.0 ± 0.1 | 100.1 ± 0.3 | 100.1 ± 0.1 | 99.4 ± 0.0 | 99.5 ± 0.0 | 100.6 ± 0.4 | 99.4 ± 0.0 |

[a] No single component comprised >4.1% or 0.8% of applied in irradiated and dark samples, respectively.

[b] NA = Not applicable since no volatile radioactivity was observed in the current study.

On the basis of linear first order kinetic reactions, the decomposition $DT_{50}$ of oxamyl was calculated (Table ). The structures of the identified photoproducts, their common names, and their maximum relative distribution are shown in Figure and Table .

**Table 130 Photolytic $DT_{50}$ values and rate constants for oxamyl**

| Sample | Analyte | Half-life (days) | k ($day^{-1}$) | Half-life (day) (natural sunlight)[a] | Method of calculation ($r^2$) | Quantum yield |
|---|---|---|---|---|---|---|
| Irradiated | Oxamyl | 3.5 | 0.19790 | 4.1 | First-order (0.996) | NC[b] |
| Non-irradiated | | 530 | 0.00131 | | First-order (0.310) | |

[a] Half-life was estimated for Phoenix, AZ (33.3 °N) based on the xenon lamp intensity.

[b] NC—Quantum yield was not calculated since oxamyl does not have any significant absorbance at wavelengths >290 nm.

**Figure 8 Proposed aqueous photolytic degradation pathways of oxamyl**

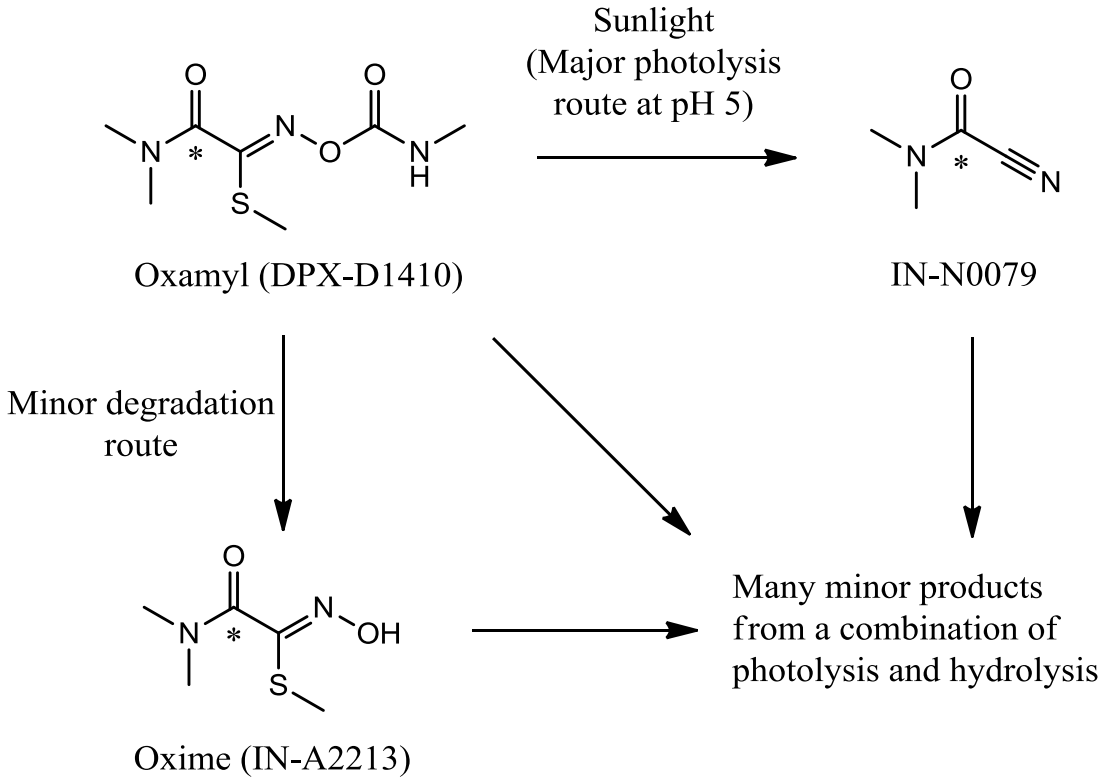



**Table 131 Characterisation of the photolytic degradation products of oxamyl identified**

| Photoproduct | Maximum product (% applied) | Time of maximum product (days) | mg/kg |
|---|---|---|---|
| IN-N0079 | 67.6 | 10 | 0.74 |
| IN-A2213 | 1.8 | 7 | 0.02 |

## III. CONCLUSION

Oxamyl was degraded by photolysis to a significant extent following exposure to artificial light with environmental half-lives estimated as 4.1, 5.8, 6.3, 7.9, or 8.7 mid-summer days in Phoenix, Arizona (USA, 33.3 °N); Edmonton, Alberta (Canada, 53.3 °N); Athens, Greece (EU, 38.0 °N); London, Great Britain (EU, 51.3 °N); and Tokyo (Japan, 35.1 °N), respectively. Photolytic degradation leads primarily to IN-N0079, which comprised a maximum of 67.6% of the applied amount at study termination. Several minor degradates were observed in irradiated samples, but none of these photodegradates comprised >4.1% of applied amount at study termination (Table ). The study demonstrated that oxamyl will degrade rapidly in aquatic systems under sunlight to IN-N0079.

(Hall, L. 2014)

The direct photolytic degradation study DuPont-38008 Revision No. 1, submitted for the first time in this submission and conducted with test material [14C]-oxamyl, was conducted under guidelines OECD 316 (2008), and US EPA OPPTS 835.2240 (2008). A review of this study indicates that it fully meets the current guideline (OECD 316) and it is relied upon.

**RMS comments and conclusion**

The net photodegradation of oxamyl was rapid with a $DT_{50}$ of 3.5 experimental days. Therefore, photolysis is expected to be a significant route for the dissipation of oxamyl residues from aquatic environments.

**B.8.2.1.2/02**

| **Reference:** -- | **Report:** | Tuffy, C. (2000a); Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-A2213<br>**DuPont Report No.:** DuPont-4514<br>**Guidelines:** OECD 101 (1981), OPPTS 830.7050 |
|---|---|---|

1. Test material: IN-A2213 technical metabolite
   Lot/Batch #: A2213-11
   Purity: 100%

**Materials and Methods:**

The UV-visible absorption of non-radiolabelled IN-A2213 (100% purity) was determined in aqueous solutions of pH <2, pH 7 and pH >10 at 25 ±1 °C.

Solution concentrations of 25 µg/mL and 500 µg/mL were used (with the higher concentration solutions being used to determine the absorbance at 290 nm). Prior to sample analysis, the solutions and samples were equilibrated to 25 ±1 °C in a water bath. The temperature of the water bath was checked during each set of scans. The pH of each test solution was also measured using pH paper. Spectra were obtained immediately after the test solutions were prepared.

Absorbance measurements were made in duplicate using a spectrophotometer equipped with a holographic diffraction grating monochromator, programmed optical filters, and a rotating chopper dividing the sample and reference beams focused at the sample and reference cells. Deuterium and tungsten lamps were used as the light sources and a side-window photo- multiplier was the detector.

Molar absorptivities (εL/mol·cm) were calculated for the absorption maxima and at 290 nm using the following equation:

$$\varepsilon_i = A_i/C \cdot d$$

where $\varepsilon_i$ = the molar absorption coefficient at wavelength i, $A_i$ = the sample absorbance at wavelength i, C = the molar concentration of the test substance solution and d = the solution cell path length (1.000 cm).

**Findings:**

The results are summarised in Table . The spectra obtained are different, reflecting changes in electronic configuration at the different pH values.

The UV-Vis spectrum of the test substance in pH <2 solution (25 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 212 nm was calculated to be 9310 ±37 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 45.2 L/mol·cm.

The UV-Vis spectrum of the test substance in pH 7 solution (25 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of approximately 211.5 nm was calculated to be 9450 ±45 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 56.9 L/mol·cm.

The UV-Vis spectrum of the test substance in pH >10 solution (25 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 218 nm was calculated to be 8490 ±129 L/mol·cm. The molar extinction coefficient at 270 nm (a shoulder) was calculated to be 2330 ±37 L/mol·cm. Unlike the other pH values tested, at pH >10 there was measurable absorbance at the wavelength of 290 nm for the 25 µg/mL solution concentration (tail-end of shoulder at 270 nm). The molar extinction coefficient at 290 nm (25 µg/mL) was calculated to be 1110 ±28 L/mol·cm. [Visual inspection of the absorbance spectra at this pH (noted as pH 13 in the report) suggests that IN- A2213 may have been structurally changed in this extremely alkaline solution altering the absorbance characteristics.] The absorbance was approximately zero from 305-750 nm.

Representative spectra of matrix blanks for each pH value tested indicated that there was no significant interference in absorbance over the wavelength range.

**Table 132 Absorption characteristics of IN-A2213**

| pH | Concentration (µg/mL) | Absorbance maximum wavelength (nm) | Absorbance | | Average molar absorptivity, ε (L/mol·cm) |
|---|---|---|---|---|---|
| | | | Scan 1 | Scan 2 | |
| <2 | 25.0 | 212 | 1.4295 | 1.4374 | 9310 ± 37 |
| <2 | 500 | 290* | 0.1383 | 0.1398 | 45.2 ± 0.4 |
| 7 | 25.0 | 211-212 | 1.4603 | 1.4506 | 9450 ± 45 |
| 7 | 500 | 290* | 0.1754 | 0.1749 | 56.9 ± 0.1 |
| >10 | 25.0 | 218 | 1.3214 | 1.2934 | 8490 ± 129 |
| >10 | 25.0 | 270* | 0.3634 | 0.3555 | 2330 ± 37 |
| >10 | 25.0 | 290* | 0.1738 | 0.1678 | 1110 ± 28 |

The average molar absorptivity is rounded to three significant figures and is reported with ± one standard deviation.
* Not a true absorption maximum.

**Conclusions:**

UV-Vis spectra and molar absorptivities were measured for IN-A2213 at pH <2, pH 7 and pH >10. IN-A2213 did not have any absorption maxima above 290 nm at any of the pH values tested and so molar absorptivities were measured at 290 nm. The molar absorptivity of IN- A2213 at 290 nm was >10 L/mol·cm at each pH tested.

The direct photochemical degradation study DuPont-4514, originally submitted under EU Rev8 Point IIA 7.2.1.2.2, 7.2.1.2.1 and conducted with test material IN-A2213 technical metabolite, was conducted under guidelines OECD 101 (1981) and OPPTS 830.7050. A review of this study indicates that it fully meets the current guideline (OECD 101) and should be regarded as supplemental photolysis information and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The molar absorptivity of IN- A2213 at 290 nm was >10 L/mol·cm at each pH tested

**B.8.2.1.2/03**

| **Reference:** -- | **Report:** | Tuffy, C. (2000b); Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-D2708<br>**DuPont Report No.:** DuPont-4515<br>**Guidelines:** OECD 101 (1981), OPPTS 830.7050 |
|---|---|---|

1. Test material: IN-D2708 technical metabolite
   Lot/Batch #: D2708-6
   Purity: 98.1%

**Materials and Methods:**

The UV-visible absorption of non-radiolabelled IN-D2708 (98.1% purity) was determined in aqueous solutions of pH <2, pH 7 and pH >10 at 25 ±1 °C.

Solution concentrations of 20 µg/mL (pH <2 and pH 7), 60 µg/mL (pH >10) and 500 µg/mL were used (with the 500 µg/mL solutions being used to determine the absorbance at 290 nm for all the pH values tested). The experimental procedure was the same as that previously described in this section for IN-A2213.

**Findings:**

The results are summarised in Table . The spectra obtained are different, reflecting variable IN-D2708 decomposition in solution and/or changes in electronic configuration at the different pH values.

The UV-Vis spectrum of the test substance in pH <2 solution (20 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 202 nm was calculated to be 5310 ±38 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 2.3 L/mol·cm.

The UV-Vis spectrum of the test substance in pH 7 solution (20 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the wavelength of maximum absorption (200 nm) was calculated to be 7300 ±14 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 1.0 L/mol·cm.

The UV-Vis spectrum of the test substance in pH >10 solution (60 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 217 nm was calculated to be 2040 ±45 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 0.4 L/mol·cm.

Representative spectra of matrix blanks for each pH value tested indicated that there was no significant interference in absorbance over the wavelength range.

**Table 133 Absorption characteristics of IN-D2708**

| pH | Concentration (µg/mL) | Absorbance maximum wavelength (nm) | Absorbance | | Average molar absorptivity, ε (L/mol·cm) |
|---|---|---|---|---|---|
| | | | Scan 1 | Scan 2 | |
| <2 | 20.0 | 202 | 0.9119 | 0.9027 | 5310 ± 38 |
| <2 | 500 | 290* | 0.0096 | 0.0101 | 2.31 ± 0.08 |
| 7 | 20.0 | 200* | 1.2492 | 1.2458 | 7300 ± 14 |
| 7 | 500 | 290* | 0.0045 | 0.0041 | 1.01 ± 0.06 |
| >10 | 60.0 | 217 | 1.0615 | 1.0286 | 2040 ± 45 |
| >10 | 500.0 | 290* | 0.0013 | 0.0019 | 0.375 ± 0.100 |

The average molar absorptivity is rounded to three significant figures and is reported with ± one standard deviation.
* Not a true absorption maximum.

**Conclusions:**

UV-Vis spectra and molar absorptivities were measured for IN-D2708 at pH <2, pH 7 and pH >10. IN-D2708 did not have any absorption maxima above 290 nm at any of the pH values tested and so molar absorptivities were measured at 290 nm. The molar absorptivity of IN- D2708 at 290 nm was <10 L/mol·cm at each pH tested.

The direct photochemical degradation study DuPont-4515, originally submitted under EU Rev8 Point IIA 7.2.1.2.2, 7.2.1.2.1 and conducted with test material IN-D2708 technical metabolite, was conducted under guidelines OECD 101 (1981) and OPPTS 830.7050. A review of this study indicates that it fully meets the current guideline (OECD 101) and should be regarded as supplemental photolysis information. This study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The molar absorptivity of IN- D2708 at 290 nm was <10 L/mol·cm at each pH tested

**B.8.2.1.2/04**

| **Reference:** -- | **Report:** | Tuffy, C. (2000c); Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-N0079<br>**DuPont Report No.:** DuPont-4516 |
|---|---|---|

| | | |
|---|---|---|
| | | **Guidelines:** OECD 101 (1981), OPPTS 830.7050 |

1. Test material: IN-N0079 technical metabolite
   Lot/Batch #: N0079-8
   Purity: 99.5%

**Materials and Methods:**

The UV-visible absorption of non-radiolabelled IN-N0079 (99.5% purity) was determined in aqueous solutions of pH <2, pH 7 and pH >10 at 25 ±1 °C.

Solution concentrations of 24.9 µg/mL (pH <2), 25.0 µg/mL (pH 7), 150 µg/mL (pH >10) and 500 µg/mL were used (with the 500 µg/mL solutions being used to determine the absorbance at 290 nm for all the pH values tested). The experimental procedure was the same as that previously described in this section.

**Findings:**

The results are summarised in Table . The spectra obtained are different (acidic and neutral are similar), reflecting variable IN-N0079 decomposition in solution and/or changes in electronic configuration at the different pH values.

The UV-Vis spectrum of the test substance in pH <2 solution (24.9 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 229 nm was calculated to be 5700 ±1 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 3.3 L/mol·cm.

The UV-Vis spectrum of the test substance in pH 7 solution (25.0 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 229 nm was calculated to be 5820 ±42 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 5.3 L/mol·cm.

The UV-Vis spectrum of the test substance in pH >10 solution (150 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of approximately 218 nm was calculated to be 843 ±83 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (500 µg/mL) was calculated to be 0.4 L/mol·cm.

Representative spectra of matrix blanks for each pH value tested indicated that there was no significant interference in absorbance over the wavelength range.

**Table 134 Absorption characteristics of IN-N0079**

| pH | Concentration (µg/mL) | Absorbance maximum wavelength (nm) | Absorbance | | Average molar absorptivity, ε (L/mol·cm) |
|---|---|---|---|---|---|
| | | | Scan 1 | Scan2 | |
| <2 | 24.9 | 229 | 1.4472 | 1.4470 | 5700 ± 1 |
| <2 | 500 | 290* | 0.0166 | 0.0169 | 3.28 ± 0.04 |
| 7 | 25.0 | 229 | 1.4926 | 1.4775 | 5820 ± 42 |
| 7 | 500 | 290* | 0.0266 | 0.0274 | 5.30 ± 0.11 |
| >10 | 150 | 218 | 1.3805 | 1.1994 | 843 ± 83 |
| >10 | 500 | 290* | 0.0020 | 0.0019 | 0.383 ± 0.013 |

The average molar absorptivity is rounded to three significant figures and is reported with ± one standard deviation.
* Not a true absorption maximum.

**Conclusions:**

UV-Vis spectra and molar absorptivities were measured for IN-N0079 at pH <2, pH 7 and pH >10. IN-N0079 did not have any absorption maxima above 290 nm at any of the pH values tested and so molar absorptivities were measured at 290 nm. The molar absorptivity of IN- N0079 at 290 nm was <10 L/mol·cm at each pH tested.

The direct photochemical degradation study DuPont-4516, originally submitted under EU Rev8 Point IIA 7.2.1.2.2, 7.2.1.2.1 and conducted with test material IN-N0079 technical metabolite, was conducted under guidelines OECD 101 (1981) and OPPTS 830.7050. A review of this study indicates that it fully meets the current guideline (OECD 101) and should be regarded as supplemental photolysis information.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The molar absorptivity of IN- N0079 at 290 nm was <10 L/mol·cm at each pH tested

**B.8.2.1.2/05**

| **Reference:** -- | **Report:** | Tuffy, C. (2000d); Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-T2921<br>**DuPont Report No.:** DuPont-4517<br>**Guidelines:** OECD 101 (1981), OPPTS 830.7050 |
|---|---|---|

1. Test material: IN-T2921 technical metabolite
   Lot/Batch #: T2921-1
   Purity: 97.5%

**Materials and Methods:**

The UV-visible absorption of non-radiolabelled IN-T2921 (97.5% purity) was determined in aqueous solutions of pH <2, pH 7 and pH >10 at 25 ±1 °C.

Solution concentrations of 25.8 µg/mL (pH <2), 15.5 µg/mL (pH 7), 61.8 µg/mL (pH >10) and 800 µg/mL were used (with the 800 µg/mL solutions being used to determine the absorbance at 290 nm for all the pH values tested). The experimental procedure was the same as that previously described in this section.

**Findings:**

The results are summarised in Table . The spectra obtained are different, reflecting changes in electronic configuration at the different pH values.

The UV-Vis spectrum of the test substance in pH <2 solution (25.8 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of 203 nm was calculated to be 6380 ±49 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (800 µg/mL) was calculated to be 6.3 L/mol·cm.

The UV-Vis spectrum of the test substance in pH 7 solution (15.5 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the wavelength of maximum absorption (200 nm) was calculated to be 8050 ±13 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm (800 µg/mL) was calculated to be 6.0 L/mol·cm.

The UV-Vis spectrum of the test substance in pH >10 solution (61.8 µg/mL) showed no absorption maxima beyond 290 nm. The molar extinction coefficient at the absorption maximum of approximately 218 nm was calculated to be 2430 ±172 L/mol·cm. The absorbance was approximately zero from 290-750 nm. The molar extinction coefficient at 290 nm was calculated to be 2.1 L/mol·cm.

Representative spectra of matrix blanks for each pH value tested indicated that there was no significant interference in absorbance over the wavelength range.

**Table 135 Absorption characteristics of IN-T2921**

| pH | Concentration (µg/mL) | Absorbance maximum wavelength (nm) | Absorbance | | Average molar absorptivity, ε (L/mol·cm) |
|---|---|---|---|---|---|
| | | | Scan 1 | Scan 2 | |

| | | | | | |
|---|---|---|---|---|---|
| <2 | 25.8 | 203 | 1.4081 | 1.4236 | 6380 ± 49 |
| <2 | 800 | 290* | 0.0432 | 0.0435 | 6.29 ± 0.03 |
| 7 | 15.5 | 200* | 1.0699 | 1.0723 | 8050 ± 13 |
| 7 | 800 | 290* | 0.0418 | 0.0413 | 6.03 ± 0.06 |
| >10 | 61.8 | 218 | 1.3551 | 1.2255 | 2430 ± 172 |
| >10 | 800.0 | 290* | 0.0144 | 0.0146 | 2.11 ± 0.02 |

The average molar absorptivity is rounded to three significant figures and is reported with ± one standard deviation.
* Not a true absorption maximum.

**Conclusions:**

UV-Vis spectra and molar absorptivities were measured for IN-T2921 at pH <2, pH 7 and pH >10. IN-T2921 did not have any absorption maxima above 290 nm at any of the pH values tested and so molar absorptivities were measured at 290 nm. The molar absorptivity of IN- T2921 at 290 nm was <10 L/mol·cm at each pH tested.

The direct photochemical degradation study DuPont-4517, originally submitted under EU Rev8 Point IIA 7.2.1.2.2, 7.2.1.2.1 and conducted with test material IN-T2921 technical metabolite, was conducted under guidelines OECD 101 (1981) and OPPTS 830.7050. A review of this study indicates that it fully meets the current guideline (OECD 101) and should be regarded as supplemental photolysis information. This study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The molar absorptivity of IN- T2921 at 290 nm was <10 L/mol·cm at each pH tested

#### B.8.2.1.3 Indirect photochemical degradation

No study on the indirect photolysis of oxamyl was performed. The photodegradation of oxamyl was well defined in the direct photolysis study (see Point B.8.2.1.2 in this document). There is no evidence to suggest that indirect photolysis of oxamyl would be different from that observed in the direct study.

### B.8.2.2 Route and rate of biological degradation in aquatic systems

#### B.8.2.2.1 Ready biodegradability

**Ready biodegradability**

**B.8.2.2.1/01**

| | | |
|---|---|---|
| **Reference:**<br>-- | **Report:** | Barnes, S.P. (2001); Oxamyl (DPX-D1410): Assessment of ready biodegradability by modified Sturm test<br>**DuPont Report No.:** DuPont-6650<br>**Guidelines:** EEC Method C.4-C. (1992), OECD 301 B (1992) |

1. Test material: Oxamyl technical
   Lot/Batch #: D1410-367
   Purity: 100%

**Material and Methods:**

Oxamyl was added to two vessels containing mineral salts medium inoculated with activated sludge, which contained 30 mg solids/L. The test concentration of oxamyl was 26.1 mg/L, corresponding to 10 mg carbon/L. Another vessel contained inoculated mineral salts medium plus sodium benzoate (10 mg carbon/L), which was

used as a positive control. An additional mixture, containing sodium benzoate (10 mg carbon/L) and oxamyl (10 mg carbon/L), was established in order to assess the potential inhibitory effects of the test substance on the microbial inoculum. Two control vessels, containing inoculated mineral salts medium alone, were also established.

The test, control and reference mixtures were incubated aerobically for 29 days at temperatures nominally in the range 20-24 °C, using air that had been treated to remove carbon dioxide. The cultures were acidified on day 28 in order to drive off dissolved inorganic carbon, and final titrations were performed on day 29. Carbon dioxide produced from test and control samples was precipitated in 0.025N barium hydroxide solution and the amount produced determined by acid titration of residual barium hydroxide. The amount of carbon dioxide was measured to assess the percentage of biodegradation.

**Findings:**

The results are summarized in Table , which shows blank-corrected cumulative carbon dioxide production for the reference and test mixtures as a percentage of the theoretical maximum amount of carbon dioxide. The theoretical maximum amount of carbon dioxide that can be generated by a 3.0 L mixture containing 10 mg carbon/L is 110 mg.

*Theoretical maximum carbon dioxide*
*= (molecular weight of carbon dioxide (44)/ atomic weight of carbon (12))*
*x 3.0 L x 10 mg carbon/L*

Oxamyl was not inhibitory to the activity of the microbial inoculum, as confirmed by the results for sodium benzoate biodegradation. In the absence of oxamyl, cumulative carbon dioxide production due to sodium benzoate biodegradation reached 59% of the theoretical maximum by day 6 and 90% by day 29, whereas cumulative carbon dioxide production from the mixture of oxamyl and sodium benzoate reached 61% of the theoretical maximum by day 6. Cumulative levels of carbon dioxide production in the control vessels after 29 days (80.9 and 75.4 mg CO2) were typical for this type of test and inoculum source and were within the acceptable range for this assay system (recommended maximum = 120 mg CO2 for a three- litre culture). These results confirm that the inoculum was viable and that the test was valid.

Mean cumulative carbon dioxide production by the oxamyl test mixtures reached 11% of the theoretical maximum by day 23 and 19% by the end of the test on day 29. A biodegradation plateau was not considered to have been achieved by the end of the test.

Substances are considered to be readily biodegradable in the CO2 Evolution Test if CO2 production is equal to or greater than 60% of the theoretical maximum value within 10 days of achieving the 10% level. Therefore, oxamyl cannot be considered to be readily biodegradable.

However, because of the stringency of this type of test, substances that fail to show the required rate of biodegradation are not necessarily poorly degradable under more relevant environmental conditions. Biodegradability may progress at a higher rate where the substance is exposed to a larger and more diverse microbial population.

**Table 136 Blank-corrected cumulative $CO_2$ production for the reference and test mixtures**

| Day | Sodium Benzoate (10 mg C/L) | | Sodium Benzoate in Oxamyl + Sodium Benzoate mixture (Both at 10 mg C/L) | | Oxamyl (10 mg C/L) | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Culture 1 | | Culture 2 | | Mean |
| | $CO_2$ (mg) | % $TCO_2$ | $CO_2$ (mg) | % $TCO_2$ | $CO_2$ (mg) | % $TCO_2$ | $CO_2$ (mg) | % $TCO_2$ | % $TCO_2$ |
| 2 | 24.2 | 22 | 22.6 | 20 | 0.0 | 0 | 0.0 | 0 | 0 |
| 4 | 47.6 | 43 | 45.9 | 42 | 0.0 | 0 | 0.0 | 0 | 0 |
| 6 | 65.5 | 59 | 67.1 | 61 | 0.0 | 0 | 0.0 | 0 | 0 |
| 8 | 75.4 | 68 | - | - | 0.5 | 0 | 1.6 | 1 | 1 |
| 10 | 82.5 | 75 | - | - | 1.7 | 1 | 2.8 | 2 | 2 |
| 13 | 86.9 | 79 | - | - | 2.8 | 2 | 3.9 | 3 | 3 |
| 17 | 93.2 | 85 | - | - | 4.7 | 4 | 6.3 | 6 | 5 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **23** | **96.5** | **88** | **-** | **-** | **11.3** | **10** | **12.4** | **11** | **11** |
| **28** | **98.5** | **89** | **-** | **-** | **17.6** | **16** | **18.7** | **17** | **16** |
| **29** | **99.0** | **90** | **-** | **-** | **19.8** | **18** | **21.5** | **19** | **19** |

$TCO_2$ = theoretical maximum amount of carbon dioxide (110 mg)

**Conclusions:**

Although oxamyl appreciably degraded at 26 mg/L (10 mg carbon/L), it did not attain >60% biodegradation within 10 days of reaching 10% biodegradation. Therefore, oxamyl cannot be classified as readily biodegradable within the strict terms of the method used.

The "ready biodegradability" study DuPont-6650, originally submitted under EU Rev8 Point IIA 7.2.1.3.1 and conducted with test material oxamyl technical, was conducted under guidelines EEC Method C.4-C (1992) and OECD 301 B (1992). A review of this study indicates that it fully meets the current guideline (OECD 301) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Oxamyl cannot be classified as readily biodegradable within the strict terms of the method used.

**Aerobic biodegradation in aquatic systems**

Tests involving the aerobic biodegradation of oxamyl in aquatic systems were submitted under Point B.8.2.2.2 and Point B.8.2.2.3 in this document (DuPont-40441 and AMR 3143-94).

**Anaerobic biodegradation in aquatic systems**

Tests involving the anaerobic biodegradation of oxamyl in aquatic systems were submitted under Point B.8.2.2.3 in this document (DuPont-34157).

### B.8.2.2.2 Aerobic mineralisation in surface water

**Study submitted to the EU for the first time in this submission.**

**B.8.2.2.2/01**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.2.2.2/01** | **Report:** | Allan, J. (2015); Oxamyl (DPX-D1410): Aerobic mineralization in surface water<br>**DuPont Report No.:** DuPont-40441<br>**Guidelines:** OECD 309 (April 2004), OPPTS 835.3190 (October 2008)<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** 81140<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary**:

The aerobic mineralization of [1-$^{14}$C]oxamyl was studied in one surface water system from North America under pelagic conditions at 20 ± 2 ºC in the dark. The pH, biological oxygen demand, and total suspended solids were 7.2, 28.1 mg-$O_2$/L, and 12 ppm, respectively.

A total of 14 bioreactors (1-liter capacity) containing 700 mL of surface water were used. To 12 bioreactors, [1-$^{14}$C]oxamyl was applied at nominal rates of 20 µg a.s./L and 200 µg a.s./L. Four bioreactors, one for each replicate for each concentration, were used for mineralization testing at zero time, 1, 2, 3, 5, 7, 14, 28, 46, and 60 days after dosing. To confirm microbial viability of the surface water, two bioreactors were prepared with a reference compound (sodium benzoate) at 500 µg/L and were analysed in parallel for mineralization. Eight bioreactors, one for each replicate, concentration, and two intervals were prepared and analysed for material balance at study initiation and termination.

Results indicated that oxamyl undergoes limited mineralization in natural surface water (<7% in phase) over the study duration. Mineralization rate constants were calculated to be 552 and 671 day $^{-1}$ for the 20 µg/L and 200 µg/L dose rates, respectively. The reference compound (sodium benzoate) was mineralized up to a mean of 95.6% at termination, confirming the viability of the system.

The mean material balance at study initiation of the low and high dose rates in the mass balance bioreactors were 98.6% and 99.3% of the dosed radioactivity, respectively. At termination (Day 60), the mean material balance of the low and high dose rates were 97.4% and 98.8% of the dosed radioactivity, respectively.

The amount of [1-$^{14}$C]oxamyl in the surface water decreased from 93.9%AR at Day0 to 0.0%AR at the end of 60 days of incubation. The major intact transformation product detected was the oxime (IN-A2213), which reached a maximum concentration at Day 46 of 85.5% AR, decreasing slightly to 85.2% AR at termination (Day 60).

## I. MATERIALS AND METHODS

### A. MATERIALS

1. Radiolabeled test material: [1-$^{14}$C]oxamyl
   Radiolabeled Lot/Batch #: 1841000
   Radiochemical purity: 99.7%
   Specific activity: 75.6 µCi/mg (16.6 mCi/mol)
   Non-radiolabeled Lot#: D101163-00005.04
   Stability of test compound: The test material was stable in the aqueous phase under the test conditions

2. Surface Water:

   The study was conducted using surface water collected from Chula, Georgia, USA. The water was clear, free of debris and used without filtration (pelagic) on the day of collection. A summary of the physical and chemical properties of the surface water is provided in Table .

**Table 137 Surface water characteristics**

| Parameter | Value | Parameter | Value |
|---|---|---|---|
| Water Identity | Chula | Total Nitrogen (ppm) | 1.1 |
| pH | 7.2 | Nitrate-Nitrogen (ppm) | <0.1 |
| Biochemical Oxygen Demand (mg-$O_2$/L) | 28.1 | Total Phosphorus (ppm) | 2.9 |
| Hardness (mg-equivalent $CaCO_3$/L) | 74 | Phosphate-P (ppm) | <0.1 |
| Alkalinity (mg $CaCO_3$/L) | 37 | Total Suspended Solids (ppm) | 12 |
| Conductivity (mmhos/cm) | 0.20 | Total Organic Carbon (ppm) | 8.4 |

### B. STUDY DESIGN

1. Experimental conditions

   Stock solutions of $^{14}$C-labeled oxamyl in acetonitrile were prepared and aliquots added to bioreactors containing 700 mL of surface water to give a concentration range. Two concentrations were tested consisting of bulk samples at nominal values of 20 µg/L (low rate) and 200 µg/L (high rate) of test substance.

   For mineralization testing, a sub-sample (40 mL) from the bulk sample in the bioreactor was removed and assayed for radioactivity followed by acidification to pH 2–3 using concentrated hydrochloric acid. Samples were then purged with air to expel any $CO_2$. Decreases in radioactivity following acidification

were evaluated as possible mineralization. The potential for biotransformation was determined by HPLC analysis of the surface water prior to acidification. Material balance was determined by direct measurement of the mass balance bioreactor flasks.

2. Sampling

Sampling was performed following test substance application (zero time) and after the following intervals of incubation: 1, 2, 3, 5, 7, 14, 28, 46, and 60 days.

3. Description of analytical procedures

Radioactivity was determined by LSC. The mean LOD was 0.064 µg/L, which corresponded to 0.318% and 0.032% AR for the low and high rates, respectively. The mean LOQ was 0.245 µg/L, which corresponded to 1.23% and 0.123% AR for the low and high rates, respectively.

Aliquots of the highest test concentration samples were analysed prior to acidification by reverse phase HPLC (Phenomenex, Aqua C18 [250 mm × 4.6 mm × 5 µm id]) eluted with a gradient of water and acetonitrile. The mean LOD and LOQ were 0.59 µg/L (0.297% AR) and 1.19 µg/L (0.594% AR), respectively.

## II. RESULTS AND DISCUSSION

### A. MASS BALANCE

The mean material balances of the low and high dose rates in the mass balance bioreactors ($F_M$) were 98.6% and 99.3% at Day 0 for the [1-$^{14}$C]oxamyl of the dosed radioactivity. The mean material balances at termination (Day 60) for the low and high dose rates were 97.4% and 98.8% for the [1-$^{14}$C]oxamyl. A summary of mass balance results are presented in Table .

### B. MINERALIZATION

At termination, the mean mineralization was 5.2% (range: -1.5% to 6.1%) and 6.4% (range: -0.7% to 6.4%) for low and high dose samples, respectively. The reference compound (sodium benzoate) was mineralized up to a mean of 95.6% at termination, confirming the viability of the test system. Mineralization rate constants were calculated to be 552 day $^{-1}$ and 671 day $^{-1}$ for the 20 µg/L and 200 µg/L dose rates, respectively. A summary of results is presented in Table through Table .

### C. TRANSFORMATION OF PARENT COMPOUND

Oxamyl undergoes limited mineralization in natural surface water, but is rapidly degraded *via* hydrolysis to form its oxime (IN-A2213). Oxamyl decreased in the surface water from 93.9% AR at Day 0 to 0.0% AR at the end of 60 days of incubation. The major intact transformation product detected was the oxime (IN-A2213), which reached maximum concentration at Day 46 of 85.5% AR, decreasing slightly to 85.2% AR by Day 60. IN-D2708 was a minor degradation product reaching a maximum of 1.5% at Day 0 and Day 45 and then declined to 0% AR by Day 5. All remaining unidentified degradation products detected in surface water consisted of multiple components. No single component was ≥5 % AR. A summary of biotransformation results is presented in Table and Table 44.

**Table 138 Material balance**

| | **Time (day)** | | | | |
|---|---|---|---|---|---|
| | **0** | **60** | | | |
| **Sample ID** | **Water phase** | **Water phase** | **NaOH taps** | **Glassware rinse** | **Total** |
| FM-591W-20-R1 | 98.3 | 95.0 | 2.9 | 0.3 | 98.2 |
| FM-591W-20-R2 | 98.9 | 93.4 | 2.7 | 0.4 | 96.5 |
| **Mean** | **98.6** | 94.2 | 2.8 | 0.4 | **97.4** |
| FM-591W-200-R1 | 99.1 | 96.2 | 1.9 | 0.5 | 98.6 |
| FM-591W-200-R2 | 99.5 | 96.3 | 2.3 | 0.3 | 98.9 |
| **Mean** | **99.3** | 96.3 | 2.1 | 0.4 | **98.8** |

**Table 139 Mineralization results of oxamyl (20 µg/L)**

| Chula pond surface water | Rep. | Sampling interval (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **2** | **3** | **5** | **7** | **14** | **28** | **46** | **60** |
| Percentage in Surface Water (%)[a] (Pre-Acidification) | R1 | 98.7 | 98.8 | 98.6 | 98.9 | 99.0 | 98.0 | 98.0 | 94.1 | 93.6 | 94.0 |
| | R2 | 96.0 | 98.5 | 97.6 | 97.6 | 98.6 | 97.7 | 97.1 | 92.2 | 95.8 | 93.4 |
| | **Mean** | **97.3** | **98.7** | **98.1** | **98.3** | **98.8** | **97.9** | **97.6** | **93.1** | **94.7** | **93.7** |
| Percentage in Surface Water (%) (Post Acidification) | R1 | 99.4 | 98.8 | 98.6 | 99.8 | 99.3 | 99.0 | 98.3 | 91.4 | 93.4 | 93.1 |
| | R2 | 98.2 | 96.9 | 97.7 | 96.9 | 96.4 | 97.1 | 95.5 | 91.1 | 93.4 | 91.1 |
| | **Mean** | **98.8** | **97.9** | **98.1** | **98.3** | **97.9** | **98.1** | **96.9** | **91.2** | **93.4** | **92.1** |
| Mineralization (%)[b] | R1 | -0.7 | -0.1 | 0.1 | -1.1 | -0.7 | -0.3 | 0.4 | 7.3 | 5.2 | 5.5 |
| | R2 | -2.2 | -1.0 | -1.7 | -0.9 | -0.4 | -1.1 | 0.5 | 4.9 | 2.6 | 4.9 |
| | **Mean** | **-1.5** | **-0.5** | **-0.8** | **-1.0** | **-0.5** | **-0.7** | **0.4** | **6.1** | **3.9** | **5.2** |

[a] Percentages are based on the concentration (dpm/ mL) at each interval sampling relative to the dose control concentration.

[b] Mineralization is the calculated difference between the percentage in phase (pre-acidification) at Day 0 and the percentage in the water (post-acidification) at each sampling interval.

**Table 140 Mineralization results of oxamyl (200 µg/L)**

| **Chula pond surface water** | **Rep.** | **Sampling interval (days)** | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **2** | **3** | **5** | **7** | **14** | **28** | **46** | **60** |
| **Percentage in Surface Water (%)[a] (Pre-Acidification)** | **R1** | **98.7** | **98.8** | **97.9** | **98.9** | **98.3** | **99.1** | **97.8** | **92.0** | **96.4** | **97.3** |
| | **R2** | **98.3** | **98.4** | **98.0** | **98.7** | **98.8** | **99.2** | **97.3** | **93.2** | **96.7** | **97.2** |
| | **Mean** | **98.5** | **98.6** | **98.0** | **98.8** | **98.5** | **99.1** | **97.5** | **92.6** | **96.5** | **97.2** |
| **Percentage in Surface Water (%) (Post Acidification)** | **R1** | **96.8** | **97.5** | **97.6** | **99.3** | **98.2** | **98.7** | **97.2** | **92.4** | **93.2** | **91.2** |
| | **R2** | **98.3** | **98.2** | **97.2** | **99.2** | **98.2** | **98.2** | **97.2** | **92.6** | **94.9** | **93.1** |
| | **Mean** | **97.5** | **97.8** | **97.4** | **99.3** | **98.2** | **98.4** | **97.2** | **92.5** | **94.0** | **92.1** |
| **Mineralization (%)[b]** | **R1** | **2.0** | **1.3** | **1.1** | **-0.6** | **0.5** | **0.0** | **1.5** | **6.3** | **5.5** | **7.6** |
| | **R2** | **0.0** | **0.1** | **1.1** | **-0.9** | **0.0** | **0.1** | **1.1** | **5.7** | **3.4** | **5.2** |
| | **Mean** | **1.0** | **0.7** | **1.1** | **-0.7** | **0.3** | **0.1** | **1.3** | **6.0** | **4.5** | **6.4** |

**[a] Percentages are based on the concentration (dpm/ mL) at each interval sampling relative to the dose control concentration.**

**[b] Mineralization is the calculated difference between the percentage in phase (pre-acidification) at Day 0 and the percentage in the water (post-acidification) at each sampling interval.**

**Table 141 Mineralization results of reference compound [$^{14}$C]sodium benzoate (500 µg/L)**

| Chula pond surface water | Rep. | Sampling interval (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **1** | **2** | **3** | **5** | **7** | **14** | **28** | **46** | **60** |
| Percentage in Surface Water (%)[a] (Pre-Acidification) | R1 | 100.0 | 89.0 | 70.0 | 71.1 | 61.0 | 48.8 | 35.4 | 27.3 | 22.5 | 21.2 |
| | R2 | 100.0 | 96.0 | 85.9 | 75.4 | 62.3 | 56.0 | 42.2 | 36.0 | 28.1 | 30.2 |
| | **Mean** | **100.0** | **92.5** | **78.0** | **73.3** | **61.6** | **52.4** | **38.8** | **31.6** | **25.3** | **25.7** |
| Percentage in Surface Water (%) (Post Acidification) | R1 | 101.8 | 40.1 | 51.8 | 36.3 | 30.3 | 24.9 | 18.5 | 12.3 | 5.3 | 4.4 |
| | R2 | 100.1 | 28.8 | 29.5 | 30.5 | 25.8 | 25.1 | 10.7 | 12.8 | 6.4 | 4.4 |
| | **Mean** | **101.0** | **34.5** | **40.6** | **33.4** | **28.1** | **25.0** | **14.6** | **12.6** | **5.9** | **4.4** |
| Mineralization (%)[b] | R1 | -1.8 | 59.9 | 48.2 | 63.7 | 69.7 | 75.1 | 81.5 | 87.7 | 94.7 | 95.6 |
| | R2 | -0.1 | 71.2 | 70.5 | 69.5 | 74.2 | 74.9 | 89.3 | 87.2 | 93.6 | 95.6 |
| | **Mean** | **1.0** | **65.5** | **59.4** | **66.6** | **71.9** | **75.0** | **85.4** | **87.4** | **94.1** | **95.6** |

[a] Percentages are based on the concentration (dpm/ mL) at each interval sampling relative to 100%.

[b] Mineralization is the calculated difference between 100% (pre-acidification) and the percentage in the water (post-acidification).

**Table 142 Biotransformation of [1-$^{14}$C]oxamyl, expressed as percentage of applied radioactivity**

| Compound | Sampling interval (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | **0** | **1** | **2** | **3** | **5** | **7** | **14** | **28** | **46** | **60** |
| As oxamyl | 93.9 | 85.4 | 77.3 | 69.0 | 50.1 | 44.4 | 26.6 | 11.5 | 3.8 | 0.0 |
| As IN-D2708 | 1.5 | 1.1 | 0.6 | 1.4 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| As IN-A2213 | 0.6 | 10.2 | 18.2 | 26.8 | 44.0 | 50.2 | 69.3 | 76.5 | 85.5 | 85.2 |
| As unassigned[a] | 2.5 | 1.9 | 1.9 | 1.6 | 4.4 | 4.5 | 1.6 | 4.7 | 7.3 | 12.2 |
| Total Recovered Residue | 98.5 | 98.6 | 98.0 | 98.8 | 98.5 | 99.1 | 97.5 | 92.6 | 96.5 | 97.4 |
| Overall Mean = 97.6% Standard Deviation = ± 1.9% | | | | | | | | | | |

[a] No individual unidentified % peak accounted for ≥5%

**Table 44 Biotransformation of [1-$^{14}$C]oxamyl, expressed as ppm parent equivalent**

| Compound | Sampling interval (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | **0** | **1** | **2** | **3** | **5** | **7** | **14** | **28** | **46** | **60** |
| As oxamyl | 209.7 | 190.9 | 172.6 | 154.1 | 112.0 | 99.3 | 59.4 | 25.7 | 8.4 | 0.0 |
| As IN-D2708 | 3.4 | 2.3 | 1.4 | 3.2 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| As IN-A2213 | 1.4 | 22.9 | 40.6 | 59.8 | 98.4 | 112.2 | 154.8 | 170.8 | 190.9 | 190.4 |
| As unassigned[a] | 5.5 | 4.2 | 4.2 | 3.7 | 9.7 | 10.1 | 3.6 | 10.4 | 16.3 | 27.2 |
| Total Recovered Residue | 220.0 | 220.3 | 218.9 | 220.7 | 220.1 | 221.5 | 217.8 | 206.9 | 215.6 | 217.6 |

[a] No individual unidentified % peak accounted for ≥5%

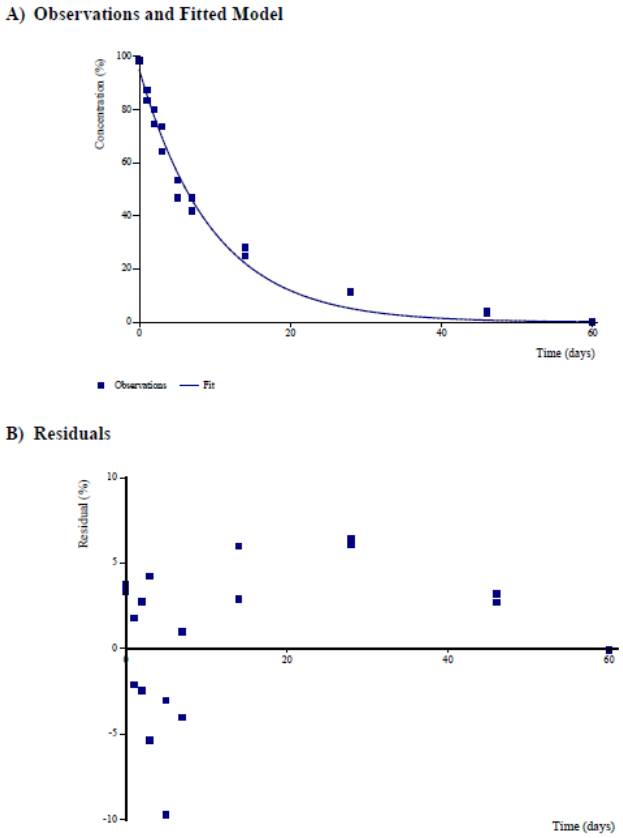
D. KINETIC ANALYSIS

The $DT_{50}$ and $DT_{90}$ values for DPX-D1410 are summarized in the table below.

| COMPONENTS MODELED | MODEL | OPTIMIZED PARAMETERS ± STANDARD ERROR | $\chi^2$ | $r^2$ | $DT_{50}$ (DAYS) | $DT_{90}$ (DAYS) |
|---|---|---|---|---|---|---|
| Surface Water (Chula) | SFO | $M_0$ (%AR) = 94.95 ± 2.151<br>*k* ($d^{-1}$) = 0.1038 ± 0.006696 | 6.04 | 0.9862 | 6.68 | 22.2 |

Plots of the observed and fitted data for DPX-D1410 using the single first-order (SFO) are presented in Figure 9.

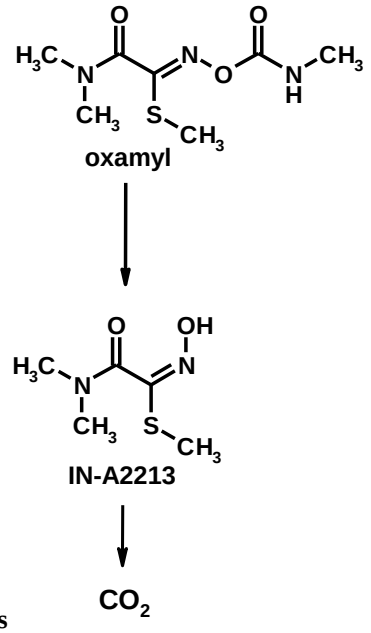
**Figure 9: Single First Order (SFO) regression analysis of the degradation data from surface water (Chula) treated with DPX-D1410**




## III. CONCLUSION

The results of this study indicate that oxamyl undergoes limited mineralization in natural surface water, but is rapidly degraded *via* hydrolysis to form its oxime (IN-A2213). The proposed degradation pathway of oxamyl in surface water is given in **Errore. L'origine riferimento non è stata trovata.**.

**Figure 10 Proposed degradation pathway of oxamyl (DPX-D1410) in surface water under aerobic conditions**




(Allan, J., 2015)

The aerobic mineralization in surface water study DuPont-40441, submitted for the first time in this submission, and conducted with test material [14-C]oxamyl, was conducted under guidelines OECD 309 (April 2004), OPPTS 835.3190 (October 2008) A review of this study indicates that it fully meets the current guideline (OECD 309) and is relied upon.

**RMS comments and conclusion**

The aerobic mineralization of radiolabeled DPX-1410 was studied in one natural surface water.
The DT50 values estimated from the SFO model for the degradation of oxamyl in the surface water was 6.68 days .

#### B.8.2.2.3 Water/sediment study

**B.8.2.2.3/01**

| | | |
|---|---|---|
| **Reference:**<br>-- | **Report:** | Spare, W.C. (1995); Degradability and fate of [1-$^{14}$C]oxamyl in water/sediment systems<br>**DuPont Report No.:** AMR 3143-94<br>**Guidelines:** BBA IV 5-1 (1990) |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: 2868-229
   Purity: Radiochemical purity - 98.5%

**Materials and Methods:**

The metabolism and degradation of oxamyl was studied in two aquatic systems (Red Oak, a stream, and Town Park, a pond) for 100 days. The physical and chemical properties of the aquatic systems are summarised in Table .

**Table 144 Physical and chemical properties of the water/sediment test systems**

| Parameter | Red Oak stream | Town Park pond |
|---|---|---|
| **Water** | | |
| pH | 7.3 | 7.5 |
| Calcium (mg/L) | 41 | 23 |
| Magnesium (mg/L) | 15 | 7 |
| Sodium (mg/L) | 35 | 5 |
| Organic matter (ppm) | 726 | 402 |
| Total nitrogen (ppm) | <1 | <1 |
| Hardness (mg equivalent $CaCO_3$/L) | 164 | 89 |
| Sodium adsorption ratio | 1.19 | 0.24 |
| Total phosphorus (ppm) | <0.01 | <0.01 |
| **Sediment** | | |
| Sand (%) | 66 | 46 |
| Silt (%) | 30 | 48 |
| Clay (%) | 4 | 6 |
| Texture | sandy loam | sandy loam |
| Organic matter (%) | 2.4 | 5.1 |
| Organic carbon (%) | 1.4 | 3.0 |
| pH | 6.7 | 6.1 |
| Olsen phosphorus (ppm) | 15 | 11 |
| Total nitrogen (%) | 0.082 | 0.203 |
| Cation exchange capacity (meq/100 g) | 10.5 | 10.0 |
| Moisture-holding capacity at 33 kPa (%) | 21.9 | 37.3 |
| Bulk density (g/cm$^3$) | 1.37 | 1.11 |

Sediment (50 g dry weight equivalent) and associated water (approximately 200 mL) were placed in each test vessel (Nalgene bottle), and aerobic (water) and anaerobic (sediment) conditions were established over 51 days. [1- $^{14}$C]-oxamyl (specific activity 18.65 kBq/mg, radiochemical purity 98.5%) was then applied to the test vessels at a nominal rate of 2 mg/L, and the samples were incubated in the dark at 20 ±1 °C. Two dosing methods were evaluated. Test systems were either vigorously hand shaken to mix water and sediment after oxamyl application or the oxamyl was gently applied into the water phase only, without mixing of the water and sediment.

Samples were taken 0 and 6 hours after both dosing methods, and on days 1, 2, 7, 14, 30, 61, and 100 after treatment. Traps to collect volatiles and carbon dioxide were changed at each sampling time point or on a twice per month basis. Soil sediment samples were exhaustively extracted with methanol/water and methanol after separation from the water phase. Sediment extracts and water phase samples were analysed by LSC and reversed phase HPLC with radiochemical detection. Degradates were identified by comparison of retention times with authentic standards. Degradate identity was confirmed with TLC and comparison to authentic standards.

Non-extractable radioactivity in soil samples was determined by combustion and LSC. Polyurethane foam volatile traps were analysed by solvent extraction and LSC. Ethanolamine and potassium hydroxide volatile traps were analysed by LSC, both before and after addition of barium chloride to confirm the presence of radiolabelled carbon dioxide.

**Findings:**

The fate of oxamyl was investigated in two natural water/sediment systems. Two differing application techniques were investigated. Firstly, addition of compound to the water phase (reflecting the more likely method of entry into natural water systems) and, secondly, vigorous mixing of water and sediment after application of compound. The second method unsurprisingly resulted in a greater initial association of radioactivity with the sediment, and much higher initial amounts of IN-N0079. The rapid appearance of IN-N0079 after vigorously mixing the water and anaerobic sediment was likely due to oxamyl reduction by ferrous iron (FeII). The ferrous-iron mediated reduction reaction of oxamyl to IN-N0079 is a well-documented process (see footnote 2, section B.8.1.1.2.2).

The total recovery of applied radioactivity was in the range of 91% to 105% for both the stream and pond systems (Table 45 and Table ).

**Table 45 Recovery of radioactivity from Red Oak stream water/sediment system after application of [1-$^{14}$C]-oxamyl at 2 mg/L (% of applied radioactivity)**

| Sample | Water | Sediment | | | Volatile products | | | Total |
|---|---|---|---|---|---|---|---|---|
| | | Extract | Residue | Total | PUF | KOH | E2E | |
| Day 0* | 88 | 14 | 1 | 15 | np | np | np | 103 |
| Day 0, 6 hr* | 90 | 10 | 1 | 11 | 0.0 | 0.1 | 0.0 | 101 |
| Day 0** | 100 | np | np | np | np | np | np | 100 |
| Day 0, 6 hr** | 100 | np | np | np | np | np | np | 100 |
| Day 1 | 97 | 6 | 1 | 7 | 0.0 | 0.3 | 0.0 | 104 |
| Day 2 | 96 | 8 | 1 | 9 | 0.0 | 0.3 | 0.0 | 105 |
| Day 7 | 87 | 11 | 2 | 13 | 0.1 | 4.0 | 0.1 | 104 |
| Day 14 | 81 | 11 | 4 | 15 | 0.1 | 7.0 | 0.2 | 103 |
| Day 30 | 79 | 11 | 4 | 15 | 0.1 | 10.2 | 0.3 | 105 |
| Day 61 | 52 | 10 | 11 | 21 | 0.1 | 24.8 | 0.8 | 99 |
| Day 100 | 2 | 2 | 18 | 20 | 0.2 | 60.9 | 8.0 | 91 |

np = not performed; PUF = polyurethane foam volatile organics trap; KOH = 1M KOH, $CO_2$ trap E2E = ethanolamine/2-ethoxyethanol, $CO_2$ and volatile organics trap
* vigorously mixed water and sediment
** water only

**Table 146 Recovery of radioactivity from Town Park pond water/sediment system after application of [1-$^{14}$C]-oxamyl at 2 mg/L (% of applied radioactivity)**

| Sample | Water | Sediment | | | Volatile products | | | Total |
|---|---|---|---|---|---|---|---|---|
| | | Extract | Residue | Total | PUF | KOH | E2E | |
| Day 0* | 80 | 18 | 2 | 20 | np | np | np | 100 |
| Day 0, 6 hr* | 83 | 15 | 3 | 18 | 0.1 | 0.0 | 0.0 | 101 |
| Day 0** | 100 | np | np | np | np | np | np | 100 |
| Day 0, 6 hr** | 99 | np | np | np | np | np | np | 99 |
| Day 1 | 97 | 5 | 1 | 6 | 0.0 | 0.3 | 0.0 | 103 |
| Day 2 | 95 | 5 | 1 | 6 | 0.1 | 0.5 | 0.0 | 102 |
| Day 7 | 87 | 9 | 3 | 12 | 0.3 | 3.8 | 0.0 | 103 |
| Day 14 | 70 | 12 | 4 | 16 | 0.1 | 10.9 | 0.3 | 97 |
| Day 30 | 67 | 11 | 5 | 16 | 0.2 | 16.3 | 0.8 | 100 |
| Day 61 | 57 | 12 | 6 | 18 | 0.1 | 19.7 | 1.2 | 96 |
| Day 100 | 46 | 11 | 9 | 20 | 0.1 | 27.9 | 1.7 | 96 |

np = not performed; PUF = polyurethane foam volatile organics trap; KOH = 1M KOH, $CO_2$ trap; E2E = ethanolamine/2-ethoxyethanol, $CO_2$ and volatile organics trap
* vigorously mixed water and sediment
** water only

The difference in distribution of the dose between the vigorously mixed samples and those not mixed was significant. In the vigorously mixed samples 15-20% AR was immediately associated with the sediment. This value decreased to 11% AR for the stream system and 18% AR for the pond system at 6 hours. In contrast, gently dosed systems (dosing to the water phase, no mixing) required between 14 and 61 days of incubation before 15-21% AR was associated with the sediment phase. No more than 21% AR was observed to be associated with the sediment from either system over the entire 100-day incubation. The results obtained from the vigorous mixing approach are not discussed further, since the relevance of the vigorous mixing approach of dosing is likely to be low in natural conditions and the effect of mixing aerobic and anaerobic phases is difficult to predict.

In both test systems, the non-extractable sediment radioactivity (residue) gradually increased, reaching 9% AR in the pond system and 18% AR in the stream system after 100 days.

Both systems presented the same degradation pattern of [1- $^{14}$C]-oxamyl, but with different product ratios over the course of the study (Table 46 and Table ). The major (>10% AR) degradates were IN-A2213, IN-D2708, IN-N0079 and CO2. Another degradate, IN-T2921, was also observed in the water and sediment of both test systems between day 1 and day 61; exceeding 10% AR (11.4% AR) at day 14 in the pond system water and reaching 8.6% AR on day 14 in the stream system water. IN-D2708 was the only degradate observed in sediment above 10% AR (10.4-12.1% AR). Both systems generated consistent quantities of $^{14}$CO2 (and small amounts of other volatiles) through 61 days incubation (21.0% AR and 25.7% AR), but from 61 to 100 days the stream system generated another 43.4% AR whereas the pond system generated only another 8.7% AR as volatile products. The cumulative amounts of carbon dioxide evolved by the end of the study accounted for 27.9% AR and 60.9% AR.

**Table 46 Distribution of radioactive degradates in Red Oak stream water/sediment system after application of [1-$^{14}$C]-oxamyl at 2 mg/L (% of applied radioactivity)**

| Sample | Oxamyl | IN- A2213 | IN- D2708 | IN- N0079 | IN-T2921 | IN-L2953 | PROD1 |
|---|---|---|---|---|---|---|---|
| **Water** | | | | | | | |
| Day 0* | 42.9 | 0.8 | nd | 44.3 | nd | nd | nd |
| Day 0, 6 hr* | 56.2 | 9.2 | 1.7 | 23.6 | nd | nd | nd |
| Day 0** | 95.8 | 4.2 | nd | nd | nd | nd | nd |
| Day 0, 6 hr** | 88.0 | 11.7 | nd | nd | nd | nd | nd |
| Day 1 | 43.1 | 42.3 | 1.2 | 10.3 | nd | nd | nd |
| Day 2 | 30.6 | 48.8 | 5.0 | 9.2 | 2.8 | nd | nd |
| Day 7 | 12.5 | 42.1 | 18.2 | 11.3 | 2.6 | nd | nd |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Day 14 | 4.4 | 28.9 | 33.0 | 3.7 | 8.6 | 1.1 | nd |
| Day 30 | nd | 12.2 | 66.8 | nd | nd | nd | nd |
| Day 61 | nd | nd | 48.5 | nd | 3.5 | nd | nd |
| Day 100 | nd | 0.3 | nd | nd | nd | 1.8 | nd |
| **Sediment** | | | | | | | |
| Day 0* | np | np | np | np | np | np | np |
| Day 0, 6 hr* | np | np | np | np | np | np | np |
| Day 0** | np | np | np | np | np | np | np |
| Day 0, 6 hr** | np | np | np | np | np | np | np |
| Day 1 | 0.3 | 3.1 | 2.5 | 0.4 | 0.3 | nd | nd |
| Day 2 | nd | 4.4 | 3.4 | nd | nd | nd | nd |
| Day 7 | nd | 3.7 | 6.1 | 0.7 | 0.3 | 0.2 | 0.3 |
| Day 14 | nd | 2.2 | 7.8 | 0.6 | 0.4 | 0.2 | 0.2 |
| Day 30 | nd | 0.7 | 10.4 | 0.3 | nd | nd | 0.1 |
| Day 61 | 1.2 | 0.3 | 8.2 | nd | nd | 0.1 | nd |
| Day 100 | nd | nd | 1.6 | nd | nd | nd | nd |

nd = not detected; np = not performed; PROD1 is an unidentified degradate (<1% AR)
* vigorously mixed water and sediment
** water only

**Table 148 Distribution of radioactive degradates in Town Park pond water/sediment system after application of [1-$^{14}$C]-oxamyl at 2 mg/L (% of applied radioactivity)**

| Sample | Oxamyl | IN-A2213 | IN-D2708 | IN-N0079 | IN-T2921 | IN-L2953 | PROD1 |
|---|---|---|---|---|---|---|---|
| **Water** | | | | | | | |
| Day 0* | 21.4 | nd | nd | 58.3 | nd | nd | nd |
| Day 0, 6 hr* | 5.8 | 0.8 | 0.9 | 75.5 | nd | nd | nd |
| Day 0** | 97.2 | 0.7 | nd | 2.1 | nd | nd | nd |
| Day 0, 6 hr** | 87.6 | 11.1 | nd | nd | nd | nd | nd |
| Day 1 | 36.8 | 21.4 | 3.2 | 35.2 | nd | nd | nd |
| Day 2 | 13.1 | 25.3 | 2.4 | 52.9 | 1.7 | nd | nd |
| Day 7 | 0.9 | 16.2 | 15.6 | 45.1 | 5.3 | 3.8 | nd |
| Day 14 | 4.0 | 5.5 | 32.0 | 16.9 | 11.4 | nd | nd |
| Day 30 | nd | 2.7 | 64.2 | nd | nd | nd | nd |
| Day 61 | nd | nd | 56.7 | nd | nd | nd | nd |
| Day 100 | 0.1 | 0.2 | 45.0 | 0.1 | nd | 0.4 | nd |
| **Sediment** | | | | | | | |
| Day 0* | np | np | np | np | np | np | np |
| Day 0, 6 hr* | np | np | np | np | np | np | np |
| Day 0** | np | np | np | np | np | np | np |
| Day 0, 6 hr** | np | np | np | np | np | np | np |
| Day 1 | <0.1 | 2.1 | 1.5 | 0.9 | nd | nd | nd |
| Day 2 | nd | 1.5 | 2.2 | 1.3 | nd | nd | nd |
| Day 7 | nd | 1.0 | 3.9 | 3.7 | 0.2 | 0.1 | 0.6 |
| Day 14 | nd | 0.7 | 6.5 | 3.1 | 0.4 | 0.1 | 0.8 |
| Day 30 | nd | 0.1 | 9.8 | 0.6 | nd | nd | 0.3 |
| Day 61 | nd | nd | 12.1 | nd | nd | nd | nd |
| Day 100 | nd | 0.1 | 10.7 | 0.2 | nd | 0.1 | 0.1 |

nd = not detected; np = not performed; PROD1 is an unidentified degradate (<1% AR)
* vigorously mixed water and sediment
** water only

Oxamyl degraded rapidly in the water phase (accounting for <50% AR on day 1) and significant amounts were never found in the sediment phase. Since both water systems were slightly alkaline it is likely that a component of this rapid degradation was the result of chemical hydrolysis. IN-A2213, the hydrolysis product, reached a maximum of 25.3-48.8% AR in the water phase on day 2 and then decreased to non-detectable levels by day 61.

Low levels were found in the sediments (maximum of 4.4% AR) and the pattern reflected that observed in the water phase.

High levels of IN-N0079 were observed in the water phase of the Town Park pond system (maximum of 52.9% AR at day 2), and the pattern of formation and decline was parallel to that of IN-A2213. As noted above, the rapid appearance of significant amounts of IN-N0079 was likely due to the ferrous-iron mediated reduction reaction of oxamyl, with FeII near or within the anaerobic sediment phase.

Following the decline of IN-A2213 and IN-N0079, levels of IN-D2708 in the water subsequently rose (maximum levels of 64.2% AR and 66.8% AR at day 30), and then declined during the remainder of the study. IN-D2708 was detected in the sediment phase on all sampling occasions, being observed at maximum levels of 10.4% AR and 12.1% AR. The pattern of occurrence of this sediment residue coincided with the maximum level of IN-D2708 in the water phase, suggesting a simple gradient diffusion of IN-D2708 from the water phase into the sediment pore water in the static test system.

Levels of IN-T2921 exceeded 10% AR in the water phase of the Town Park pond system. However, this was only at one timepoint (day 14), and the level was 11.4% AR. It subsequently degraded such that it was not detected at the next sampling timepoint (day 30).

The proposed degradation pathway in the water sediment/systems (aerobic water, anaerobic sediment) is given in Figure 8.4.3.2-1.

The original report proposed IN-N0079 to be part of a linear degradation pathway between IN-A2213 and IN-T2921. Based on information generated in subsequent degradation studies, it is proposed that IN-A2213 and IN-N0079 are actually produced in separate pathways; IN-A2213 via base-catalysed hydrolysis and IN-N0079 via reduction by FeII.

Whole-system DT50 and DT90 values for oxamyl were calculated in the report using the non- linear method developed by Gustafson and Holden, which is based on the notion of a spatially variable first order degradation rate constant for a soil system comprising multiple compartments.[1]

[The equation derived from this method, relating the amount of a substance remaining to time elapsed, describes the average degradation from an ensemble of compartments, and can be stated as:

$C = C0(1 + \beta t)^{-\alpha}$ or $\ln C = \ln C0 - \alpha \ln (1 + \beta t)$

where C is the concentration of substance at any given time t, C0 is the initial concentration, t is time and α and β are non-linear regression fitting parameters.

The expressions for DT50 and DT90 obtained from the above equation (based on the initial amount of substance present) are $DT50 = [0.5(-1/\alpha) - 1]/\beta$ and $DT90 = [0.1(-1/\alpha) - 1]/\beta$.]

However, in order to obtain degradation rates for the metabolites as well, the notifier attempted to re-estimate whole-system DT50 and DT90 values from the residue data using non-linear regression of integrated rate equations based on simple first order kinetics (with parent and metabolites considered in series). [A detailed description of the equations and calculations involved has been given in the description of the main laboratory soil degradation rate study (report no. DuPont-2957), as reported in section B.8.1.2.1.]

This latter approach enabled the re-calculation of DT50 and DT90 values for oxamyl but it was not suitable for the metabolites. Therefore, whole-system DT50 and DT90 values for the metabolites were individually re-calculated from the study data using linear least squares regression, assuming simple first order kinetics. The results of the original analysis for oxamyl and the re-calculated DT50 and DT90 values for oxamyl and metabolites are presented in Table .

**Table 149 Degradation rate values for oxamyl and metabolites in stream and pond water/sediment systems**

| Compound | Rate ($d^{-1}$) | $DT_{50}$ (day) | $DT_{90}$ (day) | Data points used (day) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| **Red Oak Stream water/sediment (total system)** | | | | | | |

[1] Gustafson, D.I. and Holden, L.R. (1990): Nonlinear Pesticide Dissipation in Soil. Environ. Sci. Technol. 1990, 24, 1032-1038.

| Compound | Rate ($d^{-1}$) | $DT_{50}$ (day) | $DT_{90}$ (day) | Data points used (day) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| Oxamyl | - | 1.0* | 7.8* | 0, 0.25, 1, 2, 7, 14, 61 | 0.993* | non-linear |
| | 0.644 | 1.1 | 3.6 | 0, 0.25, 1, 2, 7, 14, 61 | 0.972 | non-linear simple first order |
| IN-A2213 | 0.0608 | 11.4 | 37.9 | 2, 7, 14, 30, 61, 100 | 0.891 | linear simple first order |
| IN-D2708 | 0.0569 | 12.2 | 40.5 | 30, 61, 100 | 0.858 | linear simple first order |
| IN-N0079 | 0.161 | 4.3 | 14.3 | 7, 14, 30 | 0.999 | linear simple first order |
| IN-T2921 | 0.020 | 34.5 | 115 | 14, 61 | na | linear simple first order |
| **Town Park Pond water/sediment (total system)** | | | | | | |
| Oxamyl | - | 0.4* | 2.8* | 0, 0.25, 1, 2, 7, 14, 100 | 0.920* | non-linear |
| | 0.957 | 0.7 | 2.4 | 0, 0.25, 1, 2, 7, 14, 100 | 0.990 | non-linear simple first order |
| IN-A2213 | 0.0426 | 16.3 | 54.1 | 2, 7, 14, 30, 100 | 0.930 | linear simple first order |
| IN-D2708 | 0.00410 | 169 | 562 | 30, 61, 100 | 0.957 | linear simple first order |
| IN-N0079 | 0.0524 | 13.2 | 43.9 | 2, 7, 14, 30, 100 | 0.712 | linear simple first order |
| IN-T2921 | 0.298 | 2.3 | 7.8 | 14, 30 | na | linear simple first order |

* Original value from study report
na = not applicable (only two data points were used for IN-T2921)

**Conclusions:**

In two water/sediment systems collected from a pond and stream, the whole-system DT50 (non-linear, non-first order) ranged from 0.4 to 1.0 day and the whole-system DT90 ranged from 2.8 to 7.8 days. Re-calculation of the study data provided first order kinetic values (whole-system) for oxamyl and metabolites. The first order DT50 values were 0.7 to 1.1 days for oxamyl, 11.4 to 16.3 days for IN-A2213, 12.2 to 169 days for IN-D2708, 4.3 to 13.2 days for IN-N0079 and 2.3 to 34.5 days for IN-T2921. Due to limited partitioning into sediment, these rates essentially represent the degradation rates in the water phase.

Both systems presented the same degradation pattern but at different product ratios over the course of the study. The major degradates were IN-A2213, IN-D2708, IN-N0079, IN-T2921, and carbon dioxide. Overall the water/sediment study shows degradation of oxamyl to IN- A2213 or IN-N0079 and further to IN-T2921 and IN-D2708 in the water phase. No compounds reached consistently significant levels in the sediment. Carbon dioxide was the ultimate degradation product in both test systems.

The water/sediment study AMR 3143-94, originally submitted under EU Rev8 Point IIA 7.2.1.3.2 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline BBA IV 5-1 (1990). A review of this study indicates that it fully meets the current guideline (OECD 308) and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.

The study does not contain a kinetic evaluation according to recent FOCUS recommendations (FOCUS, 2006, 2011).Residue data of this study were re-evaluated to derive persistence and modelling endpoints for oxamyl and its metabolites IN-A2213, IN–D2708, IN-N0079, and IN-T2921 under aerobic conditions in water/sediment systems. See the new study below.

**Study submitted to the EU for the first time in this submission.**

**B.8.2.2.3/02**

| | | |
|---|---|---|
| **Reference: CA 7.2.2.3/02** | **Report:** | Ghafoor, A., Zillgens, B. (2015); Estimation of kinetic endpoints of oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) and IN-T2921 in water/sediment systems – Kinetic calculations following FOCUS kinetics guidelines<br>**DuPont Report No.:** DuPont-44046 EU<br>**Guidelines:** Council Directive 91/414/EEC, AIR-3, Annexes II and III, Article 79 (2) of EU Regulations (EC) No. 1107/2009 (14 June 2011), EU Regulations (EC) No. 1136/2013 (02-03 Oct 2013)<br>**Deviations:** None<br>**Testing Facility:** Dr. Knoell Consult GmbH, Mannheim, Germany<br>**Testing Facility Report No.:** DuPont-44046 EU<br>**GLP:** No<br>**Certifying Authority:** Not applicable |

**Executive Summary**

A complete summary of this study can be found in the Oxamyl EU Renewal Dossier, Document M-CP, Section 9 (DuPont-40953 EU and DuPont-42129 EU). Briefly, one aquatic degradation study has been conducted to investigate the rate of degradation of oxamyl and its metabolites in two water/sediment systems (AMR 3143-94). Since the study does not contain a kinetic evaluation according to recent FOCUS recommendations (FOCUS, 2006, 2011), residue data of this study were re-evaluated to derive persistence and modelling endpoints for oxamyl and its metabolites IN-A2213, IN–D2708, IN-N0079, and IN-T2921 under aerobic conditions in water/sediment systems.

The kinetic evaluation of the water/sediment study was carried out using the one-compartmental approach (level I) as described in FOCUS guidance documents. For oxamyl, level I consisted of the derivation of a total-system degradation half-life and dissipation half-lives for sediment and the water phase. For metabolites, level I analysis consisted of the derivation of a degradation half-life for the total-system and dissipation half-lives for total system, sediment, and water phase. The sediment level I analysis for oxamyl and the level I dissipation analyses for metabolites were conducted based upon the decline from the maximum occurrence.

The persistence $DT_{50}$ values for oxamyl in the whole system were 0.69 to 0.82 day and the $DT_{90}$ values were 2.28 to 8.31 days, but these are also effectively the values in the water phase. Robust whole system $DT_{50}$ values were also calculated for IN-A2213, and they ranged from 5.67 to 8.24.

The persistence and modelling endpoints, derived from this laboratory water/sediment study and chosen according to FOCUS (2006, 2011) guidelines, are summarised in the following tables.

**Table 150 Summary of water/sediment study persistence endpoints for oxamyl (a) and its metabolites (IN-A2213 (b), IN-D2708 (c), IN-N0079 (d), and IN-T2921 (e) ).**

**a) Oxamyl**

| System | Water/sediment system | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | System | $DegT_{50}$ = 0.82<br>$DegT_{90}$ = 8.31 | P-I; HS Best-fit Model |
| | Water | $DT_{50}$ = 0.82<br>$DT_{90}$ = 8.31 | P-I; HS Best-fit Model |
| | Sediment | - | Oxamyl appeared only in small amounts at only 2 data points |
| Town Park Pond | System | $DegT_{50}$ = 0.69<br>$DegT_{90}$ = 2.28 | P-I; SFO Best-fit Model |
| | Water | $DT_{50}$ = 0.69<br>$DT_{90}$ = 2.28 | P-I; SFO Best-fit Model |
| | Sediment | - | Oxamyl did not appear in sediment |

**b) IN-A2213**

| System | Water/sediment system | Values in days | Best fit model | Type of endpoint and comments |
|---|---|---|---|---|
| Red Oak Stream | System | $DegT_{50}$ = 8.24<br>$DegT_{90}$ = 27.38 | HS-SFO | System degradation endpoint |
| | Water | $DT_{50}$ = 14.16<br>$DT_{90}$ = 47.05 | SFO | Water decline endpoint |
| | Sediment | $DT_{50}$ = 11.62<br>$DT_{90}$ = 38.61 | SFO | Sediment decline endpoint |
| Town Park Pond | System | $DegT_{50}$ = 5.67<br>$DegT_{90}$ = 18.84 | SFO-SFO | System degradation endpoint |
| | Water | $DT_{50}$ = 6.50<br>$DT_{90}$ = 21.58 | SFO | Water decline endpoint |
| | Sediment | $DT_{50}$ = 5.15<br>$DT_{90}$ = 28.10 | HS | Sediment decline endpoint |

**c) IN-D2708**

| System | Water/sediment system | Values in days | Best fit model | Type of endpoint and comments |
|---|---|---|---|---|
| Red Oak Stream | System | - | - | M-I, No Decline, Default $DT_{50}$ |
| | Water | - | - | M-I, No Decline, Default $DT_{50}$ |
| | Sediment | - | - | M-I, No Decline, Default $DT_{50}$ |
| Town Park Pond | System | $DegT_{50}$ = 185.73<br>$DegT_{90}$ = 617.00 | SFO-SFO | System degradation endpoint |
| | Water | $DegT_{50}$ = 185.73<br>$DegT_{90}$ = 617.00 | SFO-SFO | Water decline endpoint |
| | Sediment | $DegT_{50}$ = 185.73<br>$DegT_{90}$ = 617.00 | SFO-SFO | Sediment decline endpoint |

**d) IN-N0079**

| System | Water/sediment system | Values in days | Best fit model | Type of endpoint and comments |
|---|---|---|---|---|
| Red Oak Stream | System | $DegT_{50}$ = 4.69<br>$DegT_{90}$ = 15.58 | SFO | System degradation endpoint |
| | Water | $DT_{50}$ = 4.26<br>$DT_{90}$ = 14.15 | SFO | Water decline endpoint |
| | Sediment | $DT_{50}$ = 17.79<br>$DT_{90}$ = 59.08 | SFO | Sediment decline endpoint |
| Town Park Pond | System | $DegT_{50}$ = 8.53<br>$DegT_{90}$ = 28.34 | SFO-SFO | System degradation endpoint |
| | Water | $DT_{50}$ = 8.07<br>$DT_{90}$ = 26.82 | SFO | Water decline endpoint |
| | Sediment | $DT_{50}$ = 11.38<br>$DT_{90}$ = 37.81 | SFO | Sediment decline endpoint |

**e) IN-T2921**

| System | Water/sediment system | Values in days | Best fit model | Type of endpoint and comments |
|---|---|---|---|---|
| Red Oak Stream | System | $DegT_{50}$ = 27.31<br>$DegT_{90}$ = 90.71 | HS-SFO | System degradation endpoint |
| | Water | - | - | M-I, No Decline, Default $DT_{50}$ |
| | Sediment | - | - | M-I, No Decline, Default $DT_{50}$ |
| Town Park Pond | System | - | - | M-I, No Decline, Default $DT_{50}$ |
| | Water | - | - | M-I, No Decline, Default $DT_{50}$ |
| | Sediment | - | - | M-I, No Decline, Default $DT_{50}$ |

**Table 151 Summary of water/sediment study modeling endpoints for oxamyl (a) and its metabolites (IN-A2213 (b), IN-D2708 (c), IN-N0079 (d), and IN-T2921 (e) ).**

**a) Oxamyl**

| System | FOCUS Step | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | Step 1 | $DegT_{50}$ = 2.50 | P-I Total System; HS $DegT_{90}$/3.32 |
| | Step 2 | $DegT_{50}$ = 2.50 | P-I Total System; HS $DegT_{90}$/3.32 |
| | Step 3 | Water: $DegT_{50}$ = 2.50<br>Sediment: $DT_{50}$ = 1000 | P-I Total System; HS $DegT_{90}$/3.32<br>Default assumption |
| Town Park Pond | Step 1 | $DegT_{50}$ = 0.69 | P-I Total System; SFO |
| | Step 2 | $DegT_{50}$ = 0.69 | P-I Total System; SFO |
| | Step 3 | Water: 0.69<br>Sediment: 1000 | P-I Total System; SFO<br>Default assumption |

**b) IN-A2213**

| System | FOCUS Step | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | Step 1 | $DT_{50}$ = 13.95 | M-I System decline, SFO |
| | Step 2 | $DT_{50}$ = 13.95 | M-I System decline, SFO |
| | Step 3 | Water: $DegT_{50}$ = 8.24<br>Sediment: $DT_{50}$ = 1000 | M-I System degradation, HS-SFO<br>Default assumption |
| Town Park Pond | Step 1 | $DT_{50}$ = 6.65 | M-I System decline, SFO |
| | Step 2 | $DT_{50}$ = 6.65 | M-I System decline, SFO |
| | Step 3 | Water: $DegT_{50}$ = 5.67<br>Sediment: $DT_{50}$ = 1000 | M-I System degradation, SFO-SFO<br>Default assumption |

**c) IN-D2708**

| System | FOCUS Step | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | Step 1 | $DT_{50}$ = 1000 | M-I, No Decline observed, Default $DT_{50}$ |
| | Step 2 | $DT_{50}$ = 1000 | M-I, No Decline, Default $DT_{50}$ |
| | Step 3 | Water: $DT_{50}$ = 1000<br>Sediment: $DT_{50}$ = 1000 | No Decline observed<br>Default assumption |
| Town Park Pond | Step 1 | $DT_{50}$ = 1000 | M-I, No Decline, Default $DT_{50}$ |
| | Step 2 | $DT_{50}$ = 1000 | M-I, No Decline, Default $DT_{50}$ |
| | Step 3 | Water: $DegT_{50}$ = 185.73<br>Sediment: $DT_{50}$ = 1000 | M-I System degradation, SFO-SFO<br>Sediment: Default assumption |

**d) IN-N0079**

| System | FOCUS Step | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | Step 1 | $DT_{50}$ = 4.69 | M-I System decline, SFO |
| | Step 2 | $DT_{50}$ = 4.69 | M-I System decline, SFO |
| | Step 3 | Water: $DegT_{50}$ = 1000<br>Sediment: $DT_{50}$ = 1000 | M-I System degradation not reliable, use default $DT_{50}$ SFO-SFO<br>Default assumption |
| Town Park Pond | Step 1 | $DT_{50}$ = 8.80 | M-I System decline, SFO |
| | Step 2 | $DT_{50}$ = 8.80 | M-I System decline, SFO |
| | Step 3 | Water: $DegT_{50}$ = 8.53<br>Sediment: $DT_{50}$ = 1000 | M-I System degradation, SFO-SFO<br>Default assumption |

**e) IN-T2921**

| System | FOCUS Step | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | Step 1 | $DT_{50}$ = 1000 | M-I, No decline observed, Default $DT_{50}$ |
| | Step 2 | $DT_{50}$ = 1000 | M-I, No Decline, Default $DT_{50}$ |
| | Step 3 | Water: $DegT_{50}$ = 27.31<br>Sediment: $DT_{50}$ = 1000 | M-I System degradation; HS-SFO<br>Default assumption |
| Town Park Pond | Step 1 | $DT_{50}$ = 1000 | M-I, No decline observed, Default $DT_{50}$ |
| | Step 2 | $DT_{50}$ = 1000 | M-I, No Decline, Default $DT_{50}$ |
| | Step 3 | Water: $DT_{50}$ = 1000<br>Sediment: $DT_{50}$ = 1000 | M-I, No Decline, Default $DT_{50}$<br>Default assumption |

**RMS comments and conclusion**

This study is considered corrected to harmonize the derivation of degradation parameters from water.
Residue data of aerobic degradation in one water-sediment study for oxamyl under laboratory conditions with a total of two sediment test systems, (DuPont-AMR 3143-94) were re-evaluated to derive persistence and modelling endpoints for oxamyl and its metabolites
The kinetic assessments conducted are in full compliance with the FOCUS kinetics guidance and the imput parameters can be used for surface water risk assessment.

**Study submitted to the EU for the first time in this submission.**

**B.8.2.2.3/03**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.2.2.3/01** | **Report:** | Clark, B. (2013); Anaerobic aquatic metabolism of [$^{14}$C]-DPX-D1410 (oxamyl) in two water-sediment systems<br>**DuPont Report No.:** DuPont-34157<br>**Guidelines:** OECD 308 (April 2002), OPPTS 835.4400 (June 2008), SETAC Europe (1995)<br>**Deviations:** None<br>**Testing Facility:** ABC Laboratories, Inc. (Missouri), Columbia, Missouri, USA<br>**Testing Facility Report No.:** 68067<br>**GLP:** Yes<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

**Executive summary:**

The biotransformation of [1-$^{14}$C]-oxamyl was studied in two water/sediment systems for up to 102 days under anaerobic conditions in the dark at 20 ± 2°C. The water/sediment systems were from Chula, Tift County, Georgia, USA, and Wabasha, Wabasha County, Minnesota, USA. The Tift water had a pH of 7.9, while the sediment was characterized as a sandy loam with a pH of 7.8 (1:1 soil:water ratio) and organic matter content of 0.43%. The Wabasha water had a pH of 7.4, while the sediment was characterized as a loam with a pH of 6.2 (1:1 soil: water ratio) and organic matter content of 6.3%.

The water phase of the test systems were treated with [1-$^{14}$C]oxamyl at a rate of 5.00 µg/g water. The test systems were incubated in darkness at 20 ± 2°C. Anaerobic conditions were maintained by passing a steady stream of humidified nitrogen through the test apparatus. To collect volatile radioactivity ($CO_2$ and organic volatiles), the air exiting the test vessels was passed through a set of traps: a Supelco ORBO™-100 adsorbent tube placed in-line followed by two base traps, each containing 30 mL 1 N KOH solution. Duplicate samples were taken at 0, 0.25, 0.5, 1, 5, 15, 29, 61, 81, and 102 days after application. The water and sediment layers were first separated by centrifuge and decanting. The water layer was analysed by LSC and then analysed by direct injection using reversed-phase HPLC. The sediment layer was extracted by shaking in 50:50 acetonitrile:water, then centrifuged to separate the extract. Extracts were analysed by LSC, and then concentrated, filtered, and analysed by reversed-phase HPLC. The base volatile traps were analysed by LSC, while the Orbo tubes were not further analysed, because mass balance was maintained.

The mean material balance was 95.8 ± 3.9% and 95.9 ± 4.5% of the applied radioactivity (% AR) for the [1-$^{14}$C]oxamyl treated Tift and Wabasha samples, respectively. The amount of radioactivity in the water layer generally decreased over the course of the study from a mean of 97.2% and 98.0% AR at Day 0 to a mean of 36.5% and 13.7% AR at Day 102 for the Tift and Wabasha samples, respectively. At Day 0, the mean observed amount of radioactivity in the sediment extract was 1.5% and 1.9% AR, then increased to a maximum of 5.9% (Day 15) and 13.8% (Day 15) AR, and then decreased to 3.0% and 4.8% AR at termination for the Tift and Wabasha samples, respectively. Non-extractable residues increased during the study, reaching a mean maximum of 13.4% for the Tift sample (Day 81) and 9.6% AR for the Wabasha sample (Day 61) before decreasing to 10.9 and 7.7% AR at Day 102, respectively. The amount of $^{14}CO_2$ collected increased with time in each system. Over the course of the study, the mean $^{14}CO_2$ collected from the systems was 39.2% and 64.8% AR for the Tift and Wabasha systems, respectively. No radioactivity was observed in the organic volatile traps.

In the water phase, oxamyl decreased from 67.4% AR and 80.0% AR at Day 0 to not detected % AR at Day 5 of incubation for the Tift and Wabasha systems, respectively. Significant metabolites observed in the water phase were IN-A2213, which reached a maximum concentration of 72.7% AR (Day 5) in the Tift system; IN-N0079, which reached maximum concentration of 59.6% AR (Day 1) in the Wabasha system; and IN-D2708, which reached maximum concentration of 29.7% AR (Day 61) in the Tift system. The total unassigned radioactivity was ≤4.9% AR in the water layers of both systems throughout the study.

In the sediment phase, the maximum levels of oxamyl were 0.6% AR (Day 0) and 0.2% AR (Day 0) for the Tift and Wabasha systems respectively. In the sediment phase of the Wabasha system, IN-SBY69 reached a mean maximum concentration of 9.9% (Day 61) before decreasing to 3.3% at Day 102. No other known or unknown metabolites occurred at >4.8% AR in the sediment for either system.

For the total water/sediment system, the amount of oxamyl in the Tift system generally decreased over the course of the study, from 68.0% AR at Day 0 to levels that were not detected at Day 5. The amount of oxamyl in the Wabasha system generally decreased over the course of the study, from 80.2% AR at Day 0 to levels that were not detected at Day 5.

The $DT_{50}$ values for oxamyl were calculated by the single first order model (SFO) and first order multi-compartment model (FOMC) using ModelMaker® 4.0. The best fit visually and statistically for the data from the total system and the water layer was obtained using the SFO model. The rate constants (kp), $DT_{50}$ values obtained for oxamyl dissipation in the water phase and overall degradation in the total system (SFO model) are summarized in the table below.

**Table 152 Summary of kinetic results for oxamyl degradation in anaerobic water/sediment systems**

| Water/ sediment type | Layer | Oxamyl SFO rate constant 1 ($day^{-1}$) | Oxamyl $DT_{50}$ (days) | Oxamyl $DT_{90}$ (days) | $r^2$ |
|---|---|---|---|---|---|
| Tift (Sandy Loam) | Water layer | 1.855 | 0.4 | 1.2 | 0.991 |
| | Total system | 2.671 | 0.3 | 0.9 | 0.989 |
| Wabasha (Loam) | Water layer | 1.546 | 0.4 | 1.5 | 0.983 |
| | Total system | 1.959 | 0.4 | 1.2 | 0.995 |

## I. MATERIALS AND METHODS

A. MATERIALS

1. Radiolabeled test material: [1-$^{14}$C]Oxamyl
   Lot/Batch #: 3609149
   Radiochemical purity: 98.7%
   Specific activity: 22.8 μCi/mg
   Stability of test compound: Not determined

2. Water/Sediment

Two freshly collected water/sediment systems were used, one from a river (Table , System 1), and the other from a pond (Table , System 2). The bulk water was observed to be free of debris greater than 2-mm and was therefore used unfiltered. The sediment was wet sieved through a 2-mm screen.

**Table 153Physiochemical parameters of the water/sediment systems**

| Sediment parameter | System 1 | | System 2 | |
|---|---|---|---|---|
| Geographic location | Tift County, Georgia, U.S.A. | | Wabasha County, Minnesota, U.S.A. | |
| Texture class (USDA) | Sandy Loam | | Loam | |
| % Sand (2000–50 μm)<br>% Silt (<50–2 μm)<br>% Clay (<2 μm) | 77<br>4<br>19 | | 39<br>50<br>11 | |
| pH (1:1 soil:water ratio) | 7.8 | | 6.2 | |
| Organic matter (Walkley Black) | 0.43% | | 6.3% | |
| Organic carbon[a] | 0.25 | | 3.7 | |
| Soil biomass Initial<br>Final | 15.7<br>31.3 | | 34.8<br>114.2 | |
| Cation exchange capacity (CEC) | 5.3 meq/100g | | 7.9 meq/100g | |
| Field moisture capacity at 0.33 bar | 11.2% | | 20.7% | |
| Field moisture capacity at 15 bar | 8.7% | | 13.2% | |
| Bulk density (disturbed) | 1.22 g/cc | | 0.80 g/cc | |
| Redox potential (Eh-mV) | Initial | Final | Initial | Final |
| | -216.4 | -521.6 | -211 | -269.5 |
| **Water parameter** | **System 1** | | **System 2** | |
| Temperature (°C) | 20 ± 2°C | | 20 ± 2°C | |
| pH | 7.9 | | 7.4 | |
| Hardness ($CaCO_3$) | 159 | | 33 | |
| Electrical conductivity(mmhos/cm) | 0.36 | | 0.15 | |
| Oxygen concentration (mg/L)[b] | 3.24 | | 3.75 | |
| Organic carbon Initial<br>Final | 5.6 ppm<br>3.6 ppm | | 22.8 ppm<br>26.1 ppm | |
| Redox potential (Eh-mV) | Initial | Final | Initial | Final |
| | 31.1 | 55.9 | -174.5 | 40.9 |

[a] % organic carbon = % organic matter/1.72

[b] at dosing

## B. STUDY DESIGN

### 1. Experimental conditions

The test bottles consisted of 250-mL (main study) or 1-L (biomass samples) Nalgene bottles that were incubated in a nitrogen flow-through system (positive-pressure) for the duration of the study. The study was performed in an environmental chamber set at 20°C.

For the main test samples, the corresponding replicates of a given system were placed in series (e.g., all replicate 1 bottles containing the Tift sediment treated with [1-$^{14}$C]oxamyl were placed in series). For the biomass samples, all bottles were placed in series. Humidified nitrogen was passed under positive pressure through the test vessels. Nitrogen leaving the vessels dosed with test substance was passed through a Supelco ORBO™-100 adsorbent tube to collect any organic volatiles, then through traps containing 1 N KOH to collect $^{14}CO_2$. Gas manifolds were used to provide a steady flow of nitrogen through the systems. Tygon® tubing was used for all connections. The biomass samples were assembled in the same manner as the test samples, but with no post-traps in series.

A total of 24 test bottles were prepared per sediment (ten for scheduled time points [in duplicate] and four spare test bottles for sampling as needed). These test bottles were prepared by transferring approximately 50 g dry weight of sediment to each bottle. A sufficient volume of the corresponding water was then added to the bottles to bring the total water content to 200 mL, which resulted in a 4:1 water:sediment ratio.

Six test bottles were prepared for biomass determination by adding approximately 175 g dry weight of sediment of the Tift and Wabasha sediments into each test bottle, and then adding a sufficient volume of water to bring the total water content to 492 or 476 mL (for the Tift and Wabasha sediments, respectively). These volumes resulted in similar sediment/water columns as the definitive test samples.

Control flasks were also prepared for each sediment system. These flasks were incubated under the same conditions as the main test, but were equipped with a dedicated redox probe that was permanently positioned in the sediment *via* a custom side arm. This set-up allowed accurate measurements of the redox potential in the sediment phase to be made at each sampling point without physical disruption of the layer.

Volatiles were collected from the main test system. Nitrogen exiting the test apparatus flowed through a Supelco ORBO™-100 adsorbent tube, then through two traps containing 1 N KOH for collection of any volatile organics and $^{14}CO_2$ generated from the breakdown of the radiolabeled test substance. A flow rate that sustained a gentle stream of bubbles through the entire trapping series was maintained. The samples, controls, and biomass samples were incubated at 20°C for nine days to achieve stabilized anaerobic conditions prior to application of the test substance.

2. Sampling

Main Study: Two samples for each sediment system were withdrawn immediately after treatment (Day 0), and at 0.25, 0.5, 1, 5, 15, 29, 61, 81, and 102 days after application. The sediment and water layers were separated by centrifugation. The water layers were analysed by LSC, and then filtered for HPLC analysis, while the sediment layers were extracted and prepared for analysis. The $CO_2$ volatile traps for each sample train were collected at the same time intervals (excluding Day 0), analysed for the presence of radioactive volatiles, and replenished. Since mass accountability was maintained throughout the study, the air sampling tubes for collecting organic volatiles were maintained on the system and not sampled.

Biomass Sediments: Neither $CO_2$ nor volatile organics were collected from systems intended for microbial viability measurements. Sediments were harvested for analysis at the beginning of the study and after termination of the main study samples (102 days).

3. Description of analytical procedures

The water and sediment were separated by centrifuge and decanting. Any water remaining in the sediment was thereafter treated as sediment. The water phase was analysed directly without concentration by LSC and reverse phase HPLC (Agilent, Phenomenex Aqua C18 [250 mm × 4.6 mm × 5 µm id]) eluted with a gradient of pH 3 water and acetonitrile.

After removal of the water phase, the sediment layer was extracted by shaking in 50:50 acetonitrile:water, then centrifuged to separate the extract. Extracts were analysed by LSC, and then concentrated, filtered, and analysed by reversed-phase HPLC. The base volatile traps were analysed by LSC, while the Orbo tubes were not further analysed because mass balance was maintained.

The limit of detection (LOD) for [1-$^{14}$C]oxamyl in the water phase and sediment extracts was 0.014% and 0.022% AR, respectively. For HPLC analyses, the LOD for [1-$^{14}$C]oxamyl in the water phase and concentrated sediment extracts was 0.230% and 0.030% AR, respectively. The limit of quantification (LOQ) for [1-$^{14}$C]oxamyl in the water phase and sediment extracts was 0.053% and 0.084% AR, respectively. For HPLC analyses, the LOQ for [1-$^{14}$C]oxamyl in the water phase and concentrated sediment extracts was 0.460% and 0.061% AR, respectively.

## II. RESULTS AND DISCUSSION

### A. MASS BALANCE

The mean material balance was 95.8 ± 3.9% and 95.9 ± 4.5% of the applied radioactivity (% AR) for the [1-$^{14}$C]oxamyl treated Tift and Wabasha samples, respectively. A summary of the recoveries and the distribution of the residues expressed as applied radioactivity at each sampling time interval is provided in Table 47 and Table 48.

B. FINDINGS

The radioactivity in the water layer decreased from 97.2% and 98.0% AR at Day 0 to a mean of 36.5% and 13.7% AR at Day 102 for the Tift and Wabasha samples, respectively.

The radioactivity in the sediment extracts increased from 1.5% and 1.9% AR at Day 0 to a maximum of 5.9% and 13.8% AR at Day 15, and then decreased to 3.0% and 4.8% AR at termination in the Tift and Wabasha [1-$^{14}$C]oxamyl treated systems, respectively.

As the amount of radioactivity in the water decreased, and the amount in the sediment extracts increased, the non-extractable residues increased over the course of the study, reaching a maximum of 13.4% AR at Day 81 and 9.4% AR at Day 61, and then decreased to 10.9% and 7.7% AR at termination in the Tift and Wabasha [1-$^{14}$C]oxamyl treated systems, respectively.

Volatiles as $^{14}CO_2$ increased over the course of the study, reaching a maximum of 39.2% AR at Day 102 and 64.8% AR at Day 102 in the Tift and Wabasha [1-$^{14}$C]oxamyl treated systems, respectively.

In the water layer of the Tift system, mean levels of oxamyl decreased from 67.4% AR at Day 0 to not detected at Day 5. Mean levels of IN-A2213 increased from 25.7% AR at Day 0 to 72.7% AR at Day 5, and then decreased to 14.5% AR at the end of 102 days of incubation. Mean levels of IN-N0079 increased from 2.8% AR at Day 0 to 11.4% AR at Day 1, and then decreased to 1.2% AR at the end of 102 days of incubation. Mean levels of IN-D2708 increased from 1.1% AR at Day 0 to 29.7% AR at Day 61, and then decreased to 17.5% AR at the end of 102 days of incubation. The total unassigned radioactivity was ≤ 3.3% AR throughout the study.

In the sediment layer of the Tift system, mean levels of oxamyl were ≤ 0.6% AR throughout the study. No metabolite occurred at >4.8% AR in the sediment phase. The total unassigned radioactivity was ≤0.3% AR throughout the study.

In the total system of the Tift system, levels of oxamyl decreased from 68.0% AR at Day 0 to not detected at Day 5. The total unassigned radioactivity was ≤3.3% AR throughout the study.

In the water layer of the Wabasha system, mean levels of oxamyl decreased from 80.0% AR at Day 0 to not detected at Day 5. Mean levels of IN-A2213 increased from 10.4% AR at Day 0 to 18.8% AR at Day 1, and then decreased to 0.0% AR at Day 61 to the end of 102 days of incubation. Mean levels of IN-N0079 increased from 6.2% AR at Day 0 to 59.6% AR at Day 1, and then decreased to 0.0% AR at Day 81 to the end of 102 days of incubation. Mean levels of IN-D2708 increased from 0.6% AR at Day 0 to 28.6% AR at Day 15, and then decreased to 13.7% AR at the end of 102 days of incubation. The total unassigned radioactivity was ≤ 4.9% AR throughout the study.

In the sediment layer of the Wabasha system, mean levels of oxamyl were ≤ 0.2% AR throughout the study. Mean levels of IN-SBY69 increased from 0.0% AR at Day 0 to 9.9% AR at Day 61, and then decreased to 3.3% AR at the end of 102 days of incubation. No other metabolite occurred at >3.3% AR in the sediment phase. The total unassigned radioactivity was ≤1.9% AR throughout the study.

In the total system of the Wabasha system, mean levels of oxamyl decreased from 80.2% AR at Day 0 to 0.0% AR at Day 5 to the end of 102 days of incubation. The total unassigned radioactivity was ≤5.5% AR throughout the study. The proposed degradation pathway for oxamyl in aerobic water/sediment system is shown Figure 7.

The degradation rate constants were determined for both aquatic systems by application of the best fit reaction kinetic model using ModelMaker 4.0. Oxamyl disappeared rapidly from water phase with calculated $DT_{50}$ values of 0.4 days and $DT_{90}$ values of 1.2 and 1.5 days in the sandy loam and loam systems, respectively (Table 49).

The degradation rates for the total systems were similar with calculated $DT_{50}$ values of 0.3 and 0.4 days and $DT_{90}$ values of 0.9 and 1.2 days in the sandy loam and loam systems, respectively (Table 49).

**Table 47 Biotransformation of [1-$^{14}$C]oxamyl, expressed as percentage of applied radioactivity[a], in the Tift loam system**

| Compound | Matrix | Sampling times (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **0.25** | **0.5** | **1** | **5** | **15** | **29** | **61** | **81** | **102** |
| Oxamyl (% AR) | Water | 67.4 | 40.3 | 29.1 | 8.3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | Sediment | 0.6 | 0.2 | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | **System** | **68.0** | **40.5** | **29.3** | **8.3** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** |
| IN-A2213 (% AR) | Water | 25.7 | 48.8 | 55.1 | 67.9 | 72.7 | 67.2 | 50.4 | 20.6 | 13.3 | 14.5 |
| | Sediment | 0.4 | 1.2 | 1.9 | 3.2 | 4.8 | 4.0 | 3.2 | 1.3 | 0.8 | 0.5 |
| | **System** | **26.0** | **50.0** | **57.0** | **71.1** | **77.5** | **71.2** | **53.6** | **21.9** | **14.1** | **15.0** |
| IN-N0079 (% AR) | Water | 2.8 | 6.4 | 10.3 | 11.4 | 7.0 | 4.4 | 3.6 | 1.9 | 1.7 | 1.2 |
| | Sediment | 0.5 | 0.5 | 0.6 | 0.6 | 0.4 | 0.6 | 0.6 | 0.4 | 0.0 | 0.1 |
| | **System** | **3.3** | **6.9** | **10.9** | **12.0** | **7.4** | **5.0** | **4.2** | **2.2** | **1.8** | **1.4** |
| IN-D2708 (% AR) | Water | 1.1 | 2.0 | 1.8 | 2.6 | 8.2 | 11.4 | 15.7 | 29.7 | 29.0 | 17.5 |
| | Sediment | 0.0 | 0.1 | 0.1 | 0.3 | 0.5 | 1.0 | 1.2 | 2.2 | 2.7 | 2.4 |
| | **System** | **1.1** | **2.0** | **1.9** | **2.9** | **8.8** | **12.3** | **16.9** | **32.0** | **31.7** | **19.9** |
| IN-SBY69 (% AR) | Water | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | Sediment | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | **System** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** |
| Total unassigned | Water | 0.3 | 0.6 | 0.0 | 2.7 | 3.2 | 0.6 | 0.9 | 1.7 | 0.7 | 3.3 |
| | Sediment | 0.0 | 0.0 | 0.0 | 0.0 | 0.1 | 0.3 | 0.3 | 0.3 | 0.1 | 0.0 |
| | **System** | **0.3** | **0.7** | **0.0** | **2.7** | **3.3** | **0.9** | **1.1** | **2.1** | **0.8** | **3.3** |
| Total extractable residue | Water | 97.2 | 98.0 | 96.4 | 92.9 | 91.2 | 83.6 | 70.5 | 53.9 | 44.8 | 36.5 |
| | Sediment | 1.5 | 2.2 | 2.8 | 4.2 | 5.8 | 5.9 | 5.3 | 4.2 | 3.6 | 3.0 |
| | **System** | **98.8** | **100.3** | **99.2** | **97.1** | **96.9** | **89.5** | **75.8** | **58.2** | **48.4** | **39.6** |
| Non-extractable residues | Rep. 1 | 0.1 | 0.0 | 0.3 | 0.5 | 2.5 | 5.6 | 8.6 | 11.6 | 13.3 | 10.7 |
| | Rep. 2 | 0.1 | 0.1 | 0.3 | 0.4 | 3.9 | 5.2 | 9.6 | 13.1 | 13.6 | 11.1 |
| | **Mean** | **0.1** | **0.1** | **0.3** | **0.4** | **3.2** | **5.4** | **9.1** | **12.4** | **13.4** | **10.9** |
| $CO_2$ | Rep. 1 | N/A | 0.0 | 0.0 | 0.0 | 0.2 | 1.5 | 10.3 | 23.1 | 33.4 | 42.5 |
| | Rep. 2 | N/A | 0.0 | 0.0 | 0.0 | 0.1 | 1.4 | 5.7 | 18.8 | 30.0 | 35.9 |
| | **Mean** | **N/A** | **0.0** | **0.0** | **0.0** | **0.2** | **1.4** | **8.0** | **21.0** | **31.7** | **39.2** |
| Total mass balance | Rep. 1 | 97.5 | 0.0 | 99.2 | 98.1 | 99.8 | 94.1 | 92.4 | 91.3 | 95.5 | 89.8 |
| | Rep. 2 | 100.3 | 100.3 | 99.8 | 96.9 | 100.7 | 98.6 | 93.5 | 91.7 | 91.5 | 89.5 |
| | **Mean** | **98.9** | **100.3** | **99.5** | **97.5** | **100.3** | **96.4** | **93.0** | **91.5** | **93.5** | **89.7** |
| | | | | | | | | | | **Overall Average** | 95.8 |
| | | | | | | | | | | **Standard Deviation** | 3.9 |

[a] Biodegradation figures are the average of two replicates.

**Table 48 Biotransformation of [1-$^{14}$C]oxamyl, expressed as percent of applied radioactivity[a], in the Wabasha water/sediment system**

| Compound | Matrix | Sampling times (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **0** | **0.25** | **0.5** | **1** | **5** | **15** | **29** | **61** | **81** | **102** |
| Oxamyl (% AR) | Water | 80.0 | 59.9 | 39.5 | 12.6 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | Sediment | 0.2 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | **System** | **80.2** | **60.0** | **39.6** | **12.6** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** | **0.0** |
| IN-A2213 (% AR) | Water | 10.4 | 11.8 | 15.2 | 18.8 | 13.4 | 7.5 | 1.7 | 0.0 | 0.0 | 0.0 |
| | Sediment | 0.2 | 0.5 | 0.3 | 1.1 | 0.8 | 0.4 | 0.1 | 0.0 | 0.0 | 0.0 |
| | **System** | **10.6** | **12.3** | **15.5** | **19.8** | **14.2** | **7.9** | **1.8** | **0.0** | **0.0** | **0.0** |
| IN-N0079 (% AR) | Water | 6.2 | 23.6 | 39.1 | 59.6 | 45.3 | 20.8 | 7.5 | 1.6 | 0.0 | 0.0 |
| | Sediment | 1.5 | 2.2 | 2.4 | 3.3 | 3.1 | 1.8 | 0.6 | 0.0 | 0.0 | 0.0 |
| | **System** | **7.7** | **25.8** | **41.5** | **62.9** | **48.4** | **22.6** | **8.1** | **1.6** | **0.0** | **0.0** |
| IN-D2708 (% AR) | Water | 0.6 | 0.8 | 1.5 | 3.3 | 16.1 | 28.6 | 22.2 | 11.5 | 16.6 | 13.7 |
| | Sediment | 0.0 | 0.1 | 0.3 | 0.5 | 1.8 | 2.3 | 1.8 | 1.0 | 1.4 | 1.5 |
| | **System** | **0.6** | **0.9** | **1.8** | **3.7** | **17.9** | **30.9** | **23.9** | **12.5** | **18.1** | **15.1** |
| IN-SBY69 (% AR) | Water | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | Sediment | 0.0 | 0.1 | 0.1 | 0.5 | 4.5 | 8.0 | 9.3 | 9.9 | 6.6 | 3.3 |
| | **System** | **0.0** | **0.1** | **0.1** | **0.5** | **4.5** | **8.0** | **9.3** | **9.9** | **6.6** | **3.3** |
| Total Unassigned | Water | 0.8 | 0.5 | 0.0 | 1.6 | 4.9 | 0.3 | 1.7 | 0.0 | 0.1 | 0.0 |
| | Sediment | 0.0 | 0.1 | 0.3 | 0.1 | 0.6 | 1.3 | 1.9 | 1.0 | 0.5 | 0.0 |
| | **System** | **0.8** | **0.6** | **0.3** | **1.7** | **5.5** | **1.6** | **3.6** | **1.1** | **0.6** | **0.0** |
| Total Extractable Residue | Water | 98.0 | 96.5 | 95.4 | 95.8 | 79.7 | 57.2 | 33.0 | 13.1 | 16.8 | 13.7 |
| | Sediment | 1.9 | 3.2 | 3.5 | 5.5 | 10.8 | 13.8 | 13.7 | 12.5 | 8.5 | 4.8 |
| | **System** | **100.0** | **99.7** | **98.9** | **101.4** | **90.5** | **70.9** | **52.8** | **33.5** | **25.3** | **18.4** |
| Non-Extractable Residues | Rep. 1 | 0.2 | 0.5 | 0.6 | 0.9 | 2.4 | 4.9 | 6.8 | 7.5 | 8.2 | 7.8 |
| | Rep. 2 | 0.3 | 0.4 | 0.5 | 0.9 | 2.1 | 5.4 | 6.6 | 11.4 | 7.8 | 7.5 |
| | **Mean** | **0.2** | **0.5** | **0.6** | **0.9** | **2.3** | **5.2** | **6.7** | **9.4** | **8.0** | **7.7** |
| $CO_2$ | Rep. 1 | N/A | 0.0 | 0.0 | 0.1 | 2.0 | 14.6 | 32.2 | 47.2 | 61.6 | 65.1 |
| | Rep. 2 | N/A | 0.0 | 0.0 | 0.1 | 1.8 | 20.8 | 33.7 | 47.1 | 61.4 | 64.4 |
| | **Mean** | **N/A** | **0.0** | **0.0** | **0.1** | **1.9** | **17.7** | **33.0** | **47.1** | **61.5** | **64.8** |
| Total Mass Balance | Rep. 1 | 100.4 | 101.0 | 99.8 | 100.6 | 95.9 | 97.5 | 92.8 | 88.7 | 96.2 | 91.3 |
| | Rep. 2 | 100.0 | 99.3 | 99.2 | 104.1 | 93.5 | 90.0 | 92.1 | 91.3 | 93.4 | 90.4 |
| | **Mean** | **100.2** | **100.1** | **99.5** | **102.3** | **94.7** | **93.8** | **92.4** | **90.0** | **94.8** | **90.9** |
| | | | | | | | | | | **Overall Average** | 95.9 |
| | | | | | | | | | | **Standard Deviation** | 4.5 |

[a] Biodegradation figures are the average of two replicates.

**Table 49 $T_{50}$ and $DT_{90}$ values for oxamyl and degradation products in anaerobic aquatic systems**

| Sediment type | Layer | Kinetic model | Optimized parameters ± standard error | χ2 error | $r^2$ | $DT_{50}$ (days) | $DT_{90}$ (days) |
|---|---|---|---|---|---|---|---|
| Tift (Sandy Loam) (ABC 519) | Water Layer | SFO | M0 = 67.4 ± 1.9<br>k ($d^{-1}$) = 1.855 ± 0.118 | 5 | 0.991 | 0.4 | 1.2 |
| | | FOMC | M0 = 67.4 ± 2.1<br>Alpha = 526.16 ± 9105.2<br>Beta = 283.34 ± 4904.2 | 6 | 0.991 | 0.4 | 1.2 |
| | Total System | SFO | M0 = 97.6 ± 3.1<br>k ($d^{-1}$) = 2.671 ± 0.2 | 11 | 0.989 | 0.3 | 0.9 |
| | | FOMC | M0 = 97.6 ± 3.4<br>Alpha = 184.67 ± 5288.4<br>Beta = 68.839 ± 1974.9 | 12 | 0.989 | 0.3 | 0.9 |
| Wabasha (Loam) (ABC 520) | Water Layer | SFO | M0 = 82.3 ± 2.8<br>k ($d^{-1}$) = 1.546 ± 0.125 | 6 | 0.983 | 0.4 | 1.5 |
| | | FOMC | M0 = 82.3 ± 3.4<br>Alpha = 554.99 ± 6585.9<br>Beta = 358.27 ± 4244.7 | 7 | 0.983 | 0.4 | 1.5 |
| | Total System | SFO | M0 = 100 ± 1.9<br>k ($D^{-1}$) = 1.959 ± 0.086 | 2 | 0.995 | 0.4 | 1.2 |
| | | FOMC | M0 = 100 ± 2.2<br>Alpha = 1253.7 ± 10714<br>Beta = 639.77 ± 5464.8 | 3 | 0.995 | 0.4 | 1.2 |

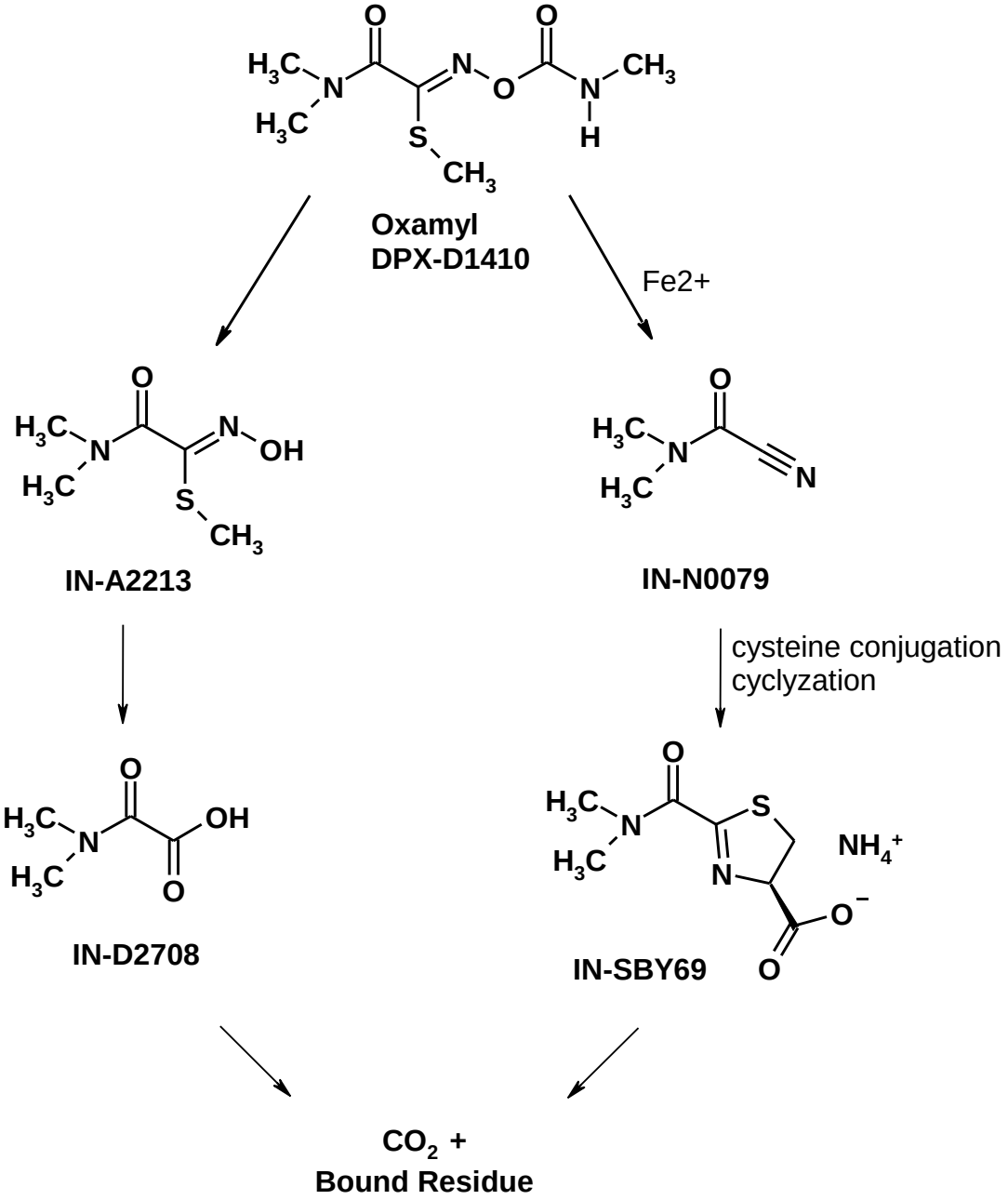
## III. CONCLUSION

The biotransformation of [1-$^{14}$C]-radiolabeled oxamyl was studied in two water/sediment systems under anaerobic conditions. Oxamyl was observed to degrade rapidly in the water phase. Dissipation of oxamyl from the water phase was fast, with a $DT_{50}$ value of 0.4 days in the Tift and Wabasha systems. Overall degradation from the total system was similarly fast, with a $DT_{50}$ value of 0.3 to 0.4 days in the Tift and Wabasha systems, respectively. Oxamyl degraded into four major metabolites, IN-A2213, IN-N0079, IN-D2708, and IN-SBY69; non-extractable residues (NER); and $CO_2$.

A proposed degradation pathway in water-sediment systems is outlined in Figure 7.

(Clark, B., 2013)

**Figure 7 Proposed metabolic pathway of [1-$^{14}$C]oxamyl in water-sediment systems**




The anaerobic water/sediment study DuPont-34157, submitted for the first time in this submission and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline OECD 308 (April 2002), OPPTS 835.4400 (June 2008), SETAC Europe (1995). A review of this study indicates that it fully meets the current guideline (OECD 308) and is relied upon.

#### B.8.2.2.4 Irradiated water/sediment study

An irradiated water/sediment study was not run with oxamyl. This higher tier study was not required and would not provide additional information above what has been demonstrated in the aqueous photolysis study and the water/sediment study.

#### B.8.2.2.5 Overall assessment: Degradation in aquatic systems

Chemical routes of oxamyl degradation in water were investigated in hydrolysis and photolysis studies. Hydrolysis was base-catalysed (E1cb elimination reaction). Oxamyl was shown to be hydrolytically stable at acidic pH 4. The rate of degradation was temperature dependent and increased with increasing temperature with $DT_{50}$ values of 21.1, 9.01, and 4.16 days at 20, 25, and 30°C for pH 7, respectively, and 0.200, 0.098, and 0.046 days at 20, 25, and 30°C for pH 9, respectively. The only product observed was IN-A2213, and this was stable to further hydrolysis. Hydrolysis studies for the degradates IN-A2213, IN-D2708, IN-N0079, and IN-T2921 at 20°C were also conducted. IN-A2213, IN-D2708, and IN-T2921 were stable ($DT_{50}$ >30–34 days) at pH 4, pH 7, and pH 9. IN-N0079 was observed to hydrolyse at pH 9 and pH 7, and the $DT_{50}$ values were 3 days and 136 days, respectively. IN-T2921 and IN-D2708 were the observed hydrolysis products from IN-N0079. IN-N0079 was stable to hydrolysis ($DT_{50}$ >30 days) at pH 4.

The photolysis of oxamyl was investigated at a hydrolytically stable pH (pH 5) using an artificial light source with wavelength cut-off of <295 nm to simulate natural sunlight. Oxamyl was degraded by photolysis to a significant extent following exposure to artificial light with a first order $DT_{50}$ of 3.5 days of irradiation corresponding to environmental half-lives estimated as 4.1, 5.8, 6.3, 7.9, or 8.7 mid-summer days in Phoenix, Arizona (USA, 33.3 °N); Edmonton, Alberta (Canada, 53.3 °N); Athens, Greece (EU, 38.0 °N); London, Great Britain (EU, 51.3 °N); and Tokyo (Japan, 35.1 °N), respectively. Photolytic degradation leads primarily to IN-N0079, which comprised a maximum of 67.6% of the applied amount at study termination. Only minor levels of IN-A2213 were observed (max 1.6%). Since oxamyl does not absorb light above 290 nm, a quantum yield could not be calculated and is thus zero. It is believed that the rapid photolytic degradation observed in the new 2014 aqueous photolysis study is attributed to enhancement of the ferrous iron ($Fe^{2+}$) reduction of oxamyl to IN-N0079 by the presence of light. This reaction has been well documented both in the public literature (see position paper DuPont-14826, Point B.8.2.3 in this document) and in the saturated zone degradation studies (DuPont-2635, Point B.8.2.3 in this document). This reaction is favoured under anoxic (reductive) conditions, and the use of sealed test vessels of minimal headspace would have promoted such reductive conditions. Aqueous photolysis studies for the major degradates IN-A2213, IN-D2708, IN-N0079, and IN-T2921 were not conducted, since their molar absorptivities at 290 nm were experimentally determined to be negligible.

The behaviour of oxamyl in natural aerobic surface waters was investigated in the mineralization in surface water study. Oxamyl undergoes limited mineralization in natural surface water, but is rapidly degraded *via* hydrolysis to form IN-A2213.

The fate of oxamyl was investigated in two natural water/sediment systems. Two differing application techniques were investigated. Firstly, addition of compound to the water phase (reflecting the more likely method of entry into natural water systems) and secondly, vigorous mixing of water and sediment after application of compound. The second method unsurprisingly resulted in a greater initial association of radioactivity with the sediment and much higher initial amounts of IN-N0079. The rapid appearance of IN-N0079 after vigorously mixing the water and anaerobic sediment is likely due to oxamyl reduction by ferrous iron ($Fe^{2+}$). The relevance of the vigorous mixing approach of dosing is likely to be low in natural conditions, and the effect of mixing aerobic and anaerobic phases is difficult to predict. Therefore, the results obtained from this second application method are not considered further.

When dosed to the surface of the water phase (in accordance with the current OECD guideline), oxamyl degraded rapidly in the water phase (<50% remaining on day 1), and significant amounts were never found in the sediment phase. Since both water systems were slightly alkaline, it is likely that a component of this rapid degradation was the result of chemical hydrolysis. IN-A2213, the hydrolysis product, reached a maximum of 25.3–48.8% AR in the water phase on Day 2 and then decreased to non-detectable levels by Day 61. Low levels were found in the sediments (max. 4.4%), and the pattern reflected that observed in the water phase. In one system, high levels of IN-N0079 (maximum 52.9% at Day 2) were observed in the water phase, and the pattern of formation and decline was parallel to that of IN-A2213. As discussed above, the rapid appearance of significant amounts of IN-N0079 was likely due to the $Fe^{+2}$-oxamyl reduction reaction with $Fe^{+2}$ near or within the anaerobic sediment phase. Following the decline of the IN-A2213 and IN-N0079, levels of IN-D2708 in the water subsequently rose (maximum 64.2–66.8% at Day 30) and then declined during the remainder of the study. Low levels of IN-D2708 (maximum 10.4% to 12.1%) were observed in the sediment, and the pattern of occurrence of this sediment residue coincided with the maximum level of IN-D2708 in the water phase, suggesting a simple gradient diffusion of IN-D2708 from the water phase into the sediment pore water in the static test system. In one system only, levels of IN-T2921 exceeded 10% in the water phase. However, this was only at one time point (Day 14), the maximum level was only 11.4%, and subsequent degradation was rapid (not

detected at the next time point, Day 30). Amounts of carbon dioxide reached 27.9 to 60.9% by the end of the study. In anaerobic water/sediment systems, oxamyl degraded similarly as in the aerobic system, with IN-A2213, IN-N0079, and IN-D2708 being the major transformation products. In one sediment system under anaerobic conditions, IN-SBY69 was observed as an additional transient metabolite. This metabolite is formed *via* cysteine conjugate cyclyzation of IN-N0079. It is believed that IN-SBY69 was observed in the anaerobic water/sediment study only because the anaerobic conditions create a reductive environment that favours the $Fe^{+2}$-oxamyl reduction reaction that produces its precursor, IN-N0079. Thus, this metabolite (IN-SBY69) was only present at major, observable levels, because of the drastically increased levels of IN-N0079 formed in this one anaerobic sediment system. In typical water/sediment systems, such high levels of IN-N0079 are not expected to form, and thus only minor, if any, levels of IN-SBY69 can be expected to occur in the environment.

Overall, the water/sediment studies show degradation of oxamyl to IN-A2213 or IN-N0079 and further to IN-T2921 and IN-D2708 in the water phase. No compounds reached consistently significant levels in the sediment. Carbon dioxide was the ultimate degradation product in both test systems. The kinetics presented in this original report are superseded by the updated FOCUS kinetic results derived in the modelling position paper DuPont-44046 EU (see summary under Point B.8.2.2.5 in this document) Based on this re-evaluation, the total system persistence $DT_{50}$s for oxamyl and IN-A2213 were 0.69–0.82 days, and 5.67–8.24 days, respectively. The results of all FOCUS modelling for the water/sediment study can be found in Table 50 and **Errore. L'origine riferimento non è stata trovata.**.

A ready biodegradability study demonstrated that oxamyl, dosed at 26.1 mg/L (10 mg carbon/L), did not have an inhibitory effect on activated sewage sludge bacteria. Biodegradation was measured *via* carbon dioxide evolution, and 19% of the theoretical maximum was reached by the end of the study on Day 29. This result is similar to the Day 30 carbon dioxide level (17.1%) observed in the high organic matter pond water/sediment system. While substantial mineralisation was observed, oxamyl cannot be classified as readily biodegradable under the strict terms of the guideline method.

**Table 50 Summary of water/sediment study persistence endpoints for oxamyl**

| System | Water/sediment system | Values in days | Kinetic level and type |
|---|---|---|---|
| Red Oak Stream | System | $DegT_{50}$ = 0.82<br>$DegT_{90}$ = 8.31 | P-I; HS Best-fit Model |
| | Water | $DT_{50}$ = 0.82<br>$DT_{90}$ = 8.31 | P-I; HS Best-fit Model |
| | Sediment | - | Oxamyl appeared only in small amounts at only 2 data points |
| Town Park Pond | System | $DegT_{50}$ = 0.69<br>$DegT_{90}$ = 2.28 | P-I; SFO Best-fit Model |
| | Water | $DT_{50}$ = 0.69<br>$DT_{90}$ = 2.28 | P-I; SFO Best-fit Model |
| | Sediment | - | Oxamyl did not appear in sediment |

**Table 158 Summary of water/sediment study persistence endpoints for IN-A2213**

| System | Water/sediment system | Values in days | Best fit model | Type of endpoint and comments |
|---|---|---|---|---|
| Red Oak Stream | System | $DegT_{50}$ = 8.24<br>$DegT_{90}$ = 27.38 | HS-SFO | System degradation endpoint |
| | Water | $DT_{50}$ = 14.16<br>$DT_{90}$ = 47.05 | SFO | Water decline endpoint |
| | Sediment | $DT_{50}$ = 11.62<br>$DT_{90}$ = 38.61 | SFO | Sediment decline endpoint |
| Town Park Pond | System | $DegT_{50}$ = 5.67<br>$DegT_{90}$ = 18.84 | SFO-SFO | System degradation endpoint |
| | Water | $DT_{50}$ = 6.50<br>$DT_{90}$ = 21.58 | SFO | Water decline endpoint |
| | Sediment | $DT_{50}$ = 5.15<br>$DT_{90}$ = 28.10 | HS | Sediment decline endpoint |

### B.8.2.3 Degradation in the saturated zone

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.2.3/01**

| **Reference:** | **Report:** | Dean, G.M. (2000); Route and rate of oxamyl degradation in saturated subsoils<br><br>**DuPont Report No.:** DuPont-2635<br><br>**Guidelines:** Dutch Environmental Criterial (1995) |
|---|---|---|
| -- | | |

1. Test material: [1-$^{14}$C]oxamyl
   Lot/Batch #: 2868-229
   Purity: Radiochemical purity - >96%

**Materials and Methods:**

The route and rate of degradation of [1- $^{14}$C]-oxamyl (18.65 µCi/mg, radiochemical purity >96%) were studied in two anaerobic subsoils (from Hooghalen and Emmer Compascuum) and two aerobic subsoils (from Roswinkel and Eeserveen) (Table ). Subsoils were sampled from a shallow saturated zone of an aquifer under agricultural fields that were representative of potato production in The Netherlands. Significant precautions were taken to ensure the collected samples maintained their chemical state as close to initial field conditions as feasible (using pH, redox as indicators).

**Table 159 Saturated zone subsoil characteristics**

| Soil name or designation | Hooghalen | Emmer Compascuum | Roswinkel | Eeserveen |
|---|---|---|---|---|
| Particle size distribution (%)<br>ISO classification | | | | |
| Clay (<2 μm) | 6.56 | 2.10 | 2.76 | 0.32 |
| Silt (2 - 63 μm) | 15.70 | 2.77 | 2.85 | 1.94 |
| Sand (63 μm - 2 mm) | 77.74 | 95.13 | 94.39 | 97.74 |
| Textural class | loamy sand | sand | sand | sand |
| Organic carbon (%) | 4.9 | 0.1 | 0.1 | <0.05 |
| Organic matter (%) | 8.4 | 0.2 | 0.2 | <0.1 |
| Subsoil pH (1 : 5 v/v in water) | 5.2 | 5.4 | 6.4 | 5.3 |
| Subsoil pH (1 : 5 v/v in 0.01 M $CaCl_2$) | 4.1 | 4.4 | 5.9 | 4.5 |
| Cation exchange capacity (mEq/100 g) | 17.8 | 2.2 | 1.7 | 0.6 |
| Ammonium-$N_{KCl\ extractable}$ (mg/kg) | 30.4 | 4.3 | 0.7 | 0.6 |
| Nitrate-$N_{KCl\ extractable}$ (mg/kg) | 0.8 | 0.7 | 2.5 | 9.2 |
| $Sulphate_{1\ :\ 5\ subsoil\ :\ water\ extract}$ (mg/kg) | 153.7 | 56.9 | 75.6 | 35.9 |
| $Phosphorus_{extractable}$ (mg/kg) | 3.3 | 10.0 | 7.5 | 1.1 |
| Iron $_{aqua\ regia\ extractable}$ (mg/kg) | 3463.8 | 2079.9 | 2064.0 | 2183.3 |
| Groundwater ferrous $iron_{ferrozine\ meth.}$ (μg/mL) | 4.9 | 5.3 | 0.07 | 0.78 |
| Dry matter content (%) | 47 | 85 | 84 | 81 |
| Water content $_{dry\ weight\ basis}$ (%) | 114 | 17 | 19 | 23 |
| Biomass $_{fumigation\ extraction}$ (μg C/g) | 92.6 | 29.2 | 40.8 | 18.8 |

Saturated subsoils were treated with [1-$^{14}$C]-oxamyl at a rate of 0.5 mg/kg dry weight (approximately equivalent to a concentration in groundwater of 0.4 to 2.4 mg/L, based on the water contents of the saturated soils), and incubated in the dark at 10 ±2 °C for up to 120 days. This application rate was considered necessary to enable the detection and quantification of oxamyl and its degradation products down to at least 1% of the applied radioactivity.

At various time intervals, subsoil samples were exhaustively extracted with aqueous/organic solvents and the total amounts of extractable oxamyl and its degradation products were separated and quantified by chromatography. Volatile radiolabelled degradates were trapped and quantified using a nitrogen (anaerobic) or air (aerobic) flow-through system. Non- extractable radiolabelled residues were also quantified.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Findings:**

The redox potential and pH of each subsoil were monitored throughout the study and showed that subsoil conditions remained similar to those measured in the field. The material balance in all subsoils was in the range 90.1-102% of applied radioactivity, with the exception of two samples where the amount extracted on day 14 and day 46 was considered to be anomalous (Table  and Table 161). In the anaerobic subsoils, small quantities of labelled carbon dioxide and organic volatiles were produced in selected samples. In aerobic Roswinkel subsoil, a total mean of 7.4% AR was mineralised to carbon dioxide by day 120. In Eeserveen aerobic subsoil, no volatile radioactivity or carbon dioxide was detected.

In anaerobic subsoils, IN-N0079, IN-T2921 and IN-D2708 were the major (>10% AR) degradates consistently observed. IN-N0079 declined from an average of 71.3% AR on day 0 to an average of 15% AR on day 60 in Hooghalen subsoil, and from an average of 59% AR on day 0 to an average of 18% AR on day 60 in Emmer Compascuum subsoil. IN-T2921 increased from an average of 3.5% AR on day 0 to an average of 10% AR on day 60 and from an average of 2.1% AR on day 0 to an average of 12% AR on day 60 for Hooghalen and Emmer Compascuum subsoils respectively. IN-D2708 increased from an average of 14% AR on day 0 to an average of 33% AR on day 60 and from an average of 1.5% AR on day 0 to an average of 48% AR on day 60 for Hooghalen and Emmer Compascuum subsoils respectively.

A transient product, IN-M2583, was observed only in Hooghalen subsoil and reached a maximum average level of 13.2% AR on day 1, but declined rapidly to 7.4% AR by the next sampling time on day 4. Another transient product, designated D2, reached 13.6% AR on day 0 in one replicate, but dropped to low or non-detectable levels by the day-4 sampling.

In aerobic subsoils, IN-A2213 was the only major (>10% AR) degradate, reaching an average maximum level of 68.8% AR in Roswinkel subsoil by day 120. In Eeserveen saturated subsoil, no significant degradation of oxamyl was observed over the 120-day incubation period.

Non-extractable radioactivity accounted for a mean maximum of 32.2% AR and 11.0% AR in Hooghalen and Emmer Compascuum saturated anaerobic subsoils after 60 days incubation, and 4.5% AR and 7.2% AR in Roswinkel and Eeserveen saturated aerobic subsoils after 60 days incubation, respectively.

Oxamyl was rapidly degraded in both anaerobic saturated subsoils, with an estimated DT90 of <6 hours. The major product present in the soil at 6 hours was IN-N0079, which was itself further degraded, with DT50 and DT90 values (calculated by assuming linear simple first order kinetics) ranging from 28 to 32 days and 94 to 107 days, respectively (Table ).

In aerobic subsoil at >pH 5.9 (Roswinkel), oxamyl was degraded to IN-A2213 with DT50 and DT90 values of 37 and 122 days, respectively. By contrast, oxamyl was not measurably degraded at <pH 5 after 120 days in aerobic subsoil (Eeserveen).

The major degradation pathway for oxamyl is reduction to IN-N0079 under anaerobic conditions, and hydrolysis to IN-A2213 under aerobic conditions (Figure 8.4.4.1-1 and Figure 8.4.4.1-2).

**Table 160 Percentage distribution of radiolabelled components in saturated anaerobic subsoils after application of [$^{14}$C]-oxamyl**

| Subsoil | Time after dosing | IN-D2708 | IN-T2921 | D2 | IN-N0079 | Oxamyl | IN-M2583 | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Hooghalen | 0 | 14.2 | 4.0 | nd | 71.0 | n | 5.7 | - | - | 3.2 | 98.0 |
| | 0 | 13.0 | 3.0 | nd | 71.5 | d | 6.9 | - | - | 3.5 | 97.9 |
| | 0.25 | 6.9 | 12.7 | 2. | 54.8 | n | 11.5 | nd | nd | 9.4 | 98.1 |
| | 0.25 | 7.7 | 9.0 | 8 | 54.7 | d | 10.9 | nd | nd | 9.1 | 97.4 |
| | 0.5 | 9.4 | 13.1 | 4.2 | 52.0 | n | 11.3 | nd | nd | 7.1 | 93.0 |
| | 0.5 | 7.4 | 10.6 | nd | 53.5 | d | 12.9 | nd | nd | 7.8 | 96.8 |
| | 1 | 11.5 | 14.2 | 1. | 50.4 | n | 12.5 | nd | nd | 4.9 | 93.4 |
| | 1 | 9.4 | 14.6 | 3 | 46.8 | d | 13.9 | nd | 0. | 5.7 | 92.3 |
| | 4 | 17.4 | 14.3 | nd | 44.5 | n | 8.7 | 0. | 3 | 9.3 | 94.7 |
| | 4 | 21.5 | 13.2 | 1. | 48.0 | d | 6.4 | 5 | nd | 9.6 | 100 |
| | 8 | 17.0 | 14.9 | 6 | 45.4 | n | 7.9 | nd | nd | 12.2 | 97.3 |
| | 8 | 21.9 | 13.4 | nd | 39.1 | d | 4.9 | nd | nd | 12.4 | 94.6 |
| | 14 | 24.7 | 11.6 | 1. | 41.4 | n | 2.0 | nd | 1. | 16.3 | 96.0 |
| | 14 | nc | nc | 5 | nc | d | nc | nd | 2 | nc | nc |
| | 29 | 16. | 13. | nd | 20. | n | 6. | nc | nd | 26. | 90. |
| | 29 | 9 | 7 | 1. | 7 | d | 4 | nd | nc | 2 | 2 |
| | 46 | 28.4 | 9.1 | 7 | 27.7 | n | nd | 0. | nd | 28.4 | 94.2 |
| | 46 | 35.4 | 3.2 | nd | 24.1 | d | 2. | 6 | nd | 26.1 | 92.8 |
| | 60 | 28.3 | 8.5 | nc | 26.5 | n | 1 | 1.9 | nd | 28.3 | 92.4 |
| | 60 | 30.8 | 9.0 | 2. | 20.6 | d | n | nd | 0. | 31.9 | 95.4 |
| Emmer Compascuum | 0 | 0.9 | 2.8 | 13.6 | 61.7 | 22.0 | nd | - | - | 0.6 | 102 |
| | 0 | 2.0 | 1.3 | 6.1 | 56.7 | 31.6 | nd | - | - | 0.6 | 99.2 |
| | 0.25 | 7.1 | 11.8 | 9.1 | 64.8 | 4.0 | nd | nd | nd | 2.3 | 99.0 |
| | 0.25 | 9.3 | 9.1 | 8.4 | 65.5 | 1.9 | nd | nd | nd | 1.1 | 95.4 |
| | 0.5 | 7.1 | 12.9 | 8.7 | 66.6 | nd | nd | nd | nd | 1.6 | 97.8 |
| | 0.5 | 5.7 | 16.8 | 5.8 | 68.6 | 0.8 | nd | nd | nd | 1.6 | 99.3 |
| | 1 | 11.8 | 17.6 | 1.9 | 67.1 | nd | nd | nd | nd | 1.2 | 99.6 |
| | 1 | 8.6 | 18.3 | 2.2 | 66.9 | 0.9 | nd | nd | nd | 1.3 | 100 |
| | 4 | 16.8 | 19.6 | nd | 58.5 | nd | nd | nd | nd | 2.0 | 96.9 |
| | 4 | 16.0 | 19.4 | nd | 62.0 | nd | nd | nd | nd | 1.7 | 99.0 |
| | 8 | 26.9 | 18.3 | nd | 47.4 | nd | nd | nd | nd | 1.9 | 95.5 |
| | 8 | 16.0 | 17.6 | 1.5 | 57.9 | nd | nd | 0.3 | nd | 0.8 | 95.9 |
| | 14 | 25.0 | 12.9 | nd | 44.1 | 1.2 | nd | nd | 1.0 | 6.4 | 90.6 |
| | 14 | 23.4 | 11.8 | nd | 53.2 | nd | nd | nd | nd | 5.0 | 94.2 |
| | 29 | 35.9 | 19.0 | nd | 29.6 | nd | nd | nd | nd | 5.6 | 90.1 |
| | 29 | 27.1 | 12.0 | nd | 43.5 | nd | nd | nd | nd | 6.0 | 90.2 |
| | 46 | nc | nc | nc | nc | nc | nc | nc | nc | nc | nc |
| | 46 | 46.5 | 19.6 | nd | 14.5 | nd | nd | 0.6 | nd | 8.9 | 91.6 |
| | 60 | 44.6 | 10.6 | nd | 22.3 | nd | nd | 0.3 | 1.2 | 11.2 | 91.5 |
| | 60 | 51.2 | 13.6 | nd | 13.4 | nd | nd | 0.7 | nd | 10.7 | 92.4 |

nd = not detected (limit of detection was equivalent to 0.3-2.4% AR), nc = not calculated (vessel recovery anomalous), NER = non-extractable residue

Total applied radioactivity calculated as the sum of extractable, non-extractable, and volatile radioactivity (not the sum of preceding columns).

**Table 161Percentage distribution of radiolabelled components in saturated aerobic subsoils after application of [$^{14}$C]-oxamyl**

| Subsoil | Time after dosing (days) | Oxamyl | IN-A2213 | IN-N0079 | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|---|---|
| Roswinkel | 0 | 99 | nd | 1.0 | - | - | <0.4 | 100 |
| | 0 | 101 | nd | 0.8 | - | - | <0.4 | 102 |
| | 0.25 | 98.4 | nd | 0.6 | nd | nd | <0.4 | 99.0 |
| | 0.25 | 96.2 | nd | 0.8 | nd | nd | <0.4 | 97.0 |
| | 0.5 | 95.6 | nd | nd | nd | nd | <0.4 | 95.6 |
| | 0.5 | 95.3 | nd | 0.6 | nd | nd | <0.4 | 95.9 |
| | 1 | 95.5 | 1.6 | 0.9 | nd | nd | <0.4 | 97.9 |
| | 1 | 96.7 | 1.3 | 0.7 | nd | nd | <0.4 | 98.7 |
| | 4 | 96.2 | 2.4 | nd | nd | nd | 0.4 | 99.0 |
| | 4 | 94.0 | 4.7 | nd | nd | nd | 0.4 | 99.1 |
| | 8 | 91.9 | 4.7 | nd | nd | nd | <0.4 | 96.6 |
| | 8 | 90.7 | 5.7 | nd | nd | nd | <0.4 | 97.1 |
| | 14 | 87.7 | 8.3 | 0.7 | nd | nd | 0.5 | 97.2 |
| | 14 | 89.2 | 8.1 | 0.7 | nd | nd | 0.5 | 98.5 |
| | 30 | 80.0 | 14.3 | nd | 0.3 | nd | 0.8 | 97.5 |
| | 30 | 75.5 | 18.6 | nd | nd | nd | 0.9 | 94.9 |
| | 45 | 69.4 | 17.1 | nd | 0.8 | nd | 1.6 | 96.2 |
| | 45 | 68.2 | 20.0 | 0.9 | 2.0 | nd | 1.6 | 94.7 |
| | 60 | 66.6 | 21.5 | 1.4 | 2.2 | nd | 4.2 | 95.9 |
| | 60 | 65.2 | 19.9 | nd | 2.3 | nd | 4.7 | 94.5 |
| | 120 | 3.0 | 76.6 | nd | 6.1 | nd | 3.4 | 92.1 |
| | 120 | 17.2 | 61.0 | nd | 8.6 | nd | 2.3 | 93.3 |
| Eeserveen | 0 | 102 | nd | 0.5 | - | - | 0.4 | 102 |
| | 0 | 100 | nd | 0.9 | - | - | 0.5 | 102 |
| | 0.25 | 98.1 | nd | 0.8 | nd | nd | 0.5 | 99.4 |
| | 0.25 | 96.0 | nd | 0.9 | nd | nd | 0.4 | 97.8 |
| | 0.5 | 95.8 | nd | 1.6 | nd | nd | 0.5 | 97.9 |
| | 0.5 | 95.2 | nd | 0.8 | nd | nd | 0.6 | 96.6 |
| | 1 | 97.3 | nd | 1.3 | nd | nd | 0.5 | 99.7 |
| | 1 | 100 | nd | nd | nd | nd | 0.5 | 101 |
| | 4 | 97.8 | nd | 0.7 | nd | nd | 0.6 | 99.1 |
| | 4 | 97.0 | nd | 0.8 | nd | nd | 0.6 | 98.4 |
| | 8 | 93.6 | nd | 0.8 | nd | nd | 0.7 | 95.1 |
| | 8 | 96.4 | nd | 0.8 | nd | nd | 0.7 | 97.9 |
| | 14 | 94.4 | nd | 0.7 | nd | nd | 0.8 | 96.5 |
| | 14 | 95.1 | nd | nd | nd | nd | 0.8 | 95.9 |
| | 30 | 96.9 | nd | nd | nd | nd | 0.8 | 97.7 |
| | 30 | 94.3 | 0.6 | 0.6 | nd | nd | 0.7 | 96.6 |
| | 45 | 94.5 | 0.8 | nd | nd | nd | 0.9 | 96.2 |
| | 45 | 97.7 | nd | nd | nd | nd | 1.1 | 98.8 |
| | 60 | 88.4 | nd | nd | nd | nd | 9.6 | 98.0 |
| | 60 | 84.2 | nd | nd | nd | nd | 4.8 | 95.2 |
| | 120 | 94.3 | 1.0 | nd | nd | nd | 1.2 | 98.0 |
| | 120 | 93.4 | 1.1 | nd | nd | nd | 1.0 | 95.5 |

nd = not detected (limit of detection was equivalent to 0.3-2.4% AR), NER = non-extractable residue
Total applied radioactivity calculated as the sum of extractable, non-extractable, and volatile radioactivity (not the sum of preceding columns).

**Table 162 $DT_{50}$ and $DT_{90}$ values for oxamyl and IN-N0079 in saturated subsoil**

| Subsoil | Anaerobic or aerobic | Texture (pH) | $DT_{50}$ | $DT_{90}$ | $r^2$ | Method |
|---|---|---|---|---|---|---|
| **Oxamyl** | | | | | | |
| Hooghalen | Anaerobic | Loamy sand (4.1) | 0.9 hr | 2.9 hr | nc | linear first order |
| Emmer Compascuum | Anaerobic | Sand (4.4) | 1.7 hr | 5.6 hr | 0.95 | linear first order |
| Roswinkel | Aerobic | Sand (5.9) | 37 d | 122 d | 0.76 | linear first order |
| Eeserveen | Aerobic | Sand (4.5) | stable (>120 d) | stable (>120 d) | nc | linear first order |
| **IN-N0079** | | | | | | |
| Hooghalen | Anaerobic | Loamy sand (4.1) | 32 | 107 | 0.84 | linear first order |
| Emmer Compascuum | Anaerobic | Sand (4.4) | 28 | 94 | 0.89 | linear first order |
| Roswinkel | Aerobic | Sand (5.9) | nc | nc | nc | - |
| Eeserveen | Aerobic | Sand (4.5) | nc | nc | nc | - |

nc = not calculated
Oxamyl degraded too rapidly in Hooghalen subsoil for precise quantitation of the degradation rate. The $DT_{50}$ and $DT_{90}$ were estimated assuming 100% oxamyl at zero time and half of the detection limit (0.8% AR) at 0.25 days. Only trace levels of IN-N0079 were observed in Roswinkel and Eeserveen subsoils. Therefore, degradation rates of IN-N0079 could not be assessed in these subsoils.

**Conclusions:**

The rate of oxamyl degradation under anaerobic conditions was extremely rapid (DT90 <6 hours), with IN-N0079, IN-T2921 and IN-D2708 being the main metabolites. Under aerobic conditions, significant degradation was observed (DT50 = 37 days) at pH >5.9, with IN-A2213 as the primary product. Under aerobic, acidic conditions (pH ~4.4) oxamyl was stable (DT50 >120 days).

The degradation in the saturated zone study DuPont-2635, originally submitted under EU Rev8 Point IIA 7.2.1.4, 7.2.1.5 and conducted with test material [1-$^{14}$C]oxamyl, was conducted under guideline Dutch Environmental Criterial (1995). A review of this study indicates that it partially meets the current guideline (OECD 307); the major deviation is that the soils used were collected from lower soil horizons than specified in the guideline. This study was performed to aid evaluations in the Netherlands and was conducted according to the Dutch Environmental Criteria (1995). The study is a scientifically valid demonstration that oxamyl can be degraded in the saturated zone. The study should be considered as higher-tier, supplemental data and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Degradation of oxamyl under anaerobic conditions in subsoils obtained from the saturated zone is extremely rapid. > 90% oxamyl had been degraded within 6 hours of application.
Under aerobic conditions, significant degradation was observed (DT50 = 37 days) at pH >5.9, with IN-A2213 as the primary product.

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.2.3./02**

| **Reference:** -- | **Report:** | Mellor, S.J. (2000); Route and rate of degradation of oxamyl metabolite IN-A2213 in saturated subsoils<br>**DuPont Report No.:** DuPont-3098<br>**Guidelines:** Dutch Environmental Criterial (1995) |
|---|---|---|

1. Test material: [$^{14}$C]IN-A2213
   Lot/Batch #: AC/1/LK
   Purity: Radiochemical purity - >98%

**Materials and Methods:**

The route and rate of degradation of [ $^{14}$C]-IN-A2213 (25.2 µCi/mg, radiochemical purity >98%) were studied in two anaerobic subsoils (from Hooghalen and Emmer Compascuum) and two aerobic subsoils (from Roswinkel and Eeserveen) (Table ). Subsoils were sampled from a shallow saturated zone of an aquifer under agricultural fields that were representative of potato production in The Netherlands.

Saturated subsoils were treated with [ $^{14}$C]-IN-A2213 at a rate of 0.4 mg/kg dry weight (approximately equivalent to a concentration in groundwater of 0.3 to 1.9 mg/L, based on the water contents of the saturated soils), and incubated in the dark at 10 ±2 °C for up to 120 days. This application rate was considered necessary to enable the detection and quantification of IN-A2213 down to at least 1% of the applied radioactivity.

At various time intervals, subsoil samples were exhaustively extracted with aqueous/organic solvents and the total amounts of extractable IN-A2213 and its degradation products were separated and quantified by chromatography. Volatile radiolabelled degradates were trapped and quantified using a nitrogen (anaerobic) or air (aerobic) flow-through system. Non- extractable radiolabelled residues were also quantified.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Table 163 Saturated zone subsoil characteristics**

| **Soil name or designation** | **Hooghalen** | **Emmer Compascuum** | **Roswinkel** | **Eeserveen** |
|---|---|---|---|---|
| Particle size distribution (%)<br>ISO classification | | | | |
| Clay (<2 µm) | 6.56 | 2.10 | 2.76 | 0.32 |
| Silt (2 - 63 µm) | 15.70 | 2.77 | 2.85 | 1.94 |
| Sand (63 µm - 2 mm) | 77.74 | 95.13 | 94.39 | 97.74 |
| Textural class | loamy sand | sand | sand | sand |
| Organic carbon (%) | 4.9 | 0.1 | 0.1 | <0.05 |
| Organic matter (%) | 8.4 | 0.2 | 0.2 | <0.1 |
| Subsoil pH (1 : 5 v/v in water) | 5.2 | 5.4 | 6.4 | 5.3 |
| Subsoil pH (1 : 5 v/v in 0.01 M $CaCl_2$) | 4.1 | 4.4 | 5.9 | 4.5 |

| | | | | |
|---|---|---|---|---|
| Cation exchange capacity (mEq/100 g) | 17.8 | 2.2 | 1.7 | 0.6 |
| Ammonium-$N_{KCl\ extractable}$ (mg/kg) | 30.4 | 4.3 | 0.7 | 0.6 |
| Nitrate-$N_{KCl\ extractable}$ (mg/kg) | 0.8 | 0.7 | 2.5 | 9.2 |
| $Sulphate_{1\ :\ 5\ subsoil\ :\ water\ extract}$ (mg/kg) | 153.7 | 56.9 | 75.6 | 35.9 |
| $Phosphorus_{extractable}$ (mg/kg) | 3.3 | 10.0 | 7.5 | 1.1 |
| $Iron_{\ aqua\ regia\ extractable}$ (mg/kg) | 3463.8 | 2079.9 | 2064.0 | 2183.3 |
| Dry matter content (%) | 47 | 85 | 84 | 81 |
| Water content $_{dry\ weight\ basis}$ (%) | 114 | 17 | 19 | 23 |
| Biomass $_{fumigation\ extraction}$ (µg C/g) | 44.2 | <0.05 | 33.0 | <0.05 |

**Findings:**

**The redox potential and pH of each subsoil were monitored throughout the study and showed that subsoil conditions remained similar to those measured in the field. The material balance in all subsoils was in the range 83.2-101% of applied radioactivity (Table and**

Table ). Lower values, found particularly in the later samples of Emmer Compascuum saturated anaerobic subsoil, may have been due to the incomplete trapping of volatile radioactivity. Radiolabelled carbon dioxide accounted for a mean total (after 120 days) of 2.2% AR and 10.0% AR in Hooghalen and Emmer Compascuum saturated anaerobic subsoils, respectively, and 13.2% AR and 7.1% AR in Roswinkel and Eeserveen saturated aerobic subsoils. No organic volatiles were produced.

IN-D2708 was the primary degradate in all subsoils, with IN-T2921 as a minor degradate. IN- D2708 increased to a mean maximum of 17% AR in Hooghalen anaerobic subsoil by day 120, but it only accounted for a mean of 1.8% AR in Emmer Compascuum anaerobic subsoil by day 120. Its mean maximum detected levels in the aerobic subsoils were 4.4% AR for Roswinkel (day 8) and 1.55% AR for Eeserveen (day 60).

Non-extractable radioactivity accounted for a mean maximum of 17.9% AR and 5.1% AR in Hooghalen and Emmer Compascuum saturated anaerobic subsoils, respectively, and 7.6% AR and 2.7% AR in Roswinkel and Eeserveen saturated aerobic subsoils, respectively.

IN-A2213 degraded slowly in all subsoils under both aerobic and anaerobic conditions, with DT50 values (calculated by assuming linear simple first order kinetics) ranging from 158 to 630 days (Table ). DT90 values were estimated to be greater than 1 year in all cases.

The degradation pathway for IN-A2213 in the study was considered to involve formation of IN-T2921 and IN-D2708, with IN-D2708, or subsequent degradation products, being incorporated into the soil humic material or being mineralised (Figure 8.4.4.2-1).

**Table 164 Percentage distribution of radiolabelled components in saturated anaerobic subsoils after application of [$^{14}$C]-IN-A2213**

| Subsoil | Time after dosing (days) | IN-A2213 | IN-D2708 | IN-T2921 | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|---|---|
| Hooghalen | 0 | 90.0 | nd | nd | - | - | 5.0 | 95.0 |
| | 0 | 93.2 | nd | nd | - | - | 2.9 | 97.4 |
| | 0.25 | 92.7 | 2.4 | nd | nd | nd | 2.2 | 97.3 |
| | 0.25 | 92.4 | nd | nd | nd | nd | 5.0 | 97.4 |
| | 0.5 | 92.3 | 0.9 | nd | nd | nd | 5.5 | 98.7 |
| | 0.5 | 91.6 | nd | nd | nd | nd | 6.5 | 98.1 |
| | 1 | 87.9 | 0.9 | 1.2 | nd | nd | 2.7 | 93.6 |
| | 1 | 92.6 | 0.9 | nd | nd | nd | 2.3 | 95.8 |
| | 4 | 85.2 | 3.1 | 1.5 | nd | nd | 3.6 | 95.0 |
| | 4 | 89.8 | 2.3 | nd | nd | nd | 5.0 | 97.1 |
| | 8 | 92.4 | 1.4 | nd | nd | nd | 4.4 | 98.2 |
| | 8 | 91.3 | 1.1 | nd | nd | nd | 4.8 | 97.2 |
| | 14 | 91.8 | nd | nd | nd | nd | 4.8 | 96.6 |
| | 14 | 85.0 | 2.6 | 2.3 | nd | nd | 6.9 | 96.8 |
| | 30 | 75.1 | 7.0 | 3.0 | nd | nd | 8.7 | 93.7 |
| | 30 | 75.1 | 5.9 | 3.9 | nd | nd | 9.8 | 95.5 |
| | 45 | 69.2 | 8.6 | 2.9 | nd | nd | 11.5 | 92.2 |
| | 45 | 69.2 | 8.3 | 3.3 | nd | nd | 12.4 | 94.3 |
| | 60 | 66.2 | 9.0 | 3.3 | 0.2 | nd | 11.9 | 90.6 |
| | 60 | 66.3 | 9.3 | 3.4 | nd | nd | 15.8 | 96.2 |
| | 120 | 56.3 | 17.5 | 3.7 | 3.0 | nd | 15.8 | 93.4 |
| | 120 | 56.5 | 16.7 | 2.0 | 1.3 | nd | 19.9 | 96.4 |
| Emmer Compascuum | 0 | 96.7 | 1.1 | nd | - | - | 0.4 | 100 |
| | 0 | 101 | nd | nd | - | - | <0.3 | 101 |
| | 0.25 | 98.6 | nd | nd | nd | nd | 0.3 | 100 |
| | 0.25 | 97.3 | nd | nd | nd | nd | <0.3 | 97.8 |
| | 0.5 | 98.8 | nd | nd | nd | nd | <0.3 | 98.8 |
| | 0.5 | 101 | nd | nd | nd | nd | 0.4 | 101 |
| | 1 | 94.8 | 1.0 | nd | nd | nd | 0.4 | 96.2 |
| | 1 | 98.0 | nd | 0.7 | nd | nd | 0.7 | 100 |
| | 4 | 94.8 | nd | nd | nd | nd | 0.9 | 96.3 |
| | 4 | 95.8 | 0.6 | nd | nd | nd | 0.5 | 97.7 |
| | 8 | 97.1 | 0.6 | nd | nd | nd | 0.4 | 98.1 |
| | 8 | 98.0 | 0.7 | nd | nd | nd | 0.6 | 99.3 |
| | 14 | 92.9 | 1.6 | nd | nd | nd | 0.6 | 95.9 |
| | 14 | 86.9 | 3.6 | 1.3 | nd | nd | 3.1 | 97.8 |
| | 30 | 74.7 | 8.9 | 4.4 | 0.3 | nd | 6.1 | 95.0 |
| | 30 | 84.7 | 4.6 | 2.4 | nd | nd | 2.4 | 94.2 |
| | 45 | 83.6 | 5.9 | 0.9 | 0.6 | nd | 2.2 | 93.2 |
| | 45 | 80.7 | 5.9 | 1.1 | nd | nd | 2.8 | 90.5 |
| | 60 | 76.8 | 4.9 | 1.6 | 1.6 | nd | 3.7 | 89.1 |
| | 60 | 74.2 | 5.7 | 1.8 | 0.5 | nd | 4.6 | 87.8 |
| | 120 | 72.0 | 1.8 | nd | 5.2 | nd | 4.2 | 83.2 |
| | 120 | 70.7 | 1.8 | 0.7 | 14.7 | nd | 6.0 | 93.9 |

nd = not detected (limit of detection was equivalent to 0.3-1.4% AR), NER = non-extractable residue
Total applied radioactivity calculated as sum of extractable, non-extractable and volatile radioactivity (not the sum of preceding columns).

**Table 165 Percentage distribution of radiolabelled components in saturated anaerobic subsoils after application of [$^{14}$C]-IN-A2213**

| Subsoil | Time after dosing (days) | IN-A2213 | IN-D2708 | IN-T2921 | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|---|---|
| Hooghalen | 0 | 90.0 | nd | nd | - | - | 5.0 | 95.0 |
| | 0 | 93.2 | nd | nd | - | - | 2.9 | 97.4 |
| | 0.25 | 92.7 | 2.4 | nd | nd | nd | 2.2 | 97.3 |
| | 0.25 | 92.4 | nd | nd | nd | nd | 5.0 | 97.4 |
| | 0.5 | 92.3 | 0.9 | nd | nd | nd | 5.5 | 98.7 |
| | 0.5 | 91.6 | nd | nd | nd | nd | 6.5 | 98.1 |
| | 1 | 87.9 | 0.9 | 1.2 | nd | nd | 2.7 | 93.6 |
| | 1 | 92.6 | 0.9 | nd | nd | nd | 2.3 | 95.8 |
| | 4 | 85.2 | 3.1 | 1.5 | nd | nd | 3.6 | 95.0 |
| | 4 | 89.8 | 2.3 | nd | nd | nd | 5.0 | 97.1 |
| | 8 | 92.4 | 1.4 | nd | nd | nd | 4.4 | 98.2 |
| | 8 | 91.3 | 1.1 | nd | nd | nd | 4.8 | 97.2 |
| | 14 | 91.8 | nd | nd | nd | nd | 4.8 | 96.6 |
| | 14 | 85.0 | 2.6 | 2.3 | nd | nd | 6.9 | 96.8 |
| | 30 | 75.1 | 7.0 | 3.0 | nd | nd | 8.7 | 93.7 |
| | 30 | 75.1 | 5.9 | 3.9 | nd | nd | 9.8 | 95.5 |
| | 45 | 69.2 | 8.6 | 2.9 | nd | nd | 11.5 | 92.2 |
| | 45 | 69.2 | 8.3 | 3.3 | nd | nd | 12.4 | 94.3 |
| | 60 | 66.2 | 9.0 | 3.3 | 0.2 | nd | 11.9 | 90.6 |
| | 60 | 66.3 | 9.3 | 3.4 | nd | nd | 15.8 | 96.2 |
| | 120 | 56.3 | 17.5 | 3.7 | 3.0 | nd | 15.8 | 93.4 |
| | 120 | 56.5 | 16.7 | 2.0 | 1.3 | nd | 19.9 | 96.4 |
| Emmer Compascuum | 0 | 96.7 | 1.1 | nd | - | - | 0.4 | 100 |
| | 0 | 101 | nd | nd | - | - | <0.3 | 101 |
| | 0.25 | 98.6 | nd | nd | nd | nd | 0.3 | 100 |
| | 0.25 | 97.3 | nd | nd | nd | nd | <0.3 | 97.8 |
| | 0.5 | 98.8 | nd | nd | nd | nd | <0.3 | 98.8 |
| | 0.5 | 101 | nd | nd | nd | nd | 0.4 | 101 |
| | 1 | 94.8 | 1.0 | nd | nd | nd | 0.4 | 96.2 |
| | 1 | 98.0 | nd | 0.7 | nd | nd | 0.7 | 100 |
| | 4 | 94.8 | nd | nd | nd | nd | 0.9 | 96.3 |
| | 4 | 95.8 | 0.6 | nd | nd | nd | 0.5 | 97.7 |
| | 8 | 97.1 | 0.6 | nd | nd | nd | 0.4 | 98.1 |
| | 8 | 98.0 | 0.7 | nd | nd | nd | 0.6 | 99.3 |
| | 14 | 92.9 | 1.6 | nd | nd | nd | 0.6 | 95.9 |
| | 14 | 86.9 | 3.6 | 1.3 | nd | nd | 3.1 | 97.8 |
| | 30 | 74.7 | 8.9 | 4.4 | 0.3 | nd | 6.1 | 95.0 |
| | 30 | 84.7 | 4.6 | 2.4 | nd | nd | 2.4 | 94.2 |
| | 45 | 83.6 | 5.9 | 0.9 | 0.6 | nd | 2.2 | 93.2 |
| | 45 | 80.7 | 5.9 | 1.1 | nd | nd | 2.8 | 90.5 |
| | 60 | 76.8 | 4.9 | 1.6 | 1.6 | nd | 3.7 | 89.1 |
| | 60 | 74.2 | 5.7 | 1.8 | 0.5 | nd | 4.6 | 87.8 |
| | 120 | 72.0 | 1.8 | nd | 5.2 | nd | 4.2 | 83.2 |
| | 120 | 70.7 | 1.8 | 0.7 | 14.7 | nd | 6.0 | 93.9 |

nd = not detected (limit of detection was equivalent to 0.3-1.4% AR), NER = non-extractable residue
Total applied radioactivity calculated as sum of extractable, non-extractable and volatile radioactivity (not the sum of preceding columns).

**Table 166 $DT_{50}$ and $DT_{90}$ values for IN-A2213 in saturated subsoil**

| Subsoil | Anaerobic or aerobic | Texture (pH) | $DT_{50}$ (days) | $DT_{90}$ (days) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| Hooghalen | Anaerobic | Loamy sand (4.1) | 158 | > 1 year | 0.9266 | linear first order |
| Emmer Compascuum | Anaerobic | Sand (4.4) | 231 | > 1 year | 0.8085 | linear first order |
| Roswinkel | Aerobic | Sand (5.9) | 239 | > 1 year | 0.8331 | linear first order |
| Eeserveen | Aerobic | Sand (4.5) | 630 | nc | 0.7844 | linear first order |

nc = not calculated

**Conclusions:**

The rate of degradation of IN-A2213 was slow, but measurable in saturated aerobic and anaerobic subsoils at 10 ±2 °C (DT50 = 158-630 days). The route of degradation was essentially the same in all subsoils.

The degradation in the saturated zone study DuPont-3098, originally submitted under EU Rev8 Point IIA 7.2.1.4 and conducted with test material [$^{14}$C]IN-A2213, was conducted under guideline Dutch Environmental Criterial (1995). A review of this study indicates that it partially meets the current guideline (OECD 307); the major deviation is that the soils used were collected from lower soil horizons than specified in the guideline. This study was performed to aid evaluations in the Netherlands and was conducted according to the Dutch Environmental Criteria (1995). The study is a scientifically valid demonstration that IN-A2213 can be degraded in the saturated zone. The study should be considered as higher-tier, supplemental data and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Degradation of oxamyl oxime under aerobic and anaerobic conditions in subsoils was slow. Less than 50% of the oxamyl oxime had degraded within 158-630 days of application.

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.2.3/03**

| Reference: | Report: | |
|---|---|---|
| -- | | Shaw, D. (2000); Route and rate of degradation of oxamyl metabolite IN-D2708 in saturated subsoils<br>**DuPont Report No.:** DuPont-3097<br>**Guidelines:** Dutch Environmental Criterial (1995) |

1. Test material: [$^{14}$C]IN-D2708
   Lot/Batch #: 3389-009
   Purity: Radiochemical purity - >97%

**Material and Methods:**

The route and rate of degradation of [ $^{14}$C]-IN-D2708 (49.53 µCi/mg, radiochemical purity >97%) were studied in two anaerobic subsoils (from Hooghalen and Emmer Compascuum) and two aerobic subsoils (from Roswinkel and Eeserveen) (Table 8.4.4.2-5). Subsoils were sampled from a shallow saturated zone of an aquifer under agricultural fields representative of potato production in The Netherlands.

**Table 167 Saturated zone subsoil characteristics**

| Subsoil name or designation | Hooghalen | Emmer Compascuum | Roswinkel | Eeserveen |
|---|---|---|---|---|
| Particle size distribution (%)<br>ISO classification | | | | |
| Clay (<2 μm) | 6.56 | 2.10 | 2.76 | 0.32 |
| Silt (2 - 63 μm) | 15.70 | 2.77 | 2.85 | 1.94 |
| Sand (63 μm – 2 mm) | 77.74 | 95.13 | 94.39 | 97.74 |
| Textural class | loamy sand | sand | sand | sand |
| Organic carbon (%) | 4.9 | 0.1 | 0.1 | <0.05 |
| Organic matter (%) | 8.4 | 0.2 | 0.2 | <0.1 |
| Subsoil pH (1 : 5 v/v in water) | 5.2 | 5.4 | 6.4 | 5.3 |
| Subsoil pH (1 : 5 v/v in 0.01 M $CaCl_2$) | 4.1 | 4.4 | 5.9 | 4.5 |
| Cation exchange capacity (mEq/100 g) | 17.8 | 2.2 | 1.7 | 0.6 |
| Ammonium-$N_{KCl\ extractable}$ (mg/kg) | 30.4 | 4.3 | 0.7 | 0.6 |
| Nitrate-$N_{KCl\ extractable}$ (mg/kg) | 0.8 | 0.7 | 2.5 | 9.2 |
| $Sulphate_{1\ :\ 5\ subsoil\ :\ water\ extract}$ (mg/kg) | 153.7 | 56.9 | 75.6 | 35.9 |
| $Phosphorus_{extractable}$ (mg/kg) | 3.3 | 10.0 | 7.5 | 1.1 |
| Iron $_{aqua\ regia\ extractable}$ (mg/kg) | 3463.8 | 2079.9 | 2064.0 | 2183.3 |
| Dry matter content (%) | 47 | 85 | 84 | 81 |
| Water content $_{dry\ weight\ basis}$ (%) | 114 | 17 | 19 | 23 |
| Biomass $_{fumigation\ extraction}$ (μg C/g) | 515.7 | 39.2 | 51.5 | 132.9 |

Saturated subsoils were treated with [ $^{14}C$]-IN-D2708 at a rate of 0.2 mg/kg dry weight (approximately equivalent to a concentration in groundwater of 0.2 to 1.0 mg/L, based on the water contents of the saturated soils), and incubated in the dark at 10 ±2 °C for up to 120 days. This rate was considered necessary to enable the detection and quantification of IN-D2708 and its degradation products down to at least 1% of the applied radioactivity.

At various time intervals, subsoil samples were exhaustively extracted with aqueous/organic solvents and the total amounts of extractable IN-D2708 and any degradation products were separated and quantified by chromatography. Volatile radiolabelled degradates were trapped and quantified using a nitrogen (anaerobic) or air (aerobic) flow-through system. Non- extractable radiolabelled residues were also quantified.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Findings:**

The redox potential and pH of each subsoil were monitored throughout the study and showed that subsoil conditions remained similar to those measured in the field. The material balance in all subsoils was in the range 84.0-101% of applied radioactivity (Table and Table ). Lower values, found particularly in later samples of Roswinkel saturated aerobic subsoil, may have been due to incomplete trapping of volatile radioactivity when

evolution of volatiles was high from this subsoil. Radiolabelled carbon dioxide accounted for maxima of 2.0% AR and 3.1% AR in Hooghalen and Emmer Compascuum saturated anaerobic subsoils, respectively, and 65.4% AR and 7.6% AR in Roswinkel and Eeserveen saturated aerobic subsoils, respectively (after 120 days). No organic volatiles were produced.

There were no extractable degradation products of IN-D2708. Non-extractable radioactivity accounted for maxima of 6.5% AR and 2.1% AR in Hooghalen and Emmer Compascuum saturated anaerobic subsoils, respectively, and 23.6% AR and 2.5% AR in Roswinkel and Eeserveen saturated aerobic subsoils, respectively.

IN-D2708 degraded slowly in both saturated anaerobic subsoils and in Eeserveen saturated aerobic subsoil, with DT50 values estimated to be greater than 1 year (Table ). After 120 days incubation, IN-D2708 accounted for 83-87% AR (mean values) in both saturated anaerobic subsoils and in Eeserveen saturated aerobic subsoil. In Roswinkel saturated aerobic subsoil there was a lag period of between 14 and 30 days, but the decline of IN-D2708 was rapid thereafter. The estimated DT50 for the decline of IN-D2708 in Roswinkel subsoil over the entire 60-day incubation period was 11 days (with a corresponding DT90 value of 36 days), whereas the estimated DT50 for the decline of IN-D2708 over the 30-45 day period of the incubation was approximately 3 days.

In both saturated anaerobic and aerobic subsoils, degradation of IN-D2708 proceeded exclusively via incorporation into the subsoil organic fraction (non-extractable residue) and formation of carbon dioxide.

**Table 168 Percentage distribution of radiolabelled components in saturated anaerobic subsoils after application of [$^{14}$C]-IN-D2708**

| Subsoil | Time after dosing (days) | IN-D2708 | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|
| Hooghalen | 0 | 87.6 | - | - | 6.0 | 93.6 |
| | 0 | 84.5 | - | - | 6.5 | 91.0 |
| | 0.25 | 88.2 | 0.5 | nd | 4.3 | 93.0 |
| | 0.25 | 90.7 | 1.0 | nd | 4.5 | 96.2 |
| | 0.5 | 90.5 | 0.3 | nd | 4.2 | 95.0 |
| | 0.5 | 89.4 | 0.5 | nd | 4.2 | 94.1 |
| | 1 | 90.9 | 0.5 | nd | 2.5 | 93.9 |
| | 1 | 92.5 | 1.6 | nd | 2.4 | 96.5 |
| | 4 | 87.1 | 1.7 | nd | 3.5 | 92.3 |
| | 4 | 86.6 | 0.4 | nd | 3.9 | 90.9 |
| | 8 | 87.9 | 1.6 | nd | 4.6 | 94.1 |
| | 14 | 84.7 | 1.1 | nd | 4.4 | 90.2 |
| | 14 | 87.8 | 0.3 | nd | 2.6 | 90.7 |
| | 30 | 81.3 | 0.8 | nd | 4.2 | 86.3 |
| | 30 | 83.9 | 0.9 | nd | 3.1 | 87.9 |
| | 45 | 84.2 | nd | nd | 4.0 | 88.2 |
| | 45 | 83.8 | 1.7 | nd | 4.1 | 89.6 |
| | 60 | 84.6 | 1.1 | nd | 4.9 | 90.6 |
| | 60 | 84.4 | 2.0 | nd | 3.7 | 90.1 |
| | 120 | 84.6 | 1.1 | nd | 3.7 | 89.4 |
| | 120 | 83.1 | 1.8 | nd | 5.5 | 90.4 |
| | 0 | 94.3 | - | - | 1.8 | 96.1 |
| | 0 | 95.1 | - | - | 2.1 | 97.2 |
| | 0.25 | 92.5 | 0.5 | nd | 1.6 | 94.6 |
| | 0.25 | 90.7 | 0.4 | nd | 1.2 | 92.3 |
| | 0.5 | 92.1 | 0.7 | nd | 1.2 | 94.0 |
| | 0.5 | 92.9 | nd | nd | 1.1 | 94.0 |
| | 1 | 90.5 | 0.4 | nd | 1.4 | 92.3 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 92.1 | 0.8 | nd | 1.3 | 94.2 |
| | 4 | 92.2 | 1.5 | nd | 1.2 | 94.9 |
| | 4 | 93.2 | 1.4 | nd | 1.7 | 96.3 |
| | 8 | 92.4 | 1.1 | nd | 1.8 | 95.3 |
| Compascuum | 8 | 90.1 | 1.0 | nd | 2.0 | 93.1 |
| | 14 | 87.3 | 1.8 | nd | 1.1 | 90.2 |
| | 14 | 80.3 | 2.0 | nd | 1.7 | 84.0 |
| | 30 | 87.3 | 1.6 | nd | 1.9 | 90.8 |
| | 30 | 92.4 | 1.9 | nd | 1.4 | 95.7 |
| | 45 | 87.5 | 1.7 | nd | 1.4 | 90.6 |
| | 45 | 86.8 | 1.9 | nd | 1.4 | 90.1 |
| | 60 | 91.8 | 1.7 | nd | 1.3 | 94.8 |
| | 60 | 86.4 | 3.1 | nd | 2.1 | 91.6 |
| | 120 | 86.5 | 1.8 | nd | 1.5 | 89.8 |
| | 120 | 87.2 | 1.2 | nd | 1.9 | 90.3 |

nd = not detected (limit of detection was equivalent to 0.2-0.4% AR), NER = non-extractable residue

**Table 169 Percentage distribution of radiolabelled components in saturated aerobic subsoils after application of [$^{14}$C]-IN-D2708**

| Subsoil | Time after dosing (days) | IN-D2708 | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|
| | 0 | 90.5 | - | - | 2.3 | 92.8 |
| | 0 | 91.1 | - | - | 1.8 | 92.9 |
| | 0.25 | 91.6 | nd | nd | 1.0 | 92.6 |
| | 0.25 | 93.6 | nd | nd | 1.3 | 94.9 |
| | 0.5 | 92.5 | 0.6 | nd | 1.0 | 94.1 |
| | 0.5 | 93.5 | 0.3 | nd | 1.1 | 94.9 |
| | 1 | 91.7 | 0.6 | nd | 1.0 | 93.3 |
| | 1 | 94.2 | 0.8 | nd | 0.9 | 95.9 |
| | 4 | 96.4 | 1.1 | nd | 0.9 | 98.4 |
| Roswinkel | 4 | 96.2 | 1.2 | nd | 0.8 | 98.2 |
| | 8 | 88.8 | 2.4 | nd | 1.7 | 92.9 |
| | 8 | 92.7 | 1.5 | nd | 1.2 | 95.4 |
| | 14 | 90.5 | 2.0 | nd | 0.7 | 93.2 |
| | 14 | 95.2 | 2.0 | nd | 1.2 | 98.4 |
| | 30 | 64.6 | 12.8 | nd | 9.9 | 87.3 |
| | 30 | 87.3 | 4.1 | nd | 1.5 | 92.9 |
| | 45 | 3.7 | 60.9 | nd | 23.6 | 88.2 |
| | 45 | 2.5 | 65.2 | nd | 22.3 | 90.0 |
| | 60 | 2.4 | 65.4 | nd | 18.5 | 86.3 |
| | 60 | 1.8 | 63.1 | nd | 19.1 | 84.0 |
| | 0 | 93.6 | - | - | 1.5 | 95.1 |
| | 0 | 91.1 | - | - | 1.5 | 92.6 |
| | 0.25 | 95.6 | nd | nd | 1.1 | 96.7 |
| | 0.25 | 95.6 | nd | nd | 1.0 | 96.6 |
| | 0.5 | 88.9 | nd | nd | 1.5 | 90.4 |
| | 0.5 | 95.2 | 0.4 | nd | 1.3 | 96.9 |
| | 1 | 90.4 | 0.9 | nd | 0.8 | 92.1 |
| | 1 | 90.2 | 0.8 | nd | 0.8 | 91.8 |
| | 4 | 98.1 | 1.5 | nd | 1.1 | 101 |
| | 4 | 96.8 | 1.8 | nd | 1.1 | 99.7 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Eeserveen | 8 | 92.0 | 1.3 | nd | 1.7 | 95.0 |
| | 8 | 89.5 | 1.3 | nd | 1.7 | 92.5 |
| | 14 | 93.1 | 1.7 | nd | 1.6 | 96.4 |
| | 14 | 93.6 | 1.6 | nd | 1.9 | 97.1 |
| | 30 | 89.4 | 1.5 | nd | 1.4 | 92.3 |
| | 30 | 90.3 | 2.0 | nd | 1.1 | 93.4 |
| | 45 | 92.8 | 2.3 | nd | 1.3 | 96.4 |
| | 45 | 94.6 | 1.6 | nd | 1.5 | 97.7 |
| | 60 | 90.0 | 2.3 | nd | 1.7 | 94.0 |
| | 60 | 94.4 | 2.6 | nd | 1.8 | 98.8 |
| | 120 | 82.6 | 7.6 | nd | 1.8 | 92.0 |
| | 120 | 83.4 | 6.0 | nd | 2.5 | 91.9 |

nd = not detected (limit of detection was equivalent to 0.2-0.4% AR), NER = non-extractable residue

**Table 170 $DT_{50}$ and $DT_{90}$ values for IN-D2708 in saturated subsoil**

| Subsoil | Anaerobic or aerobic | Texture (pH) | $DT_{50}$ (days) | $DT_{90}$ (days) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| Hooghalen | Anaerobic | Loamy sand (4.1) | 1209 | nc | 0.4208 | linear first order |
| Emmer Compascuum | Anaerobic | Sand (4.4) | 1355 | nc | 0.2949 | linear first order |
| Roswinkel | Aerobic | Sand (5.9) | 11 | 36 | 0.8314 | linear first order |
| Eeserveen | Aerobic | Sand (4.5) | 859 | nc | 0.5317 | linear first order |

nc = not calculated
The $DT_{50}$ estimates in Hooghalen, Emmer Compascuum, and Eeserveen are extrapolated well beyond the length of the study. While quantitatively unreliable, these estimates demonstrate that IN-D2708 is stable under the conditions tested.

**Conclusions:**

Dissipation of IN-D2708 proceeded exclusively via incorporation into the subsoil organic fraction (non-extractable residue) and formation of carbon dioxide. The formation of carbon dioxide was observed to a greater extent under aerobic (oxidising) conditions compared to anaerobic (reducing) conditions. Under aerobic conditions and moderately acidic pH (~5.9), the IN-D2708 DT50 was 11 days at 10 °C. The IN-D2708 DT50 was >120 days under anaerobic acidic and aerobic acidic conditions at 10 °C.

The degradation in the saturated zone study DuPont-3097, originally submitted under EU Rev8 Point IIA 7.2.1.4 and conducted with test material [$^{14}$C]IN-D2708, was conducted under guideline Dutch Environmental Criterial (1995). A review of this study indicates that it partially meets the current guideline (OECD 307); the major deviation is that the soils used were collected from lower soil horizons than specified in the guideline. This study was performed to aid evaluations in the Netherlands and was conducted according to the Dutch Environmental Criteria (1995). The study is a scientifically valid demonstration that IN-D2708 can be degraded in the saturated zone. The study should be considered as higher-tier, supplemental data and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
In both saturated anaerobic and aerobic subsoils, dissipation of DMOA proceeded exclusively via incorporation into the subsoil bound residue and evolution af carbon dioxide.
Degradation of IN-D2708 under aerobic and anaerobic conditions in subsoils was greater than 1 year.

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.2.3/04**

| **Reference:** -- | **Report:** | Millais, A.J. (2000); Route and rate of degradation of oxamyl metabolite IN-N0079 in saturated subsoils<br>**DuPont Report No.:** DuPont-3070<br>**Guidelines:** Dutch Environmental Criterial (1995) |
|---|---|---|

1. Test material: [$^{14}$C]IN-N0079
   Lot/Batch #: CFQ11389
   Purity: Radiochemical purity - >98%

**Material and Methods:**

The route and rate of degradation of [ $^{14}$C]-IN-N0079 (45 µCi/mg, radiochemical purity >98%) were studied in two anaerobic subsoils (from Hooghalen and Emmer Compascuum) and two aerobic subsoils (from Roswinkel and Eeserveen) (Table ). Subsoils were sampled from a shallow saturated zone of an aquifer under fields that were representative of potato production in The Netherlands.

Saturated subsoils were treated with [ $^{14}$C]-IN-N0079 at a rate of 0.2 mg/kg dry weight (approximately equivalent to a concentration in groundwater of 0.2 to 1.0 mg/L, based on the water contents of the saturated soils), and incubated in the dark at 10 ±2 °C for up to 60 days. This rate was considered necessary to enable the detection and quantification of IN-N0079 and its degradation products down to at least 1% of the applied radioactivity.

At various time intervals, subsoil samples were exhaustively extracted with aqueous/organic solvents and the total amounts of extractable IN-N0079 and its degradation products were separated and quantified by chromatography. Volatile radiolabelled degradates were trapped and quantified using a nitrogen (anaerobic) or air (aerobic) flow-through system. Non- extractable radiolabelled residues were also quantified.

Kinetic values were calculated using linear least squares regression, assuming first order kinetics.

**Table 171 Saturated zone subsoil characteristics**

| Subsoil name or designation | Hooghalen | Emmer Compascuum | Roswinkel | Eeserveen |
|---|---|---|---|---|
| Particle size distribution (%)<br>ISO classification | | | | |
| Clay (<2 µm) | 6.56 | 2.10 | 2.76 | 0.32 |
| Silt (2 - 63 µm) | 15.70 | 2.77 | 2.85 | 1.94 |
| Sand (63 µm - 2 mm) | 77.74 | 95.13 | 94.39 | 97.74 |
| Textural class | loamy sand | sand | sand | sand |
| Organic carbon (%) | 4.9 | 0.1 | 0.1 | <0.05 |
| Organic matter (%) | 8.4 | 0.2 | 0.2 | <0.1 |
| Subsoil pH (1 : 5 v/v in water) | 5.2 | 5.4 | 6.4 | 5.3 |
| Subsoil pH (1 : 5 v/v in 0.01 M $CaCl_2$) | 4.1 | 4.4 | 5.9 | 4.5 |
| Cation exchange capacity (mEq/100 g) | 17.8 | 2.2 | 1.7 | 0.6 |
| Ammonium-$N_{KCl\ extractable}$ (mg/kg) | 30.4 | 4.3 | 0.7 | 0.6 |
| Nitrate-$N_{KCl\ extractable}$ (mg/kg) | 0.8 | 0.7 | 2.5 | 9.2 |

| | | | | |
|---|---|---|---|---|
| $Sulphate_{1:5\ subsoil:water\ extract}$ (mg/kg) | 153.7 | 56.9 | 75.6 | 35.9 |
| $Phosphorus_{extractable}$ (mg/kg) | 3.3 | 10.0 | 7.5 | 1.1 |
| $Iron_{aqua\ regia\ extractable}$ (mg/kg) | 3463.8 | 2079.9 | 2064.0 | 2183.3 |
| Dry matter content (%) | 47 | 85 | 84 | 81 |
| Water content $_{dry\ weight\ basis}$ (%) | 114 | 17 | 19 | 23 |
| Biomass $_{fumigation\ extraction}$ (µg C/g) | <0.05 | 18.0 | <0.05 | <0.05 |

**Findings:**

The redox potential and pH of each subsoil were monitored throughout the study and showed that subsoil conditions remained similar to those measured in the field. The material balance in all subsoils was in the range 82.4-99.2% of applied radioactivity, with the exception of two samples where the amount extracted was considered to be anomalous (Table and Table ). In the anaerobic subsoils, no labelled carbon dioxide or organic volatiles were produced. In Roswinkel saturated aerobic subsoil, a mean maximum of 5.5% AR was mineralised to carbon dioxide by day 60, with volatile organic radioactivity reaching a mean maximum of 6.3% AR by day 4. In Eeserveen saturated aerobic subsoil, a mean maximum of 2.3% AR was mineralised to carbon dioxide by day 60. Significant quantities of volatile organic radioactivity were also detected in this subsoil and accounted for a mean maximum of 24.3% AR after 30 days.

IN-T2921 (the corresponding amide of IN-N0079) and IN-D2708 (the corresponding acid of IN-N0079) were the main metabolites of IN-N0079 in all soils. Two other unidentified products were observed, which were designated D2 and D3B. D2 remained below 10% AR in all soils, while D3B exceeded 10% AR in Hooghalen soil, being detected at a maximum level of 29.2% AR in one replicate (day 1). Levels of D3B in this subsoil declined to <10% AR by day 45.

In Hooghalen saturated anaerobic subsoil, extractable radioactivity declined with time to a mean of 85.9% AR by day 60, from 91.4% AR at zero time. Non-extractable radioactivity increased to a mean of 7.0% AR on day 60, and no volatile radioactivity was detected at this timepoint. IN-D2708 and IN-T2921 were first detected at day 1 (in single replicates only, at respective levels of 0.9% AR and 1.0% AR), and reached respective mean maximum levels of 35.1% AR (day 60) and 14.2% AR (day 30 and day 60) (Table ).

In Emmer Compascuum saturated anaerobic subsoil, extractability of radioactivity declined to a mean of 90.5% AR by day 60, from a mean value of 95.5% AR at zero time, whilst the non- extractable residue was not more than 1.5% AR in any individual sample analysed. IN-T2921 was first observed at day 0.25 at a level of 2.6% AR (single replicate), and reached a mean maximum level of 23.5% AR on day 45 (Table ). IN-D2708 increased from a level of 3.1% AR on day 1 (single replicate) to a mean maximum level of 61.8% AR on day 45 (Table ).

In Roswinkel saturated aerobic subsoil, extractable radioactivity declined to a mean of 82.2% AR at day 60, from a mean value of 95.1% AR at zero time. A mean maximum of 5.5% AR was mineralised to carbon dioxide by day 60. IN-T2921 was detected in one of the zero-time samples at a level of 0.7% AR, and increased to a mean maximum level of 37.8% AR after 8 days incubation. IN-D2708 was also detected in one of the zero-time samples (at a level of 6.5% AR), and reached a mean maximum level of 80.9% AR on day 14 (Table ).

In Eeserveen saturated aerobic subsoil, extractable radioactivity declined to a mean maximum of 69.2% AR at day 60, from a mean value at zero time of 94.4% AR. Significant quantities of volatile organic radioactivity were detected and accounted for a mean maximum of 24.3% AR after 30 days. IN-T2921 was first detected at day 0.25 at a mean level of 2.5% AR, and increased to a mean maximum level of 28.6% AR after 8 days incubation. IN-D2708 was first detected at day 0.5 at a mean level of 3.0% AR, and was detected at a maximum level of 38.8% AR on day 60 (single replicate) (Table ).

IN-N0079 degraded readily in all subsoils under both aerobic and anaerobic conditions, with DT50 values ranging from 1 to 45 days (Table ). The DT90 was reached by 60 days of incubation in all subsoils except Hooghalen. The rate of degradation was most rapid in the Roswinkel aerobic saturated subsoil, with a DT50 of 1.1 day. The anaerobic saturated subsoil Hooghalen gave the slowest rate of degradation, with a DT50 of 45.3 days. The other subsoils, Eeserveen (aerobic) and Emmer Compascuum (anaerobic), had intermediate DT50 values of 12.4 and 10.7 days respectively (Table ). These data suggest rapid degradation of IN-N0079 under field conditions.

The major degradation pathway for IN-N0079 in all subsoils involves addition of successive water molecules to form IN-T2921 and IN-D2708. IN-D2708 or subsequent degradation products are incorporated into the soil humic material or mineralised (Figure 8.4.4.2-2). The degradation of a nitrile to the corresponding carboxylic acid via the amide is well characterised in many soil micro-organisms.

**Table 172 Percentage distribution of radiolabelled components in saturated anaerobic subsoils after application of [$^{14}$C]-IN-N0079**

| Subsoil | Time after dosing (days) | IN-D2708 | IN-T2921 | D2 | IN-N0079 | D3B | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|---|---|---|---|
| Hooghalen | 0 | nd | nd | nd | 70.8 | 20.6 | - | - | 5.3 | 96.7 |
| | 0 | nd | nd | nd | 87.1 | 4.2 | - | - | 6.3 | 97.6 |
| | 0.25 | nd | nd | nd | 81.1 | 13.2 | nd | nd | 3.5 | 97.8 |
| | 0.25 | nd | nd | nd | 84.1 | 11.6 | nd | nd | 2.1 | 97.9 |
| | 0.5 | nd | nd | nd | 68.9 | 25.4 | nd | nd | 2.5 | 96.8 |
| | 0.5 | nd | nd | 3.8 | 86.3 | 6.9 | nd | nd | 2.2 | 99.2 |
| | 1 | 0.9 | 1.0 | nd | 62.7 | 29.2 | nd | nd | 1.5 | 95.3 |
| | 1 | nc | nc | nc | nc | nc | nd | nd | 2.0 | 79.0 |
| | 4 | 9.2 | 6.1 | nd | 66.6 | 11.9 | nd | nd | 2.1 | 95.8 |
| | 4 | 3.8 | 2.2 | nd | 63.4 | 24.6 | nd | nd | 2.0 | 95.8 |
| | 8 | 13.7 | 11.1 | nd | 59.3 | 5.9 | nd | nd | 3.1 | 93.1 |
| | 8 | 7.6 | 5.4 | nd | 54.7 | 22.6 | nd | nd | 3.8 | 94.1 |
| | 14 | 6.1 | 4.2 | nd | 76.0 | 7.4 | nd | nd | 3.6 | 97.3 |
| | 14 | 7.9 | 7.4 | nd | 64.3 | 10.2 | nd | nd | 3.8 | 93.6 |
| | 30 | 35.8 | 21.0 | nd | 18.2 | 12.5 | nd | nd | 5.4 | 92.9 |
| | 30 | 14.1 | 7.3 | nd | 61.3 | 8.2 | nd | nd | 3.2 | 94.2 |
| | 45 | 17.6 | 6.7 | nd | 58.6 | 4.2 | nd | nd | 9.5 | 98.2 |
| | 45 | 31.1 | 18.8 | nd | 26.2 | 7.9 | nd | nd | 4.9 | 91.3 |
| | 60 | 43.7 | 14.9 | nd | 22.9 | 4.5 | nd | nd | 7.7 | 93.8 |
| | 60 | 26.4 | 13.4 | nd | 40.4 | 5.4 | nd | nd | 6.2 | 91.8 |
| Emmer Compascuum | 0 | nd | nd | nd | 95.9 | na | - | - | nd | 95.9 |
| | 0 | nd | nd | nd | 94.4 | 0.6 | - | - | nd | 95.0 |
| | 0.25 | nd | nd | nd | 95.7 | na | nd | nd | nd | 95.7 |
| | 0.25 | nd | 2.6 | nd | 95.5 | nd | nd | nd | nd | 98.1 |
| | 0.5 | nc | nc | nc | nc | nc | nd | nd | nd | 79.1 |
| | 0.5 | nd | 0.7 | nd | 94.2 | na | nd | nd | nd | 94.9 |
| | 1 | 3.1 | nd | nd | 88.2 | 0.9 | nd | nd | nd | 92.3 |
| | 1 | nd | 0.9 | nd | 90.8 | na | nd | nd | nd | 91.7 |
| | 4 | 5.2 | 5.5 | nd | 84.7 | na | nd | nd | 0.4 | 96.4 |
| | 4 | 5.4 | 5.4 | nd | 80.4 | nd | nd | nd | nd | 91.3 |
| | 8 | 7.3 | 9.8 | nd | 76.0 | na | nd | nd | 0.5 | 93.7 |
| | 8 | 14.7 | 8.7 | nd | 68.9 | nd | nd | nd | nd | 92.5 |
| | 14 | 12.7 | 8.1 | nd | 73.8 | na | nd | nd | 0.9 | 95.5 |
| | 14 | 20.5 | 22.9 | nd | 46.0 | 6.7 | nd | nd | 0.7 | 97.0 |
| | 30 | 62.3 | 24.3 | nd | nd | 2. 5 | nd | nd | 1.1 | 90.5 |
| | 30 | 58.2 | 17.4 | 4.2 | 4.2 | 4.1 | nd | nd | 0.6 | 91.2 |
| | 45 | 61.5 | 16.3 | 3.2 | 2.8 | 3.1 | nd | nd | 1.1 | 92.7 |
| | 45 | 62.0 | 30.6 | nd | nd | 2.5 | nd | nd | 1.1 | 96.4 |
| | 60 | 57.0 | 25.5 | nd | 7.0 | 2.5 | nd | nd | 1.4 | 93.5 |
| | 60 | 39.5 | 6.5 | 3.8 | 26.0 | 5.1 | nd | nd | 1.5 | 90.4 |

nd = not detected (limit of detection was equivalent to 0.3-1.3% AR), nc = not calculated (vessel recovery anomalous), na = not applicable (samples analysed in HPLC method 1, which did not resolve D3B), NER = non- extractable residue

Total applied radioactivity calculated as sum of extractable, non-extractable and volatile radioactivity (not the sum of preceding columns).

**Table 173 Percentage distribution of radiolabelled components in saturated aerobic subsoils after application of [$^{14}$C]-IN-N0079**

| Subsoil | Time after dosing (days) | IN-D2708 | IN-T2921 | D2 | IN-N0079 | D3B | $^{14}CO_2$ | Organic volatiles | NER | Total recovery |
|---|---|---|---|---|---|---|---|---|---|---|
| Roswinkel | 0 | nd | nd | nd | 91.3 | 3.2 | - | - | nd | 94.5 |
| | 0 | 6.5 | 0.7 | 2.4 | 84.3 | nd | - | - | nd | 95.6 |
| | 0.25 | 5.3 | 7.7 | nd | 72.5 | 5.2 | nd | 1.4 | nd | 92.0 |
| | 0.25 | 12.4 | 11.2 | nd | 65.3 | 4.5 | nd | 0.6 | nd | 94.0 |
| | 0.5 | 9.6 | 18.3 | nd | 59.3 | 4.1 | nd | 3.5 | nd | 94.8 |
| | 0.5 | 6.1 | 15.7 | nd | 66.1 | 4.2 | nd | 0.6 | nd | 92.7 |
| | 1 | 22.0 | 22.0 | nd | 43.1 | 2.1 | nd | 2.4 | nd | 91.7 |
| | 1 | 28.8 | 22.5 | nd | 36.8 | 3.4 | nd | 1.5 | nd | 92.9 |
| | 4 | 47.3 | 31.1 | nd | 4.5 | nd | 0.5 | 7.1 | 0.4 | 91.0 |
| | 4 | 43.7 | 33.6 | nd | 7.1 | 1.0 | nd | 5.4 | nd | 91.0 |
| | 8 | 43.9 | 37.7 | nd | nd | 3.8 | nd | 6.8 | 0.5 | 92.7 |
| | 8 | 53.8 | 37.8 | nd | nd | 0.6 | nd | 3.2 | nd | 95.3 |
| | 14 | 78.3 | 6.4 | nd | nd | nd | nd | 7.1 | nd | 91.8 |
| | 14 | 83.5 | 5.7 | nd | nd | nd | nd | 5.3 | 0.8 | 95.3 |
| | 30 | 77.6 | 8.1 | nd | nd | nd | 1.1 | 4.8 | 0.9 | 92.5 |
| | 30 | 68.9 | 18.6 | nd | nd | nd | nd | 2.0 | 0.9 | 92.0 |
| | 46 | 73.6 | 5.8 | nd | nd | 3.8 | 4.0 | 1.5 | 4.0 | 92.8 |
| | 46 | 74.9 | 11.6 | nd | nd | nd | 2.0 | 4.9 | 1.6 | 95.0 |
| | 60 | 74.8 | 9.6 | nd | nd | nd | 3.7 | 1.1 | 3.5 | 94.7 |
| | 60 | 72.2 | 2.6 | nd | nd | 3.2 | 7.3 | 4.1 | 3.7 | 93.0 |
| Eeserveen | 0 | nd | nd | 6.3 | 81.4 | 6.5 | - | - | nd | 94.2 |
| | 0 | nd | nd | nd | 86.7 | 7.9 | - | - | nd | 94.6 |
| | 0.25 | nd | 2.5 | nd | 86.1 | 4.4 | nd | 1.8 | nd | 94.7 |
| | 0.25 | nd | 2.4 | nd | 81.2 | 7.9 | nd | 0.8 | nd | 92.2 |
| | 0.5 | 3.8 | 4.1 | nd | 82.8 | 3.1 | nd | 1.4 | nd | 95.3 |
| | 0.5 | 2.1 | 1.8 | nd | 78.2 | 8.3 | nd | 1.3 | nd | 91.6 |
| | 1 | 5.3 | 6.3 | nd | 68.3 | 7.4 | nd | 4.5 | nd | 91.9 |
| | 1 | 7.3 | 7.6 | nd | 69.5 | 5.9 | nd | 2.2 | nd | 92.4 |
| | 4 | 20.4 | 19.9 | nd | 37.7 | 1.9 | nd | 7.4 | nd | 89.8 |
| | 4 | 23.2 | 21.3 | nd | 37.7 | 2.5 | nd | 6.2 | nd | 91.0 |
| | 8 | 25.7 | 28.3 | nd | 29.9 | nd | nd | 8.6 | nd | 92.7 |
| | 8 | 24.8 | 28.8 | nd | 35.6 | nd | 0.9 | 1.8 | nd | 91.8 |
| | 14 | 21.9 | 16.2 | nd | 32.2 | 2.8 | nd | 16.9 | nd | 90.2 |
| | 14 | 26.5 | 12.6 | nd | 31.8 | nd | 2.4 | 15.9 | 0.5 | 89.7 |
| | 30 | 22.3 | 11.1 | nd | 17.4 | 0.9 | 7.0 | 31.1 | 0.5 | 90.3 |
| | 30 | 21.1 | 22.5 | nd | 25.7 | nd | nd | 17.4 | nd | 87.0 |
| | 46 | 17.9 | 16.8 | nd | 25.1 | 0.8 | nd | 21.0 | 0.5 | 82.4 |
| | 46 | 16.6 | 13.5 | nd | 37.4 | 2.7 | nd | 21.2 | 0.5 | 92.2 |
| | 60 | 38.8 | 13.1 | nd | nd | nd | 2.2 | 31.4 | 0.6 | 86.1 |
| | 60 | nc | nc | nc | nc | nc | 2.4 | 4.3 | 1.2 | 94.4 |

nd = not detected (limit of detection was equivalent to 0.3-1.3% AR), nc = not calculated (vessel recovery anomalous), NER = non-extractable residue
Total applied radioactivity calculated as sum of extractable, non-extractable and volatile radioactivity (not the sum of preceding columns).

**Table 174 $DT_{50}$ and $DT_{90}$ values for IN-N0079 in saturated subsoil**

| Subsoil | Anaerobic or aerobic | Texture (pH) | $DT_{50}$ (days) | $DT_{90}$ (days) | $r^2$ | Method |
|---|---|---|---|---|---|---|
| Hooghalen | Anaerobic | Loamy sand (4.1) | 45.3 | 150.0 | 0.8750 | linear first order |

| | | | | | | |
|---|---|---|---|---|---|---|
| Emmer Compascuum | Anaerobic | Sand (4.4) | 10.7 | 35.4 | 0.7574 | linear first order |
| Roswinkel | Aerobic | Sand (5.9) | 1.1 | 3.7 | 0.9977 | linear first order |
| Eeserveen | Aerobic | Sand (4.5) | 12.4 | 41.0 | 0.7222 | linear first order |

**Conclusions:**

The rate of degradation of IN-N0079 was rapid (DT50 = 1 day to 45 days) in aerobic and anaerobic saturated subsoils at 10 ±2 °C. The route of degradation was essentially the same in all subsoils.

The degradation in the saturated zone study DuPont-3070, originally submitted under EU Rev8 Point IIA 7.2.1.4 and conducted with test material [$^{14}$C]IN-N0079, was conducted under guideline Dutch Environmental Criterial (1995). A review of this study indicates that it partially meets the current guideline (OECD 307); the major deviation is that the soils used were collected from lower soil horizons than specified in the guideline. This study was performed to aid evaluations in the Netherlands and was conducted according to the Dutch Environmental Criteria (1995). The study is a scientifically valid demonstration that IN-N0079 can be degraded in the saturated zone. The study should be considered as higher-tier, supplemental data and is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
Degradation of IN-N0079 under aerobic and anaerobic conditions in subsoils obtained from the saturated zone is rapid. Less than 50% of the IN-N0079 had degraded within 1-45 days of application.

**Study submitted to the EU for the first time in this submission.**

**B.8.2.3/05**

| | | |
|---|---|---|
| **Reference:** **CA 7.2.3/01** | **Report:** | Warren, R.L. (2004); Reductive degradation of oxamyl by ferrous iron<br>**DuPont Report No.:** DuPont-14826 EU<br>**Guidelines:** Not applicable<br>**Deviations:** None<br>**Testing Facility:** DuPont Stine-Haskell Research Center, Newark, Delaware, USA<br>**Testing Facility Report No.:** DuPont-14826 EU<br>**GLP:** No<br>**Certifying Authority:** Laboratories in the USA are not certified by any government agency, but are subject to regular inspections. |

The objective of this position paper was to summarize the most pertinent work (both from open literature and DuPont studies) describing the $Fe^{II}$-reductive degradation of oxamyl. While this degradation process is applicable to saturated zone subsoils, it is also potentially important in the sediment of surface water bodies, which can contain $Fe^{II}$ due to their anoxic/suboxic, reducing nature.

In the early 1980s, it was reported that oxamyl very rapidly degraded in water-saturated subsoils incubated under anaerobic, reducing conditions. The degradation rate was substantially slower in similar subsoils incubated under aerobic, oxidizing conditions, though the cause of this difference was unknown. Later experiments with similar subsoils linked the rapid disappearance of oxamyl to the presence of ferrous iron ($Fe^{II}$), and a reductive degradation mechanism was proposed. A detailed series of experiments evaluating the effect of pH, inorganic and organic ligands, natural organic matter, and mineral surfaces on reduction of oxamyl by $Fe^{II}$

were reported during 2001–2003. These experiments validated earlier findings and fundamentally support the important role that $Fe^{II}$ can play in the environmental degradation of oxamyl. Results from a regulatory study conducted with reducing and oxidizing water-saturated subsoils collected from a variety of agricultural fields were consistent with other observations, further demonstrating the importance of $Fe^{II}$ to the environmental behavior of oxamyl.

Abiotic reduction of oxamyl by $Fe^{II}$ is a net two electron process that results in the formation of the nitrile reduction product (IN-N0079). The reduction process is independent of abiotic hydrolysis, which is strongly related to pH and results in the formation of the oxime hydrolysis product (IN-A2213).

Reduction of oxamyl (5.5 mg/L) by dissolved $Fe^{II}$ (28 mg/L) in pure buffered solution proceeded with a $DT_{50}$ of approximately 6 days at 25°C over the pH range of 2.0 to 6.6. The $DT_{50}$ value decreased as pH increased above 6.6 due to the formation of more reactive solution $Fe^{II}$-hydroxy and solid $Fe^{II}$ species.

The rate of oxamyl reduction by $Fe^{II}$ in the presence of some inorganic (e.g., $CO_3^-$, $PO_4^{-3}$) and organic (e.g., acetate, oxalate, natural organic matter) ligands, and when sorbed to mineral surfaces (e.g., kaolinite, goethite, hematite) was often faster by several orders of magnitude compared to solutions containing $Fe^{II}$ alone. This was due to the formation of various solutions and sorbed $Fe^{II}$ complex species with greater reactivity than uncomplexed $Fe^{II}$. The degree of reactivity was dependent on the ligand or mineral identity and concentration and the pH, all of which can influence $Fe^{II}$ complex formation.

In heterogeneous natural systems, such as saturated zone subsoils and surface water sediments where conditions favor the presence of $Fe^{II}$ (i.e., reducing, anoxic/suboxic), oxamyl degraded rapidly with $DT_{50}$ values of 1 day or less. $DT_{50}$ values were typically a few hours, even at 10°C. Rapid appearance of the nitrile reduction product in acidic reducing systems, appearance of the nitrile and the oxime hydrolysis product in reducing and neutral/basic systems, and appearance of just the oxime in oxidizing neutral/basic systems was consistent with observations in homogenous model systems. The results observed in natural systems were well explained by the independent, parallel reduction and hydrolysis processes of oxamyl degradation.

The position paper DuPont-14826 EU is submitted for the first time in this submission. The paper provides a valuable summary of the available literature data and DuPont investigations regarding the reductive degradation of oxamyl by ferrous iron ($Fe^{II}$). Based on the data presented in this position paper, it is clear that oxamyl reduction by ferrous iron is a relavent degradation mechanism in anoxic/anaerobic environments. This position paper is considered as supplemental information to support the observed degradation of oxamyl in the GLP environmental fate studies presented in this dossier.

### B.8.3 Fate and behaviour in air

#### B.8.3.1 Rate and route of degradation in air

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.3.1/01**

| | | |
|---|---|---|
| **Reference:**<br>-- | **Report:** | Styles, D. A. (2000); Photochemical oxidative degradation of oxamyl<br><br>**DuPont Report No.:** DuPont-4574<br><br>**Guidelines:** OECD Photochemical Oxidative Degradation in the Environment (1987a, 1988a), U.S. EPA 796.3900 (1992) |

1. Test material: Not applicable
   Lot/Batch #: Not applicable
   Purity: Not applicable

**Materials and Methods:**

The purpose of this study was to estimate the half-life in air of oxamyl, resulting from photochemical oxidative degradation by reaction with atmospheric hydroxyl radicals (an indirect photolytic process).

Gas-phase reaction with hydroxyl radicals is generally the most important and most common removal process in the troposphere, and most compounds in the troposphere can be structurally altered by reaction with hydroxyl radicals. However, the susceptibility of organic compounds to indirect photolysis in the atmosphere is difficult to measure experimentally. A widely used alternative is to estimate the half-life in air using the computational method developed by Atkinson.[2,3,4] This was done for oxamyl with a software package known as the Atmospheric Oxidation Program (AOP), version 1.83.[5]

AOP estimates the rate constant for the atmospheric, gas-phase reaction between photochemically produced hydroxyl radicals and organic chemicals using the structure- activity relationship (SAR) methods developed by Atkinson and co-workers. The rate constants estimated by the program are then used to calculate atmospheric half-lives for organic compounds based upon average atmospheric concentrations of hydroxyl radicals. [AOP also estimates the rate constant for the gas-phase reaction between ozone and olefinic/acetylenic compounds.]

**Findings:**

The reaction between hydroxyl radicals and any given organic compound can be characterised by a bimolecular rate constant (denoted kOH here). The results of the SAR analysis performed by AOP for oxamyl, showing the contribution to kOH from hydroxyl radicals reacting with various chemical fragments, are summarised in Table
.

2 Atkinson, R. (1985): Kinetics and mechanisms of the gas-phase reactions of the hydroxyl radical with organic compounds under atmospheric conditions. Chem. Rev. 85: 69-201.

3 Atkinson, R. (1987): A structure-activity relationship for the estimation of rate constants for the gas-phase reactions of OH radicals with organic compounds. Intern. J. Chem. Kinet. 19: 799-828.

4 Atkinson, R. (1988): Estimation of gas-phase hydroxyl radical rate constants for organic chemicals. Environ. Toxicol. Chem. 7: 435-442.

5 Meylan, W. and Howard, P. (1996): User's guide for the Atmospheric Oxidation Program, Version 1.8. Syracuse Research Corporation, Syracuse, NY 13210.

**Table 175 SAR analysis of oxamyl, showing contribution to bimolecular rate constant ($k_{OH}$) from reaction of hydroxyl radicals with various chemical fragments**

| Chemical fragment reaction with hydroxyl radicals | Contribution to $k_{OH}$ |
|---|---|
| Hydrogen abstraction | 3.1960 x $10^{-12}$ $cm^3$/molecule-sec |
| Reaction with N, S and -OH | 19.4000 x $10^{-12}$ $cm^3$/molecule-sec |
| Addition to triple bonds | 0.0000 x $10^{-12}$ $cm^3$/molecule-sec |
| Addition to olefinic bonds | 0.0000 x $10^{-12}$ $cm^3$/molecule-sec |
| Addition to aromatic rings | 0.0000 x $10^{-12}$ $cm^3$/molecule-sec |
| Addition to fused rings | 0.0000 x $10^{-12}$ $cm^3$/molecule-sec |
| Overall $k_{OH}$ value | 22.5960 x $10^{-12}$ $cm^3$/molecule-sec |

Compared to the organic compounds they react with, hydroxyl radicals are likely to be present in the atmosphere to a large excess; meaning that in a reaction their concentration can be regarded as effectively constant. When this assumption is made, the bimolecular gas-phase reaction between hydroxyl radicals and any given organic compound follows pseudo first order kinetics. The half-life for the reaction (t½) can be calculated with the formula

$$t = \ln 2/k_{OH}[OH\cdot]$$

where kOH is the bimolecular rate constant for the reaction with hydroxyl radicals and [OH·] is the concentration of hydroxyl radicals, which is assumed to be constant. ($k_{OH}$[OH·] is the pseudo first order rate constant.)

AOP uses a default value of 1.5 x 106 radicals/cm3 for hydroxyl radical concentration in the atmosphere (diurnal value at 298 K over 12-hour daytime). Therefore, based on a value of 22.5960 x 10-12 cm3/molecule-sec for the bimolecular rate constant kOH, the half-life for the atmospheric gas-phase reaction between oxamyl and hydroxyl radicals is 5.68 hours.

**Conclusions:**

The half-life for the atmospheric gas-phase reaction of oxamyl with hydroxyl radicals was estimated to be 5.68 hours, based on an Atkinson structure-activity relationship analysis performed with the Atmospheric Oxidation Program (assuming a constant hydroxyl concentration of 1.5 x 106 radicals/cm3). Hydrogen abstractions and reactions with nitrogen and sulphur were predicted to contribute to the overall atmospheric photochemical degradation pathway, and the overall bimolecular rate constant for the process (kOH) was calculated to be 22.5960 x 10-12 cm3/molecule-sec.

The route and rate of degradation in air study DuPont-4574, originally submitted under EU Rev8 Point IIA 7.2.2, was conducted under guidelines OECD Photochemical Oxidative Degradation in the Environment (1987a, 1988a) and U.S. EPA 796.3900 (1992). A review of this study indicates that it is a valid Atkinson calculation, which provides reliable estimation of the rate of degradation of oxamyl in the troposphere. This study is relied upon.

**RMS comments and conclusion**

Study was submitted and accepted in the EU Dossier in 2001 and included in the first EU approval for Annex I inclusion of Oxamyl.
The half-life for the atmospheric gas-phase reaction of oxamyl with hydroxyl radicals was estimated to be 5.68 hours

#### B.8.3.2 Transport *via* air

Neither oxamyl nor any of its principal degradation products have significant volatility. The vapour pressure of oxamyl is $1.80 \times 10^{-5}$ Pa at 20°C Pa in accordance with the requirements of CA 2.2 (summarized in the Oxamyl EU Renewal Dossier, Document M-CA, Section 2, DuPont-40929 EU). There is no guidance currently available for conducting meaningful studies regarding the potential breakdown of oxamyl or its degradation products in air.

Further, the Henry's law constant of oxamyl in accordance with the requirements of CA 2.2 is less than $3 \times 10^{-2}$ Pa-m$^3$/mol, suggesting little potential for volatilisation in the environment (summarized in the Oxamyl EU Renewal Dossier, Document M-CA, Section 2, DuPont-40929 EU). Henry's law constants below this value show that the compound is less volatile than water and can be considered essentially non-volatile (Lyman, W.J., 1990).

Given that oxamyl is non-volatile by virtue of its very low vapour pressure and calculated Henry's Law constant, only negligible quantities would be transferred to the troposphere. These negligible quantities would then undergo rapid photochemical oxidative degradation (half-life = 5.68 hours), and therefore, oxamyl does not have the potential for long range transport in air.

#### B.8.3.3 Local and global effects

The vapour pressure and Henry's law constant of oxamyl indicate that it has no potential to volatilise and therefore could not have any global warming potential (GWP), ozone depleting potential (ODP), photochemical ozone creation potential (POCP), or be expected to accumulated in the troposphere. Oxamyl use does not have any acidification potential (AP), because its breakdown will not introduce any known acidifying compounds, such as ammonia, into soils. Oxamyl use will not lead to significant production of nitrates or phosphates and thus has no eutrophication potential (EP).

### B.8.4 Definition of the residue

#### B.8.4.1 Definition of the residue for risk assessment

The environmental metabolites assessed for relevance were IN-A2213, IN-N0079, IN-D2708, IN-T2921, and IN-SBY69. The metabolites IN-A2213, IN-N0079, and IN-D2708 were assessed for relevance in soil, while IN-A2213, IN-N0079, IN-D2708, IN-T2921, and IN-SBY69 were assessed for relevance in surface water. In groundwater, the significance of modelled environmental concentrations of IN-A2213, IN-N0079, and IN-D2708 were considered. With regard to the air compartment, no metabolites needed to be considered for relevance because of lack of volatility. The reasons for choosing metabolites for assessment of relevance are outlined below, for each environmental compartment. Then the metabolites are assessed, on a case by case basis, for relevance in the environmental compartments where there may be present.

**Selection of metabolites for assessment of relevance in soil.**

On the basis of the described soil metabolism studies, the primary route of degradation for oxamyl is *via* microbial decomposition and hydrolysis to form IN-A2213, which is further microbially degraded to IN-D2708 and then rapid mineralization to $CO_2$ and bound residue. In the presence of light, oxamyl can also undergo a photocatalysed, iron-mediated conversion to IN-N0079 as an additional minor degradation route. Degradation *via* this alternate, light-mediated route does also proceed to formation of IN-D2708, which can then undergo microbial mineralization (see pathway in Figure 3).

In general, carbon dioxide was the principal degradation product. It was detected in the primary laboratory aerobic soil metabolism study at levels up to 108.5% of applied radioactivity. IN-A2213 was detected in the laboratory aerobic soil metabolism studies at levels up to 51% of applied radioactivity, and in the laboratory anaerobic soil metabolism study at levels up to 69.5% AR. The metabolite IN-D2708 was also a prominent soil metabolite, being detected in the laboratory aerobic soil metabolism studies, the anaerobic soil study, and the soil photolysis study at maximum levels of 78.0, 23.1, and 45.4% of applied radioactivity respectively. The metabolite IN-N0079 was not observed in any laboratory aerobic or anaerobic soil study, but was a major metabolite in the soil photolysis study (max 10.2 % AR). Therefore, IN-A2213, IN-D2708 and IN-N0079 are considered relavant for the residue definition for risk assessment in soil.

**Selection of metabolites for assessment of relevance in water**

The results of the experimental investigations on the fate and behaviour of oxamyl in water indicate that oxamyl is readily hydrolysed to IN-A2213 in neutral and basic waters. Based on its lack of absorbance above 290 nm, oxamyl does not undergo direct photolysis, but the results of the laboratory aqueous photolysis demonstrate that oxamyl can be transformed to IN-N0079 in the presence of dissolved ferrous iron ($Fe^{2+}$); this catalytic reaction however, is only favourable under reductive conditions. In the water/sediment studies, oxamyl degraded rapidly in the water phase (<50% remaining on day 1), and significant amounts were never found in the sediment phase. The hydrolysis product, IN-A2213, reached a maximum of 25.3–48.8% AR in the water phase on Day 2 and

then decreased to non-detectable levels by Day 61. Low levels were found in the sediments (max. 4.4%), and the pattern reflected that observed in the water phase. In one system, high levels of IN-N0079 (maximum 52.9% at Day 2) were observed in the water phase, and the pattern of formation and decline was parallel to that of IN-A2213. As discussed above, the rapid appearance of significant amounts of IN-N0079 was likely due to the $Fe^{+2}$-oxamyl reduction reaction with $Fe^{+2}$ near or within the anaerobic sediment phase. Following the decline of the IN-A2213 and IN-N0079, levels of IN-D2708 in the water subsequently rose (maximum 64.2–66.8% at Day 30) and then declined during the remainder of the study. Low levels of IN-D2708 (maximum 10.4–12.1%) were observed in the sediment, and the pattern of occurrence of this sediment residue coincided with the maximum level of IN-D2708 in the water phase, suggesting a simple gradient diffusion of IN-D2708 from the water phase into the sediment pore water in the static test system. In one system only, levels of IN-T2921 exceeded 10% in the water phase. However, this was only at one time point (Day 14), the maximum level was only 11.4%, and subsequent degradation was rapid (not detected at the next time point, Day 30). Therefore, IN-T2921 is not considered relevant for surface water risk assessment. Amounts of carbon dioxide evolved reached 27.9–60.9% by the end of the study. In anaerobic water/sediment systems, oxamyl degraded similarly as in the aerobic system, with IN-A2213, IN-N0079, and IN-D2708 being the major transformation products. In one sediment system under anaerobic conditions, IN-SBY69 was observed as an additional transient metabolite. This metabolite is formed *via* cysteine conjugate cyclization of IN-N0079. It is believed that IN-SBY69 was observed in the anaerobic water/sediment study only because the anaerobic conditions create a reductive environment that favours the $Fe^{+2}$-oxamyl reduction reaction that produces its precursor, IN-N0079. Thus, this metabolite (IN-SBY69) was only present at major, observable levels because of the drastically increased levels of IN-N0079 formed in this one anaerobic sediment system. In typical water/sediment systems, such high levels of IN-N0079 are not expected to form, and thus only minor, if any, levels of IN-SBY69 can be expected to occur in the environment. Therefore, IN-SBY69 is not considered relevant for risk assessment.

Based on the degradation profile of oxamyl in water, IN-A2213, IN-D2708 and IN-N0079 are considered relavant for the residue definition for risk assessment in water.

**Selection of metabolites for assessment of relevance in air**

Given that oxamyl is non-volatile by virtue of its very low vapour pressure and calculated Henry's Law, no metabolites were considered for relevance in air.

### B.8.4.2 Definition of the residue for monitoring

The oxamyl metabolites IN-A2213 and IN-D2708 were observed as the major degradates in the laboratory soil studies. IN-N0079 was observed as a major metabolite in the soil photolysis study. IN-A2213 was observed as the only major degradate in hydrolysis and only IN-N0079 was observed above 10% AR in the aqueous photolysis study. IN-A2213, IN-D2708, IN-N0079, and IN-T2921 were each observed as major degradates in the water phase of the water/sediment study. Only IN-D2708 was observed as a major metabolite in the sediment phase of the water/sediment study. The soil and water degradation products listed above have been tested for insecticidal activity, mammalian toxicological and ecotoxicological activity and should be considered non-relevant based on the following:

1. IN-A2213, IN-D2708, IN-N0079, and IN-T2921 were shown to be insecticidally inactive against five agricultural species of insects at rates equivalent to or exceeding the parent molecule on a molar basis (Oxamyl EU Renewal Dossier, Document M-CA, Section 8, DuPont-40935 EU).
2. IN-A2213, IN-N0079, and IN-D2708 have been observed in their free or conjugated forms in metabolism studies performed with oxamyl in rats and mice. IN-T2921 was not detected in either the rat or the mouse; however, this substance is a proposed intermediate in the formation of IN-D2708 from IN-N0079 and was identified in goat urine. Therefore, it can be assumed that all of the degradation products were present in oxamyl-exposed laboratory animals, and that any toxicological effects at biologically relevant levels have been expressed. Results from the animal studies with oxamyl should reflect the toxicological properties of the soil, water, and water/sediment degradates, and thus, these degradation products are not of toxicological concern. In addition, a number of toxicity studies have been conducted with IN-A2213, IN-N0079, and IN-D2708. The acute oral toxicity of these substances is relatively low in comparison to the parent molecule. In addition, IN-N0079 has been evaluated in rats for subacute and subchronic toxicity (10-day and 90-day exposures, respectively) effects on reproduction, and mutagenic activity in bacteria. The NOAEL in the 90-day feeding study was 50 ppm (4.0 mg/kg bw/day) based on body weight and RBC effects at higher dose levels (150 and 450 ppm). No reproductive effects were noted with the exception that pup weights

were reduced at the highest dose level (450 ppm) where there was clear parental toxicity. IN-N0079 was negative in the bacterial mutagenicity assay at concentrations as high as 10000 μg/plate with and without metabolic activation.

3. IN-A2213, IN-D2708, IN-N0079, and IN-T2921 have been tested on algae, daphnia, and fish and have been shown to have low to negligible effects, even at doses higher than those expected to occur in the environment (Oxamyl EU Renewal Dossier, Document M-CA, Section 8, DuPont-40935 EU, Points CA 8.2.1, 8.2.2, and CA 8.2.2.3). IN-A2213, IN-D2708, and IN-N0079 were also tested in chronic earthworm, collembolan, and soil microflora studies at rates exceeding the highest expected soil concentration (5X to 10X) and only minor effects were observed with IN-N0079 and collembola at soil concentrations >10X above PECsoil (Oxamyl EU Renewal Dossier, Document M-CA, Section 8, DuPont-40935 EU, Points CA 8.2, 8.4, and CA 8.5).

Therefore, oxamyl alone is considered the definition of the residue for monitoring.

### B.8.5 Monitoring data concerning fate and behaviour of the active substance, metabolites, degradation and reaction products

Environmental fate data available for oxamyl regarding these endpoints in the open literature were reviewed and found not to be relevant to the risk assessment in the context of this assessment. A reference for the article reviewed can be found in Table .

National groundwater monitoring data from the Environmental Agency in England and Wales has been compiled for oxamyl. The magnitudes of the detections of oxamyl from 2005 through 2011 are summarized in the following table.

**Table 176Details on groundwater monitoring data provided by Environmental Agency in England and Wales in 2010/2011**

| Dataset | Date | Samples[a] | Location | Results [μg/L] |
|---|---|---|---|---|
| 1 | Jan 2010–Jun 2010 | 458 | Across England and Wales | all <0.005 |
| | Mar 2005–Nov 2005 | 4 | Agrevo abstraction B/H 2 Hauxton | 2 <0.05<br>1 <0.074<br>1 <0.099 |
| | Feb 2006–Nov 2006 | 4 | Agrevo abstraction B/H 2 Hauxton | all <0.05 |
| | Nov 2009–Dec 2009 | 2 | Across England and Wales | all <0.005 |
| 2 | May 2010–Dec 2010 | 1595 | Across England and Wales<br>Horticulture Research Intern. Groundwater F94/11 | 1594 <0.005<br><br>1 <0.03 |
| | Jan 2011–Jun 2011 | 705 | Across England and Wales | all <0.005 |

[a] Data contain Environment Agency information "© Environment Agency and database right"

The following two position papers (DuPont-31291 UK and DuPont-33725 UK) analyse the vulnerability of these monitoring sites with respect to the site hydrology, soil pH, and the environmental properties of oxamyl. The results demonstrate that many of the sampling boreholes can be classified as vulnerability for oxamyl leaching from potato uses. In particular, soil pH at the monitoring sites was analysed since the rapid hydrolytic degradation of oxamyl does not occur in acidic soils. However, the data from the monitoring database clearly illustrate that significant levels of oxamyl were not detected at any of the identified vulnerable sampling sites. Thus, these monitoring data demonstrate that the risk of oxamyl leaching to groundwater following typical use on potatoes in the U.K. regions is low and not pH dependent.

**Study submitted to the EU for the first time in this submission.**

**B.8.5/01**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.5/01** | **Report:** | Hollis, J.M., Holman, I.P., Truckell, I. (2010); Derivation of data on the soil pH, leaching potential and hydrogeological conditions at selected groundwater abstraction borehole sites in the UK where potatoes are likely to be a significant crop |

| | | |
|---|---|---|
| | | **DuPont Report No.:** DuPont-31291 UK<br><br>**Guidelines:** Not applicable<br><br>**Deviations:** None<br><br>**Testing Facility:** Pesticide Environmental Fate (UK), St. Albans, Herts, UK<br><br>**Testing Facility Report No.:** DuPont-31291 UK<br><br>**GLP:** No<br><br>**Certifying Authority:** Not given |

**Executive summary:**

The purpose of this study was to identify a limited number of sites within a set of 468 Environment Agency groundwater abstraction boreholes where monitoring of oxamyl has been carried out in 2010, where the local conditions are likely to make them most vulnerable to leaching of the compound. This was achieved firstly by using National level cropping, soil, and hydrogeological spatial data to select two groups of 'potentially vulnerable' boreholes as follows:

- Top priority sites that have, within a 1 km radius around the borehole, at least 3% of land under potatoes and at least 20% coverage of soils in high leaching classes and with a pH class 5.6–6.5;
- Other vulnerable sites that have, within a 1 km radius around the borehole, at least 1% of land under potatoes and at least 10% coverage of soils in high leaching classes and with a pH class 5.6–6.5.

This gave a total of 31 potentially vulnerable boreholes that are distributed across all the main potato growing areas in England and Wales boreholes apart from those around the Wash, which has no boreholes but is a non-aquifer area anyway, the south west Lancashire coastal plain, Pembrokeshire, Cornwall, and Sussex, all of which have no boreholes in potato growing areas, and a small area in north east Kent that has few if any soils in the high leaching risk classes and no soils in the pH ranges <6.5.

Secondly, for each of the 31 identified potentially vulnerable boreholes, primary source data was used to identify the most likely pH in both the soil and aquifer groundwater within the 1 km radius area of interest around the borehole. This more detailed investigation indicated that the 31 boreholes could be separated into two broad groups: One in which the 1 km radius area of interest around boreholes was likely to have significant amounts of land under potatoes with soil pH values between 5.5 and 6.5; One where such areas were unlikely to have significant amounts of land under potatoes with soil pH <6.5.

The most vulnerable boreholes occur in the West Midlands, The Sherwood Forest area of Nottinghamshire, and west Yorkshire, where potatoes are grown on coarse textured soils, and the boreholes exploit aquifers in Permo-Triassic sandstone, 'Magnesian' limestone, or Carboniferous Millstone Grit sandstone. Local groundwater quality sampling data for all these aquifers show that a small but significant percentage of samples have a pH <6.5, and a significant proportion of the overlying coarse textured soils are also likely to have pH <6.5. In some of these boreholes however, slowly permeable or impermeable deposits underlie the coarse textured soils and are thus likely to afford some protection to the underlying aquifer. Using the borehole data, the NSI pH data, and the groundwater quality data, it is estimated that up to 1.5% of the main potato growing areas in England and Wales are likely to be in the most vulnerable areas for leaching and have a pH of <6.5 in both the soil and underlying aquifer groundwater.

Slightly less vulnerable boreholes occur on sandstone aquifers of the 'Barren' Upper Coal Measures in the West Midlands and the Permo-Triassic rocks in South Yorkshire. Here again, significant amounts of potatoes are grown on coarse textured soils, a significant proportion of which are likely to have a pH <6.5. However, local groundwater sampling in the underlying aquifers indicates that pH is unlikely to be much less than 6.5, and these boreholes are thus less vulnerable than those described above. There are also some boreholes where slowly permeable or impermeable deposits underlie the coarse textured soils and afford protection for the underlying aquifer.

Most other potentially vulnerable boreholes exploit aquifers in East Anglia. Although they usually have the largest amounts of potatoes grown on soils with high leaching class, they constitute the least vulnerable group of the 31 potentially vulnerable boreholes. This is because all their soils have pH >6.5 and usually >7.0, except for some peat areas on the edge of the Fens that may have pH <6.5. Even for these soils however, their peat nature is likely to reduce leaching and thus vulnerability. In addition, many of the underlying aquifers are in the chalk formations that have pH greater than 6.5 and usually at least 7.0. Those boreholes exploiting aquifers in the Pleistocene 'Crag' formation are likely to have the greatest vulnerability in the East Anglia area because groundwater quality sampling indicates that a small percentage (about 5%) of samples have pH <6.5.

A single borehole (No. 63) in Lincolnshire is also assessed to be the least vulnerable of the potentially vulnerable boreholes. This is likely to exploit a deep aquifer in the Jurassic 'Oolitic' limestone formation that is protected by overlying impermeable clay deposits but in any case is unlikely to have groundwater pH <6.5 and the surface soils on which potatoes are grown all have pH >7.0.

**Study submitted to the EU for the first time in this submission.**

**B.8.5/02**

| | | |
|---|---|---|
| **Reference:**<br>**CA 7.5/02** | **Report:** | Hollis, J.M., Holman, I.P., Truckell, I. (2011); Identification of potato growing areas in England & Wales most likely to be vulnerable to groundwater leaching of oxamyl, taking into account pH-dependent degradation<br><br>**DuPont Report No.:** DuPont-33725 UK<br><br>**Guidelines:** Not applicable<br><br>**Deviations:** None<br><br>**Testing Facility:** Pesticide Environmental Fate (UK), St. Albans, Herts, UK<br><br>**Testing Facility Report No.:** DuPont-33725 UK<br><br>**GLP:** No<br><br>**Certifying Authority:** Not given |

**Executive summary:**

The purpose of this study was to extend the results of project DuPont-31291 UK by using national soil hydrogeological and cropping databases, together with an Environment Agency borehole monitoring database for May 2010 to June 2011 and a confidential DuPont database of soil pH at farms using oxamyl, to identify and rank different potato growing areas in England and Wales according to their vulnerability to leaching of oxamyl to groundwater.

Initially, the national soil and cropping databases were used to identify and characterize 49 different regions of the main potato growing areas in England and Wales where soil and growing conditions are similar. The database of soil pH at farms using oxamyl was then used to characterize the range of pH in each of the different potato growing regions and to prioritize them according to their vulnerability to leaching of oxamyl, taking into account pH-dependent soil degradation. Of the 49 regions, 12 had already been assessed in terms of the soil pH and hydrogeological conditions at representative boreholes (Study DuPont-31291 UK), 3 contain no boreholes or fields with data and thus cannot be assessed, 8 have no fields with data and are assessed as being minor usage areas for oxamyl, whilst 5 are assessed as being of low risk because all their field pH values are >7.0. This left 21 areas as having, at least in some part, field pH values under potatoes that could indicate vulnerability to leaching, based on pH dependent hydrolytic degradation.

In each of these areas, the Environment Agency borehole monitoring database was used to select one or more boreholes that were located close to a field that the farm usage database showed to have one of the lowest pH values in that area. This selection resulted in 26 boreholes around which their local hydrogeological

characteristics were identified using the sources available to the project. Hydrogeological conditions at two additional boreholes were also identified because, although not in a main potato growing areas, they were the only boreholes that showed a positive detection of oxamyl (albeit at concentrations <0.1 μg $L^{-1}$) during the 2010 to 2011 monitoring seasons.

Based on the assessments carried out in this and the previous study (DuPont-31291 UK), each of 49 potato growing areas were grouped according to their vulnerability to leaching of oxamyl. Vulnerability was assessed according to the likely connectivity between soil leachate and aquifer groundwater and the measured range of pH within field topsoils and aquifer groundwaters.

Vulnerability in three of the areas could not be assessed, because they did not contain any boreholes from the monitoring network. It may be that the lack of borehole monitoring data in these areas reflects the fact they do not overlie any major aquifers and only locally overlie minor aquifers. Two of the areas (29 and 41) are relatively minor potato growing areas, whereas the other (8) is dominated by calcareous soils with pH >7.0. The groundwater vulnerability of these areas could thus be contextualized from their limited aquifer potential and available usage data. Of the 46 other areas, 26 were assessed as being most vulnerable, with a further 9 being less vulnerable, either because connectivity between any soil leachate and the aquifer groundwater is unlikely, or because all their measured topsoil pH values were greater than 7.0. The remaining 8 potato growing areas have insignificant use of oxamyl.

None of the Environment Agency groundwater monitoring sites within the 46 potato growing areas that were able to be assessed showed any detection of oxamyl.

Neither of the two monitoring sites where oxamyl was detected was in an area where potatoes are a significant crop. At one site (SP2704356489), connectivity between soil leachate and aquifer groundwater was assessed as being unlikely. At the other site (TL4334052500), connectivity is likely, but the local soils are all calcareous, whilst the aquifer groundwater has no measured pH values below 6.6 and few below 7.0. It may be significant that both the sites are located at what are (or were) agrochemical experimental facilities, one for a commercial company and one for Horticultural Research.

**Study submitted in the EU Dossier in 2001 and included in the first EU approval review.**

**B.8.5/03**

| | | |
|---|---|---|
| **Reference:**<br>-- | **Report:** | Dust, M. (1999); Synopsis of groundwater monitoring data for oxamyl in the UK taken from the national database of pesticide monitoring in the environment 1992-97 compiled by the Environment Agency, National Centre for Ecotoxicology and Hasardous Substances, Wallingford, Oxfordshire, UK<br>**DuPont Report No.:** DuPont-3145<br>**Guidelines:** Not applicable |

1. Test material: Not applicable
   Lot/Batch #: Not applicable
   Purity: Not applicable

**Material and Methods:**

Groundwater monitoring data have been obtained from the UK Environment Agency database for the years 1992-1997. These data are generally obtained in response to particular concerns and incidents and are not the result of a detailed random monitoring program. Therefore, they do not provide a comprehensive picture of oxamyl concentrations in groundwater, but are likely to show the worst case situations.

Two regions have been monitored for oxamyl in groundwater, namely Anglia and Wales. As oxamyl is used on potatoes and sugar beet, it is unlikely that there has been significant use of the compound in the Welsh region. However, the Anglia region is a major potato and sugar beet growing area and, therefore, a link between oxamyl use and oxamyl groundwater monitoring data exists at the regional level in this case.

A total of 169 samples were analysed from the Anglia region, where oxamyl use is likely, and 26 from the Welsh region, where oxamyl use is less likely. The method detection limit was <0.1 µg/L for all samples analysed, except one from the Anglia region where the detection limit was reported as <0.25 µg/L.

**Findings:**

The results of the groundwater monitoring for oxamyl in Anglia and Wales during the period 1992-1997 are shown in Table 51. Oxamyl was not detected (<0.1 µg/L) in any of the samples from the Welsh region. Only three of 169 samples from the Anglia region were reported at levels exceeding 0.1 µg/L (0.18, 0.329 and 0.541 µg/L). It should be noted that these samples represent a single timepoint, not an annual average, which is likely to be lower than single timepoint samples collected during the growing season.

**Table 51 Groundwater monitoring data for oxamyl**

| Year | Number of samples analysed | Number of samples exceeding 0.1 µg/l | Concentrations in samples >0.1 µg/l |
|---|---|---|---|
| Anglia | | | |
| 1992 | 78 | 0 | - |
| 1993 | 29 | 0 | - |
| 1994 | 6 | 0 | - |
| 1995 | 8 | 0 | - |
| 1996 | 11 | 3 | 0.18, 0.329, 0.541 |
| 1997 | 37 | 0 | - |
| Wales | | | |
| 1996 | 8 | 0 | - |
| 1997 | 18 | 0 | - |

One of the samples for 1995 had a reported detection limit of 0.25 µg/L; thus it can only be established that oxamyl, if present in this sample, was lower than the reported detection limit.

**Conclusions:**

The overall conclusion from the results of the groundwater monitoring data is that oxamyl is not likely to adversely impact groundwater under actual use conditions.

The monitoring data study DuPont-3145 was originally submitted under EU Rev8 Point IIA 7.4. Guidelines were not applicable. A review of this study indicates that it is a valid review of available monitoring data, and it is relied upon.

**RMS comments and conclusion**

The overall conclusion from the results of the groundwater monitoring data is that oxamyl is not likely to adversely impact groundwater under actual use conditions

### B.8.6 References relied on

Studies marked in yellow are submitted for the first time.

List of information, tests and studies which are considered as relied upon by the RMS for the evaluation with a view to the approval of the active substance.

## Sorted by Annex Point

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.1.1/01 | Smyser, B.P., Mattson, S.L. | 2000 | Route of degradation of oxamyl in aerobic soil<br>DuPont Experimental Station<br>DuPont-2958<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.1/02 | Mattson, S.L., Smyser, B.P. | 2000 | Rate of degradation of oxamyl in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2957<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.1/03 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.1/04 | Clark, B. | 2015 | Aerobic rate of degradation of [$^{14}$C]-DPX-D1410 (oxamyl) in four acidic soils<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-39014<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.1.2/01 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.2/02 | Spare, W.C. | 1992 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil, supplemental report<br>Agrisearch, Inc<br>AMR 1851-90, Supplement No. 1<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.1.3/01 | Habeeb, S.B. | 2011 | Photodegradation of $^{14}$C-oxamyl on soil<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-31501<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.2.1.1/01 | Mattson, S.L., Smyser, B.P. | 2000 | Rate of degradation of oxamyl in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2957<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.1.1/02 | Smyser, B.P., Mattson, S.L. | 2000 | Route of degradation of oxamyl in aerobic soil<br>DuPont Experimental Station<br>DuPont-2958<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.1/03 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.1/04 | Clark, B. | 2015 | Aerobic rate of degradation of [$^{14}$C]-DPX-D1410 (oxamyl) in four acidic soils<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-39014<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.2.1.1/05 | Ghafoor, A., Zillgens, B. | 2015 | Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) from laboratory soil degradation studies<br>Dr. Knoell Consult GmbH<br>DuPont-41859 EU<br>GLP: No<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.1.2/01 | Swain, R.S., Anderson, J. J. | 2000 | Rates of degradation of oxamyl metabolite [$^{14}$C](dimethylamino) oxoacetic acid (IN-D2708) in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2675<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.2/02 | Hashinger, B.M., III, Gaddamidi, V. | 2000 | Rates of degradation of oxamyl metabolite [$^{14}$C]dimethylcarbonocyanidic amide [IN-N0079] in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2674<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.2/03 | Ghafoor, A., Zillgens, B. | 2015 | Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) from laboratory soil degradation studies<br>Dr. Knoell Consult GmbH<br>DuPont-41859 EU<br>GLP: No<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.1.3/01 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.4/01 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.4/02 | Spare, W.C. | 1992 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil, supplemental report<br>Agrisearch, Inc<br>AMR 1851-90, Supplement No. 1<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.2.2.1/01 | Zietz, E. | 2002 | Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10G to bare soil at sites in a typical potato growing region of England – Season 2000<br>DuPont Report No.: DuPont-3026<br>GLP: n/a<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.2.1/02 | Mol, J.G.J. | 2002 | Field soil dissipation study of oxamyl in The Netherlands<br>DuPont Report No.: DuPont-2815<br>GLP: n/a<br>Published: No | N | N | | DuPont |
| B.8.1.2.2.1/03 | LeNoir, J.S., Zietz, E. | 2003 | Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10 L by means of drip irrigation to cucurbits in a greenhouse in Spain, season 2000<br>DuPont Stine-Haskell Research Center, Newark, Delaware, USA<br>**DuPont Report No.:** DuPont-4719<br>GLP: Yes<br>Published: No | N | N | | DuPont |
| B.8.1.2.2.1/04 | Zietz, E. | 2002 | Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10 L by means of drip irrigation to cucurbits in a greenhouse in Italy - Season 2000<br>Institut Fresenius Chemische und Biologische/Gmbh<br>DuPont-4800<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP . | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.2.1/05 | Zietz, E. | 2003 | Determination of the storage stability of oxamyl (DPX-D1410) and its metabolite oxamyl oxime (IN-A2213) in soil samples from a field soil dissipation study conducted in the United Kingdom, season 2000<br>Institut Fresenius Chemische und Biologische Laoratorien AG<br>DuPont-10099<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP . | DuPont |
| B.8.1.2.2.1/06 | Mol, J.G.J. | 2003 | Freezer storage stability of oxamyl, IN-A2213 and IN-D2708 residues in soil samples from field soil dissipation studies conducted in Spain and Italy, season 2000<br>TNO Nutrition and Food Research<br>DuPont-9342<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP . | DuPont |
| B.8.1.2.2.1/07 | Partsch, S., Zillgens, B. | 2015 | Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213) and DMOA (IN-D2708) from field soil dissipation studies<br>Dr. Knoell Consult GmbH<br>DuPont-41859 EU, Supplement No. 1<br>GLP: No<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.3.1.1/01 | Ohm, M.B. | 2001 | Adsorption/desorption of $^{14}$C-oxamyl in five soils<br>DuPont Experimental Station, DuPont Stine-Haskell Research Center<br>DuPont-3166, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.3.1.1/02 | Malin, J.N. | 2015 | Adsorption/desorption of $^{14}$C-oxamyl in five soils<br>DuPont Experimental Station, DuPont Stine-Haskell Research Center<br>DuPont-3166, Revision No. 2<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.3.1.1/03 | Allan, J. | 2012 | [$^{14}$C]DPX-D1410: Batch equilibrium (adsorption/desorption) in six soils<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-33692<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.3.1.2/01 | Berg, D.S. | 2000a | Adsorption/desorption of [$^{14}$C]IN-A2213 in five soils<br>DuPont Experimental Station<br>DuPont-3929, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.3.1.2/02 | Berg, D.S. | 2000b | Adsorption/desorption of [$^{14}$C] IN-D2708 (a metabolite of oxamyl) in five soils<br>DuPont Experimental Station<br>DuPont-3930, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.3.1.2/03 | Berg, D.S. | 2000c | Adsorption/desorption of [$^{14}$C] IN-N0079 in five soils<br>DuPont Experimental Station<br>DuPont-3931<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.1/01 | Clark, B. | 2014 | Hydrolysis of [$^{14}$C]-DPX-D1410 (oxamyl) as a function of pH<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-39015<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.1.1/02 | Lee, D.Y., Berg, D.S. | 2001a | Hydrolysis of IN-A2213 in buffer solutions of pH 4, 7, and 9<br>DuPont Stine-Haskell Research Center<br>DuPont-4024<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.2.1.1/03 | Lee, D.Y., Berg, D.S. | 2001b | Hydrolysis of IN-D2708 in buffer solutions of pH 4, 7, and 9<br>DuPont Stine-Haskell Research Center<br>DuPont-4388<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.1/04 | Lee, D.Y. | 2001 | Hydrolysis of IN-N0079 in buffer solutions of pH 4, 7, and 9<br>DuPont Stine-Haskell Research Center<br>DuPont-4389, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.1/05 | Van-Nguyen, A., Theilacker, W.M. | 2001 | Hydrolysis of IN-T2921 in buffer solutions of pH 4, 7, and 9<br>DuPont Report No.: DuPont-4390<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/01 | Hall, L. | 2014 | Aqueous photolysis of [$^{14}$C] DPX-D1410 (oxamyl)<br>ABC Laboratories, Inc.<br>DuPont-38008 Revision No. 1<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.2.1.2/02 | Tuffy, C. | 2000a | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-A2213<br>ABC Laboratories Europe Ltd<br>DuPont-4514<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/03 | Tuffy, C. | 2000b | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-D2708<br>ABC Laboratories Europe Ltd<br>DuPont-4515<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/04 | Tuffy, C. | 2000c | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-N0079<br>ABC Laboratories Europe Ltd<br>DuPont-4516<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/05 | Tuffy, C. | 2000d | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-T2921<br>ABC Laboratories Europe Ltd<br>DuPont-4517<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.2.2.1/01 | Barnes, S.P. | 2001 | Oxamyl (DPX-D1410): Assessment of ready biodegradability by modified Sturm test<br>Huntingdon Life Sciences Ltd<br>DuPont-6650<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.2.2/01 | Allan, J. | 2015 | Oxamyl (DPX-D1410): Aerobic mineralization in surface water<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-40441<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.2.3/01 | Spare, W.C. | 1995 | Degradability and fate of [1-$^{14}$C]oxamyl in water/sediment systems<br>Agrisearch, Inc<br>AMR 3143-94<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.2.3/02 | Ghafoor, A., Zillgens, B. | 2015 | Estimation of kinetic endpoints of oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) and IN-T2921 in water/sediment systems – Kinetic calculations following FOCUS kinetics guidelines<br>Dr. Knoell Consult GmbH<br>DuPont-44046 EU<br>GLP: No<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.2.2.3/03 | Clark, B. | 2013 | Anaerobic aquatic metabolism of [$^{14}$C]-DPX-D1410 (oxamyl) in two water-sediment systems<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-34157<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.3/01 | Dean, G.M. | 2000 | Route and rate of oxamyl degradation in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-2635<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | |
| B.8.2.3/02 | Mellor, S.J. | 2000 | Route and rate of degradation of oxamyl metabolite IN-A2213 in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-3098<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.3/03 | Shaw, D. | 2000 | Route and rate of degradation of oxamyl metabolite IN-D2708 in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-3097<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.2.3/04 | Millais, A.J. | 2000 | Route and rate of degradation of oxamyl metabolite IN-N0079 in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-3070<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.3/05 | Warren, R.L. | 2004 | Reductive degradation of oxamyl by ferrous iron<br>DuPont Stine-Haskell Research Center<br>DuPont-14826 EU<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.3.1/01 | Styles, D. A. | 2000 | Photochemical oxidative degradation of oxamyl<br>DuPont Stine-Haskell Research Center<br>DuPont-4574<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.5/01 | Hollis, J.M., Holman, I.P., Truckell, I. | 2010 | Derivation of data on the soil pH, leaching potential and hydrogeological conditions at selected groundwater abstraction borehole sites in the UK where potatoes are likely to be a significant crop<br>Pesticide Environmental Fate (UK)<br>DuPont-31291 UK<br>GLP: No<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.5/02 | Hollis, J.M., Holman, I.P., Truckell, I. | 2011 | Identification of potato growing areas in England & Wales most likely to be vulnerable to groundwater leaching of oxamyl, taking into account pH-dependent degradation<br>Pesticide Environmental Fate (UK)<br>DuPont-33725 UK<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.5/03 | Dust, M. | 1999 | Synopsis of groundwater monitoring data for oxamyl in the UK taken from the national database of pesticide monitoring in the environment 1992-97 compiled by the Environment Agency, National Centre for Ecotoxicology and Hasardous Substances, Wallingford, O<br>DuPont de Nemours (France) S.A. European Research and Development Centre<br>DuPont-3145<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

## Sorted by Author

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|---|---|---|---|---|---|---|---|
| B.8.1.3.1.1/03 | Allan, J. | 2012 | [$^{14}$C]DPX-D1410: Batch equilibrium (adsorption/desorption) in six soils<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-33692<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.2.2/01 | Allan, J. | 2015 | Oxamyl (DPX-D1410): Aerobic mineralization in surface water<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-40441<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.2.1/01 | Barnes, S.P. | 2001 | Oxamyl (DPX-D1410): Assessment of ready biodegradability by modified Sturm test<br>Huntingdon Life Sciences Ltd<br>DuPont-6650<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.3.1.2/01 | Berg, D.S. | 2000a | Adsorption/desorption of [$^{14}$C]IN-A2213 in five soils<br>DuPont Experimental Station<br>DuPont-3929, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.3.1.2/02 | Berg, D.S. | 2000b | Adsorption/desorption of [$^{14}$C] IN-D2708 (a metabolite of oxamyl) in five soils<br>DuPont Experimental Station<br>DuPont-3930, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.3.1.2/03 | Berg, D.S. | 2000c | Adsorption/desorption of [$^{14}$C] IN-N0079 in five soils<br>DuPont Experimental Station<br>DuPont-3931<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.1/04 | Clark, B. | 2015 | Aerobic rate of degradation of [$^{14}$C]-DPX-D1410 (oxamyl) in four acidic soils<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-39014<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.2.1.1/04 | Clark, B. | 2015 | Aerobic rate of degradation of [$^{14}$C]-DPX-D1410 (oxamyl) in four acidic soils<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-39014<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.2.1.1/01 | Clark, B. | 2014 | Hydrolysis of [$^{14}$C]-DPX-D1410 (oxamyl) as a function of pH<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-39015<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.2.3/03 | Clark, B. | 2013 | Anaerobic aquatic metabolism of [$^{14}$C]-DPX-D1410 (oxamyl) in two water-sediment systems<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-34157<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.3/01 | Dean, G.M. | 2000 | Route and rate of oxamyl degradation in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-2635<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.5/03 | Dust, M. | 1999 | Synopsis of groundwater monitoring data for oxamyl in the UK taken from the national database of pesticide monitoring in the environment 1992-97 compiled by the Environment Agency, National Centre for Ecotoxicology and Hasardous Substances, Wallingford, O<br>DuPont de Nemours (France) S.A. European Research and Development Centre<br>DuPont-3145<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.1/05 | Ghafoor, A., Zillgens, B. | 2015 | Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) from laboratory soil degradation studies<br>Dr. Knoell Consult GmbH<br>DuPont-41859 EU<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.2/03 | Ghafoor, A., Zillgens, B. | 2015 | Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) from laboratory soil degradation studies<br>Dr. Knoell Consult GmbH<br>DuPont-41859 EU<br>GLP: No<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.2.2.3/02 | Ghafoor, A., Zillgens, B. | 2015 | Estimation of kinetic endpoints of oxamyl and its metabolites oxamyl oxime (IN-A2213), DMOA (IN-D2708), DMCF (IN-N0079) and IN-T2921 in water/sediment systems – Kinetic calculations following FOCUS kinetics guidelines<br>Dr. Knoell Consult GmbH<br>DuPont-44046 EU<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.1.1.3/01 | Habeeb, S.B. | 2011 | Photodegradation of $^{14}$C-oxamyl on soil<br>ABC Laboratories, Inc. (Missouri)<br>DuPont-31501<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.1.2/01 | Hall, L. | 2014 | Aqueous photolysis of [$^{14}$C] DPX-D1410 (oxamyl)<br>ABC Laboratories, Inc.<br>DuPont-38008 Revision No. 1<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.2.1.2/02 | Hashinger, B.M., III, Gaddamidi, V. | 2000 | Rates of degradation of oxamyl metabolite [$^{14}$C]dimethylcarbonocyanidic amide [IN-N0079] in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2674<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.5/01 | Hollis, J.M., Holman, I.P., Truckell, I. | 2010 | Derivation of data on the soil pH, leaching potential and hydrogeological conditions at selected groundwater abstraction borehole sites in the UK where potatoes are likely to be a significant crop<br>Pesticide Environmental Fate (UK)<br>DuPont-31291 UK<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.5/02 | Hollis, J.M., Holman, I.P., Truckell, I. | 2011 | Identification of potato growing areas in England & Wales most likely to be vulnerable to groundwater leaching of oxamyl, taking into account pH-dependent degradation<br>Pesticide Environmental Fate (UK)<br>DuPont-33725 UK<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.2.1.1/04 | Lee, D.Y. | 2001 | Hydrolysis of IN-N0079 in buffer solutions of pH 4, 7, and 9<br>DuPont Stine-Haskell Research Center<br>DuPont-4389, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.2.1.1/02 | Lee, D.Y., Berg, D.S. | 2001a | Hydrolysis of IN-A2213 in buffer solutions of pH 4, 7, and 9<br>DuPont Stine-Haskell Research Center<br>DuPont-4024<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.1/03 | Lee, D.Y., Berg, D.S. | 2001b | Hydrolysis of IN-D2708 in buffer solutions of pH 4, 7, and 9<br>DuPont Stine-Haskell Research Center<br>DuPont-4388<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.2.1/03 | LeNoir, J.S., Zietz, E. | 2003 | Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10 L by means of drip irrigation to cucurbits in a greenhouse in Spain, season 2000<br>DuPont Stine-Haskell Research Center, Newark, Delaware, USA<br>**DuPont Report No.:** DuPont-4719<br>GLP: Yes<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.3.1.1/02 | Malin, J.N. | 2015 | Adsorption/desorption of $^{14}$C-oxamyl in five soils<br>DuPont Experimental Station, DuPont Stine-Haskell Research Center<br>DuPont-3166, Revision No. 2<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.1.1/02 | Mattson, S.L., Smyser, B.P. | 2000 | Rate of degradation of oxamyl in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2957<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.1/01 | Mattson, S.L., Smyser, B.P. | 2000 | Rate of degradation of oxamyl in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2957<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.3/02 | Mellor, S.J. | 2000 | Route and rate of degradation of oxamyl metabolite IN-A2213 in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-3098<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.2.3/04 | Millais, A.J. | 2000 | Route and rate of degradation of oxamyl metabolite IN-N0079 in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-3070<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.2.1/02 | Mol, J.G.J. | 2002 | Field soil dissipation study of oxamyl in The Netherlands<br>DuPont Report No.: DuPont-2815<br>GLP: n/a<br>Published: No | N | N | | DuPont |
| B.8.1.2.2.1/06 | Mol, J.G.J. | 2003 | Freezer storage stability of oxamyl, IN-A2213 and IN-D2708 residues in soil samples from field soil dissipation studies conducted in Spain and Italy, season 2000<br>TNO Nutrition and Food Research<br>DuPont-9342<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP . | DuPont |
| B.8.1.3.1.1/01 | Ohm, M.B. | 2001 | Adsorption/desorption of $^{14}$C-oxamyl in five soils<br>DuPont Experimental Station, DuPont Stine-Haskell Research Center<br>DuPont-3166, Revision No. 1<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.2.1/07 | Partsch, S., Zillgens, B. | 2015 | Estimation of kinetic endpoints for oxamyl and its metabolites oxamyl oxime (IN-A2213) and DMOA (IN-D2708) from field soil dissipation studies<br>Dr. Knoell Consult GmbH<br>DuPont-41859 EU, Supplement No. 1<br>GLP: No<br>Published: No | N | N | | DuPont |
| B.8.2.3/03 | Shaw, D. | 2000 | Route and rate of degradation of oxamyl metabolite IN-D2708 in saturated subsoils<br>Huntingdon Life Sciences Ltd<br>DuPont-3097<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.1/01 | Smyser, B.P., Mattson, S.L. | 2000 | Route of degradation of oxamyl in aerobic soil<br>DuPont Experimental Station<br>DuPont-2958<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.1/02 | Smyser, B.P., Mattson, S.L. | 2000 | Route of degradation of oxamyl in aerobic soil<br>DuPont Experimental Station<br>DuPont-2958<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.1.1/03 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.2/01 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.1.2/02 | Spare, W.C. | 1992 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil, supplemental report<br>Agrisearch, Inc<br>AMR 1851-90, Supplement No. 1<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.1.2.1.1/03 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.1.2.1.3/01 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.4/01 | Spare, W.C. | 1991 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil<br>Agrisearch, Inc<br>AMR 1851-90<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.4/02 | Spare, W.C. | 1992 | Anaerobic soil metabolism of [1-$^{14}$C]Oxamyl in Madera, California soil, supplemental report<br>Agrisearch, Inc<br>AMR 1851-90, Supplement No. 1<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP and has not previously been protected or submitted | DuPont |
| B.8.2.2.3/01 | Spare, W.C. | 1995 | Degradability and fate of [1-$^{14}$C]oxamyl in water/sediment systems<br>Agrisearch, Inc<br>AMR 3143-94<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

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|---|---|---|---|---|---|---|---|
| B.8.3.1/01 | Styles, D. A. | 2000 | Photochemical oxidative degradation of oxamyl<br>DuPont Stine-Haskell Research Center<br>DuPont-4574<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.1.2.1.2/01 | Swain, R.S., Anderson, J. J. | 2000 | Rates of degradation of oxamyl metabolite [$^{14}$C](dimethylamino) oxoacetic acid (IN-D2708) in three aerobic soils<br>DuPont Experimental Station<br>DuPont-2675<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/02 | Tuffy, C. | 2000a | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-A2213<br>ABC Laboratories Europe Ltd<br>DuPont-4514<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/03 | Tuffy, C. | 2000b | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-D2708<br>ABC Laboratories Europe Ltd<br>DuPont-4515<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.2.1.2/04 | Tuffy, C. | 2000c | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-N0079<br>ABC Laboratories Europe Ltd<br>DuPont-4516<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.2/05 | Tuffy, C. | 2000d | Determination of the ultraviolet-visible absorption of oxamyl metabolite IN-T2921<br>ABC Laboratories Europe Ltd<br>DuPont-4517<br>Previously submitted at the EU level for Annex I inclusion<br>Published: No | N | N | | DuPont |
| B.8.2.1.1/05 | Van-Nguyen, A., Theilacker, W.M. | 2001 | Hydrolysis of IN-T2921 in buffer solutions of pH 4, 7, and 9<br>DuPont Report No.: DuPont-4390<br>Published: No | N | N | | DuPont |
| B.8.2.3/05 | Warren, R.L. | 2004 | Reductive degradation of oxamyl by ferrous iron<br>DuPont Stine-Haskell Research Center<br>DuPont-14826 EU<br>GLP: No<br>Published: No | N | N | | DuPont |

| Data requirement no., reference no. | Author(s) | Year | Title<br>Source<br>Company Report No.<br>GLP or GEP Status (where relevant)<br>Published or not | Vertebrate study Y/N | Data protection Y/N | Justification if data protection is claimed | Owner |
|---|---|---|---|---|---|---|---|
| B.8.1.2.2.1/01 | Zietz, E. | 2002 | Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10G to bare soil at sites in a typical potato growing region of England – Season 2000<br>DuPont Report No.: DuPont-3026<br>GLP: n/a<br>Published: No | N | N | | DuPont |
| B.8.1.2.2.1/04 | Zietz, E. | 2002 | Field soil dissipation of oxamyl nematicide and insecticide applied as Vydate 10 L by means of drip irrigation to cucurbits in a greenhouse in Italy - Season 2000<br>Institut Fresenius Chemische und Biologische/Gmbh<br>DuPont-4800<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP . | DuPont |
| B.8.1.2.2.1/05 | Zietz, E. | 2003 | Determination of the storage stability of oxamyl (DPX-D1410) and its metabolite oxamyl oxime (IN-A2213) in soil samples from a field soil dissipation study conducted in the United Kingdom, season 2000<br>Institut Fresenius Chemische und Biologische Laoratorien AG<br>DuPont-10099<br>GLP: Yes<br>Published: No | N | Y | The study is necessary for the regulatory decision, conducted according to GLP . | DuPont |