



128994

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MAY 24 1990

MEMORANDUM

SUBJECT: Dithiopyr - New Chemical Registration Standard
Environmental Fate and Groundwater Branch
Science Chapter

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Environmental Chemistry Review Section #2 *28 May 90*
Environmental Fate and Groundwater Branch/EFED (H7507C)

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Attached is the Environmental Fate and Groundwater Branch (EFGWB) Science Chapter and EFGWB Data Requirements for Dithiopyr New Chemical Registration Standard.

Dithiopyr is the common name of the active ingredient in the new herbicide Dimension. The chemical name for Dithiopyr is 2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-3,5-pyridinedicarboethoic acid, s,s-dimethyl ester. The Chemical Abstract Registry Number for this chemical is 97886-45-8.

Dithiopyr, a pyridine compound, is a herbicide proposed for use as a preemergent and postemergent to control annual grasses and broadleaf weeds in ornamental turf (terrestrial nonfood and domestic outdoor uses). Single active ingredient formulations include 1 and 3 lb/gal EC, 4 lb/gal Mcap, and 0.5% G. The proposed maximum application rate for dithiopyr is 1 lb ai/A.

Dithiopyr, codename Mon-7200, is referred to by Monsanto as a "rice/turf herbicide". In spite of this reference, Monsanto does not intend to register dithiopyr for use on rice or other food crops in the United States. In fact, rice grown in the United States is entirely direct-seeded (i.e. is not transplanted), and application of dithiopyr severely injures direct-seeded rice.

The current status of environmental fate data requirements to support the registration of Dithiopyr for use to control annual grasses and broadleaf weeds in ornamental turf (terrestrial nonfood and domestic outdoor uses) is as follows:

<u>Environmental Fate Data Requirements</u>	<u>Status of Data Requirement</u> /	<u>MRID No.</u>
Degradation Studies-Lab		
161-1 Hydrolysis	Fulfilled (AR;09/06/88)	40638627
161-2 Photodegradation in water	Fulfilled (AR;09/06/88)	40638628
161-3 Photodegradation on soil	Not Required (see 40638628)	
Metabolism Studies-Lab		
162-1 Aerobic (Soil)	Fulfilled (AR;09/06/88) (WGM;04/05/90)	40638629 41001517
Mobility Studies		
163-1 Leaching, Adsorption/ Desorption	Fulfilled (AR;09/06/88) (WGM;04/05/90)	40638630 41135601
Dissipation Studies-Field		
164-1 Terrestrial	Fulfilled (WGM;04/05/90)	41001519
Accumulation Studies		
165-4 In fish	Fulfilled (WGM;04/05/90)	41001518

ENVIRONMENTAL FATE OF DITHIOPYR

The rate of hydrolysis in water increases with increasing pH. The reported data gave no degradation of dithiopyr in sterile distilled water, in sterile rice paddy water, in sterile pH 5 buffer, and in sterile pH 7 buffer after 30 days of incubation in the dark at 25°C. Less than 2% of the applied dithiopyr degraded to the normal acid [(2-difluoromethyl)-4-(2-methylpropyl)-5-((methylthio)carbonyl)-6-(trifluoromethyl)-3-pyridinecarboxylic acid] in sterile pH 9 buffer after 30 days. Assuming that the rate of degradation of dithiopyr at pH 9 followed first order kinetics, the half-life was determined to be 1053 days. These results suggest that ester hydrolysis, which appears to be the degradation pathway for dithiopyr, will not be a significant route of degradation for dithiopyr in the environment.

When exposed to artificial light continuously at 25°C dithiopyr degraded with a half-life of 61.8 hours in a sterile, nonsensitized, pH 7 buffer aqueous solution and 72.4 hours in a similar solution amended with humin acid (as a photosensitizer). Half-life in sunlight days was determined to be 17.6 and 20.6 for the nonsensitized and humic acid solutions, respectively. The dark controls showed no degradation of dithiopyr. At completion of the photodegradation experiment with nonsensitized solutions, the parent material comprised 24.5% of the applied radioactivity. Two major degradates, normal acid and reverse acid {6-(difluoromethyl)-4-(2-methylpropyl)-5-[(methylthio)carbonyl]-2-(trifluoromethyl)-3-pyridinecarboxylic acid}, comprised 27.3 and 14.2%, respectively. The accountability of the applied material in the soil photodegradation experiment was 104.7% of which 101.9% was extractable. The parent compound accounted for 94% of the extractable material. A small amount, approximately 5%, of the degradate normal acid was the only other peak in the chromatogram after an exposure equivalent of 33.6 days, corresponding to a half-life of 444.3 days and a classification of photostable. These results indicate that photodegradation in water could contribute significantly to the overall degradation of dithiopyr in the environment. Dithiopyr in water can be expected to degrade to a mixture of normal acid, reverse acid, and lesser amounts of polar compounds upon exposure to sunlight. Photodegradation of dithiopyr on soils is insignificant. Therefore, photodegradation on soil will not contribute significantly to its degradation in the environment.

BIODEGRADATION OF DITHIOPYR

For dithiopyr, the only degradation process that appears to be occurring is ester hydrolysis to the corresponding diacid and two monoacids. Only small amounts, less than 6% of each, of the diacid and monoacid were observed after greater than one year of incubation. Many different bacterial isolates have been found to convert diesters to monoesters as in the case of alkyl phthalates and the conversion of esters to acid. It is postulated that the monoacids of dithiopyr are initially formed followed by degradation to the diacid.

The aerobic metabolism half-lives of dithiopyr in silt loam, in sandy loam, in clay, and in two Japanese loam soils were reported to be 523 days, 625 days, 639 days, and 1125 and 2300 days, respectively, when incubated in the dark at 25°C and 75% of 0.33 bar moisture for 12 - 18 months. At 12 - 18 months post-treatment, dithiopyr comprised 51.0-73.1 of the applied material in the five soils. The degradates (normal acid, reverse acid, and diacid) accounted for

6.3%, 5.0%, and 7.3% of the applied at maximum concentration, respectively. The highest concentrations of all three degradates were isolated from the silt loam soil. The maximum concentrations of the degradates in all five soils, in general, were reached at 6 to 12 months posttreatment. At termination of the study, unextractable radioactivity comprised 3.48 to 19.08% of the applied, $^{14}\text{CO}_2$ comprised 0.11 to 1.33%, and other volatiles, identified as dithiopyr in selected samples, comprised 7.3 to 25.9%. The volcanic soils from Japan which has the much higher organic content and much larger field moisture capacity than the other soils in the experiment seemed to have a lesser affinity for reaction with dithiopyr.

The results of the metabolism study demonstrated that the dissipation rate of dithiopyr in soil was slow, and that volatilization generally contributed more to the dissipation than degradation. Mineralization of dithiopyr to CO_2 is not extensive under aerobic dark conditions.

LEACHING POTENTIAL OF DITHIOPYR AND ITS DEGRADATES

Using batch equilibrium technique, dithiopyr was determined to be slightly mobile to relatively immobile in soil:0.01M calcium sulfate solution slurries. Freundlich K values were 6.59 in a sandy loam soil, 7.91 in a Dupon silt loam soil, 10.93 in a Drummer silt loam soil, 26.92 in a clay soil, 34.74 in a "volcanic ash up-land" loam soil, and 64.77 in a "volcanic ash paddy" loam soil. The K_d values were 99.6 for volcanic ash paddy, 52.3 for volcanic ash upland, 45.9 for Sharkey clay soil, 20.9 for Drummer silt loam, 12.8 for Dupon silt loam, and 7.9 for Sarpy loam soil. The K_{oc} ($100 \times K'/\text{organic C}$) values varied from 1141 to 3748. The percentage of desorption from the soils following adsorption ranged from a low 5.5% in the "volcanic ash paddy" loam soil to a high of 62% in the sandy loam soil. Based upon the batch equilibrium studies dithiopyr is adsorbed and slightly mobile to immobile in the soil environment. The highest Freundlich K values correlated with the soils having the highest Cation Exchange Capacity values and highest organic matter content (e.g. volcanic ash soils). ✓

Adsorption/desorption batch equilibrium studies with synthesized ^{14}C -labelled monoacid (normal and reverse acid) and diacid metabolites, in the four soil types used in the aerobic soil metabolism study were conducted to determine the mobility of these metabolites.

Based on batch equilibrium on the metabolites, the ^{14}C -labelled normal acid, reverse acid, and diacid were very mobile in sandy loam, clay, and two silt loam soils. However, the metabolites form slowly and at very low concentrations. Freundlich K_{ads} were 0.064 to 0.196 for the normal acid, 0.197 for the reverse acid, and 0 for the diacid. In the desorption phase of the study, 65 to 90% of the dithiopyr degradates adsorbed by soil were desorbed.

Dithiopyr dissipates in the field (all with vegetation coverage) with an average half-life of between 17 and 61 days depending on soil composition, weather conditions, and formulation applied. Three major metabolites of dithiopyr (the normal acid, reverse acid, and diacid metabolite) are formed and dissipate within 365 days. Vertical mobility of dithiopyr and its acid metabolites through the soil, even in conditions highly susceptible to herbicide leaching, was found not to exceed 24 inches, and was often not more than 9 to 12 inches. In no instance was dithiopyr or its major metabolites detected above the lower limit of 0.010 ✓

ppm between 24 and 96 inches, establishing a pesticide free zone of 6 feet. At the typical use rate, 0.5 lb/A, or the maximum labelled rate, 1.0 lb/A, of dithiopyr, metabolites would be observed at only barely or nondetectable levels. Therefore, dithiopyr residues when it is used at typical and maximum labelled application rates should not persist beyond the growing season and will not to any significant extent move vertically in the soil.

A comparison of dithiopyr's physical chemical properties to pesticide properties which have been found characteristic of some pesticides known to leach (Cohen et al) to ground water are given below:

	<u>Pesticides found in Ground-Water</u>	<u>Dithiopyr</u>
Aqueous solubility	> 30 mg/L	1.38 mg/L
K _d	< 5.0 usually < 1.0 or 2.0	45.9 to 7.9
K _{oc}	< 300 to 500	1141 to 3748
Speciation	negative charge	not soluble
Henry's constant	< 10 ⁻² atm-m ³ /mol	2 X 10 ⁻⁶ atm-m ³ /mol
Hydrolysis	> 25 weeks	1053 days at pH 9
Photolysis	> 1 week	61.8 hrs in H ₂ O stable on soil
Soil	> 2 to 3 weeks	17 to 61 days ?

Pesticides that are generally persistent and mobile are capable of leaching to ground water. The persistence characteristics are typical of pesticides found in ground water. However, the physical mobility characteristics are not characteristic of pesticides found in water. The physical nature of dithiopyr, low solubility in water and its high tendency to bind to soil, prevents it from being carried downward through the soil. Therefore, dithiopyr should have only a low potential for leaching to ground water contamination. The potential for surface water contamination by erosion of soil particles to which dithiopyr is adsorbed may be greater, since dithiopyr is persistent and binds to the upper layers of soil.

Monsanto contends that even though the adsorption of each of the acid metabolites is very low (K_{oc} range 0 to 26) which suggests that the acidic metabolites are highly mobile in soil the data is merely indicative rather than conclusive. The field soil dissipation studies demonstrate that even in the worst-case scenario with exaggerated application rates, and very sandy soil receiving 98 inches of irrigation water, dithiopyr and its metabolites do not move below the two foot depth. Moreover, deep core data collected from the worst-case location demonstrated a 6-foot residue free zone below the lowest penetration of the metabolites. Monsanto contends, also, sampling frequency and sampling depth was sufficient to monitor any possible movement of dithiopyr and its acidic metabolites, therefore, demonstrating conclusively that the proposed use of dithiopyr on turf poses no treat to groundwater.

BIOACCUMULATION OF DITHIOPYR

Exposure of bluegill sunfish to dithiopyr at an average of 0.006 ppm during a 35 day bioconcentration phase resulted in tissue total residue concentrations of 0.48 to 5.6 ppm in whole fish, 0.15 to 2.0 ppm in fillet, and 0.73 to 8.7 ppm in the viscera. The daily BCFs ranged from 81 to 930 in the whole fish, from 25 to 320 in the fillet, and from 120 to 1400 in the viscera. Three degradates identified in the fish tissues were the reverse acid with a maximum concentration of 0.013 ppm in whole fish, 0.014 ppm in edible tissues, and 0.29 ppm in non-edible tissues, the normal acid with a maximum concentrations of 0.053 ppm in whole fish and 0.15 ppm in nonedible tissues, and the diacid with a maximum concentration of 0.057 in whole fish and 0.31 ppm in nonedible tissues.

At the completion of a 14 day depuration phase, the dithiopyr concentration in whole fish was 0.29 ppm, in edible tissues was 0.15 ppm, in nonedible tissues was 0.62 ppm. The depuration rates represented were 94% in whole fish, 92% in fillet, and 93% in viscera.

RECOMMENDATIONS

The environmental fate data are adequate to support the proposed registration of Dithiopyr for terrestrial nonfood and domestic outdoor uses.

The environmental fate and transport studies submitted in support of registering dithiopyr demonstrate that the chemical is persistent and has the potential to run off into surface water. In lieu of actual field run off data, modelling is used to estimate the amount of dithiopyr in surface water over time. Important factors in the model are the dissipation rates in various aquatic systems. Rather than using the field dissipation rate of aerobic metabolism rate, it would be more appropriate to use a nonsterile pond water dissipation rate and an anaerobic aquatic metabolism data which give a more accurately reflection of the various aquatic degradation pathways.

Also, based on the submitted data, there is a large difference between the half-life calculated from the aerobic metabolism study and the field dissipation study (1.63 - 6.3 years and 17 - 61 days, respectively). The reason for these differences is not apparent and the registrant needs to address these different dissipation rates. As required in the present guidance document two bare ground field dissipation studies carried out at representative sites must be submitted. These studies should be initiated by the applicant and submitted to the Agency within the appropriate time frame.

The following conditional data are required:

1. Pond water (nonsterile) dissipation rate study.
2. Anaerobic aquatic metabolism study.
3. Bare ground field dissipation studies.

Also, since the pond water dissipation rate study is not a routinely required study, the applicant should discuss their protocol with EFGWB prior to undertaking the study. These data can be developed as a condition of registration.

Prior to registering any additional new uses, the company must address the deficiencies in the submitted photodegradation in soil study. These deficiencies should be addressed due to the severity of the light intensity and lack of data comparing natural and artificial sunlight. If the deficiencies cannot be corrected, the company must submit a new photodegradation in soil study.

TABLE A. GENERIC DATA REQUIREMENTS FOR DITHIOPYR

Data Requirement	Test Substance ¹	Use Pattern ²	Does EPA have data to satisfy the requirement?	Biblio. Citation	Must additional data be submitted?
<u>\$158.290 Environmental Fate</u>					
<u>DEGRADATION STUDIES-LAB:</u>					
161-1 - Hydrolysis	TGAI/PAIRA	B/H	Yes	40638627	No
<u>Photodegradation:</u>					
161-2 - In Water	TGAI/PAIRA	B	Yes	40638628	No
161-3 - On Soil			No		Yes ³
161-4 - In Air			No		No ⁴
<u>METABOLISM STUDIES-LAB:</u>					
162-1 - Aerobic Soil	TGAI/PAIRA	B/H	Yes	40638629 41001517	No ⁵
162-2 - Anaerobic Soil			No		No ⁶
162-3 - Anaerobic Aquatic			No		Yes ¹⁷
162-4 - Aerobic Aquatic			No		No ⁶
<u>MOBILITY STUDIES:</u>					
163-1 - Leaching and Adsorption/Desorp.	TGAI/PAIRA	B/H	Yes	40638630 41135601	No ⁷
163-2 - Volatility (Lab)			No		No ⁴
163-3 - Volatility (Field)			No		No ⁴

TABLE A. GENERIC DATA REQUIREMENTS FOR DITHIOPYR (continued)

Data Requirement	Test Substance ¹	Use Pattern ²	Does EPA have data to satisfy the requirement?	Biblio. Citation	Must additional data be submitted?
<u>\$158.290 Environmental Fate (cont.)</u>					
<u>DISSIPATION STUDIES-FIELD:</u>					
164-1 - Terrestrial (Soil)	TEP	B	Yes	41001519	No ¹⁸
164-2 - Aquatic (Sediment)			No		No ⁸
164-3 - Forestry			No		No ⁹
164-4 - Combination/Tank Mix			No		No ¹⁰
164-5 - Long Term Terrestrial	TEP		No		No ¹¹
<u>ACCUMULATION STUDIES:</u>					
165-1 - Confined Rotat. Crops			No		No ¹²
165-2 - Field Rotat. Crops			No		No ¹³
165-3 - Irrigated Crops			No		No ¹⁴
165-4 - Fish	TGAI/PAIRA	B	No	41001518	No
165-5 - Aquatic Non-target Organisms			No		No ¹⁵

TABLE A. GENERIC DATA REQUIREMENTS FOR DITHIOPYR (continued)

Data Requirement	Test Substance ¹	Use Pattern ²	Does EPA have data to satisfy the requirement?	Biblio. Citation	Must additional data be submitted?
<u>\$158.440 Spray Drift Data Requirements</u>					
201-1 - Droplet size spectrum			No		Reserved ¹⁶
202-1 - Drift field evaluation			No		Reserved ¹⁶

TABLE A. GENERIC DATA REQUIREMENTS FOR DITHIOPYR (continued)

FOOTNOTES:

- (1) Test substance abbreviations: TGAI = technical grade active ingredient; PAIRA = radiolabeled pure active ingredient; TEP = typical end product.
- (2) Use pattern codes: A = terrestrial food crops; B = terrestrial non-food; C = aquatic food crop; D = aquatic non-food; E = greenhouse food crops; F = greenhouse non-food; G = forestry; H = domestic outdoor; I = indoor; NA = not applicable.
- (3) One photodegradation study on soil (40638628) was submitted. The study was not acceptable to fulfill the data requirement. The deficiencies addressed must be satisfied before registration of any new uses which require the photodegradation on soil study.
- (4) No laboratory volatility, field volatility, on photodegradation in air data were submitted. However, those data requirements are not required for terrestrial non-food and domestic uses. As well, the vapor pressure of dithiopyr is 4.0×10^{-6} mm/Hg which is at the 10^{-6} mm/Hg trigger point of requiring a laboratory volatility study.
- (5) The aerobic soil metabolism study (40638629) was submitted with missing sample data. This study was acceptable for fulfillment of the EUP data requirement. The missing sample data was submitted (41001517) to fulfill the data requirement for full registration and was acceptable to fulfill the data requirement.
- (6) No anaerobic soil, anaerobic aquatic, and aerobic aquatic metabolism data were submitted, but none are required to support the registration of terrestrial non-food or domestic uses.
- (7) Two leaching adsorption/desorption studies were submitted. Study (40638630) was on unaged parent material and was acceptable. After 30 days of aging, the metabolites were not at a concentration sufficient to analysis for leaching. Monsanto met with the EFGWB and determined that to satisfy the data requirement synthesized 14 C-labelled metabolite standards would be used to determine the leaching ability of the metabolites. Study (41135601) was submitted and acceptable to meet the data requirement for aged material. Therefore, the leaching, adsorption/desorption data requirement for both unages and aged has been fulfilled.

TABLE A. GENERIC DATA REQUIREMENTS FOR DITHIOPYR (continued)

- (8) No aquatic (sediment) field dissipation data were submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (9) No forestry field dissipation data were submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (10) Combination/tank mix data are no longer required.
- (11) No long term terrestrial field dissipation data were submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (12) No confined accumulation in rotational crops data have been submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (13) No field accumulation in rotational crops data have been submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (14) No accumulation in irrigated crops data were submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (15) No accumulation in aquatic non-target organism data were submitted, but none are required to support the registration of terrestrial non-food and domestic uses.
- (16) No droplet size spectrum or drift field evaluation are required at this time but this data may be required in the future.
- (17) Data from anaerobic aquatic metabolism studies is currently being used to support in house modelling efforts to predict movement/fate of selected pesticides in aquatic compartments of the environment. Since no such data is currently available for dithiopyr, the anaerobic aquatic metabolism study is being required postregistration.
- (18) Normally two bare soil terrestrial field dissipation studies are required. Since there was no urgency seen for this data, this data is being required postregistration.

MON-15151

DITHIOPYR

Task 1: Review and Evaluation
of Individual Studies

Task 2: Environmental Fate
Assessment

August 30, 1988

See Feb. 9, 1990

Final Report

Contract No. 68-02-4250

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
11140 Rockville Pike
Rockville, MD 20852

DITHIOPYR

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DATA EVALUATION RECORD

STUDY 1

SHAUGHNESSY NO. 128994

MON-15151

Sec. 16 -1

FORMULATION--00--ACTIVE INGREDIENT

MRID NO. 40638627

Pantano, L.K. 1988. The hydrolysis of MON-7200/15100, 3,5-pyridine-dicarbo-thioic acid, 2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-,S,S-dimethyl ester. Laboratory Project No. MSL-7690. R.D. No. 866. Unpublished study prepared and submitted by Monsanto Agricultural Company, St. Louis, MO.

DIRECT REVIEW TIME = 24

REVIEWED BY: W. Hurtt TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

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8-30-88

SIGNATURE:

CONCLUSIONS:

Degradation - Hydrolysis

This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the hydrolysis of ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-7200/15100 (the active ingredient of MON-15151) in sterile aqueous buffered solutions at pH 5, 7, and 9.

SUMMARY OF DATA BY REVIEWER:

Ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON 7200/15100 (radiochemical purity of the 1:1 isotopic mixture >99.0%), at 1 ppm, did not degrade in sterile deionized water or buffered pH 5 and 7 solutions at 25°C over a 30-day period. In a buffered pH 9 solution after 30 days, <2% of the test substance had degraded to 2-(difluoromethyl)-4-(2-methylpropyl)-5-[(methylthio)carbonyl]-6-(trifluoromethyl)-3-pyridinecarboxylic acid (normal acid; II).

The registrant-calculated half-life in the pH 9 buffer solution was 1053 days (2.9 years).

DISCUSSION:

1. HPLC procedures were modified during the course of the experiments to include an acetonitrile wash when it was determined that MON 7200/15100 was adhering to the walls of the HPLC syringe. The registrant stated that the correction for syringe adhesion had only a slight effect on the results of the experiments.
2. Apparent typographical errors exist with regard to sample (tube) number versus days posttreatment. Sample D25-48 (Table 9) was used for critical confirmation analyses, presumably at 25 days. In the original document, this sample was variously referenced as both 25 and 30 days in Figure 10, and as 30 days in Figures 11, 12, and 13 and in various portions of the text.
3. A hydrolysis study was conducted with MON 7200/15100 using sterile rice paddy water (pH 7.8); no degradation was observed during 30 days of incubation. The data were not evaluated in detail because they are not required by current EPA guidelines for registering pesticides.

MATERIALS AND METHODS

-1.3-

MATERIALS AND METHODS:

Ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON 7200/15100 [3,5-pyridinedicarbothioic acid, 2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-,S,S-dimethyl ester; radiochemical purity of the 1:1 isotopic mixture >99.0%, specific activity 18.58 mCi/mmol, Monsanto Company] was added to sterile deionized water and to sterile buffered water adjusted to pH 5, 7, and 9 to make a final concentration of 1 ppm. The solutions were incubated in the dark at 25°C in culture tubes, and samples were taken at 0, 2, 4, 9, 15, 21, 25, and 30 days after treatment.

Total radioactivity at each sampling interval was determined by ISC. Quantification of the effluent from HPLC analyses was determined by ISC or radioactivity flow detection. The identification of degradate structures was based upon comparisons between retention times of the radioactive peaks of the test samples and the UV peaks of the coinjected non-radiolabeled standards. Confirmation of degradate structure was determined by HPLC/MS analysis of the 25-day samples.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

1.5

Dithiopyr Science Review

Page _____ is not included in this copy.

Pages 20 through 26 are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product quality control procedures.

_____ Identity of the source of product ingredients.

_____ Sales or other commercial/financial information.

_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action

X FIFRA registration data.

_____ The document is a duplicate of page(s) _____

_____ The document is not responsive to the request.

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

DATA EVALUATION RECORD

STUDY 2

SHAUGHNESSY N 128994 MON-15151 Sec. 161-2 and 161-3

FORMULATION—00—ACTIVE INGREDIENT

MRID NO. 40638028

Klemm, G.H., R.G. Nadeau, and R.T. Solsten. 1988. The photolysis of MON-7200 [3,5-pyridine-dicarbothioic acid, 2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-,S,S-dimethyl ester] in water and in soil. Laboratory Project Identification MSL-7049, R. D. No. 866. Unpublished study prepared and submitted by Monsanto Agricultural Company, St. Louis, MO.

DIRECT REVIEW TIME = 24

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September 1, 1988

CONCLUSIONS:

Degradation - Photodegradation in Water

This part of the report is acceptable and fulfills EPA Data Requirements for an aqueous photolysis study by providing information on the photodegradation of ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-15151 in sterile pH 7 buffered nonsensitized and sensitized aqueous solutions.

Degradation - Photodegradation on Soil

For the purposes of an EUP this part of the report satisfies the requirement for a photolysis study on soil by providing information on the photodegradation of ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-15151 on a silt loam soil.

SUMMARY OF DATA BY REVIEWER:

Degradation - Photodegradation in Water

Ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-15151 (radiochemical purity >99%), at 0.7 ppm, degraded with a half-life of 61.8 hours in a sterile, nonsensitized pH 7 buffered aqueous solution and 72.4 hours in a similar solution amended with humic acid (as a photosensitizer) during continuous irradiation under artificial light at 25°C. The intensity of the light source (one 500-watt xenon arc lamp) was 198 mW/cm² compared to a registrant-computed sunlight intensity of 58 mW/cm². Half-lives in sunlight days, as computed by the registrant using 58 mW/cm² as a reference, were 17.6 and 20.6 for the nonsensitized and humic acid solutions, respectively. At the termination of the photolysis experiment (≈128 hours artificial light or 36.4 sunlight days) with nonsensitized solution, MON-15151 comprised 24.5% of the applied radioactivity and two degradate monoacids, . . .

2-(difluoromethyl)-4-(2-methylpropyl)-5-[(methylthio)carbonyl]-6-(trifluoromethyl)-3-pyridinecarboxylic acid (normal acid; II) and

6-(difluoromethyl)-4-(2-methylpropyl)-5-[(methylthio)carbonyl]-2-(trifluoromethyl)-3-pyridinecarboxylic acid (reverse acid; III) . . .

comprised 27.3 and 14.2%, respectively, of the applied radioactivity. Two minor degradates, each of which comprised ≈4-5% of the applied radioactivity, were . . .

2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-3,5-pyridinedicarboxylic acid (diacid; IV) and

3,5-bis(methylthiocarbonyl)-2-formyl-4-(2-methylpropyl)-6-trifluoromethyl pyridine (V).

Two additional minor degradates together comprised 7.3% of the applied radioactivity . . .

3-(methylthiocarbonyl)-2-difluoromethyl-4-(2-methylpropyl)-6-trifluoromethylpyridine (VI) and

3-(methylthiocarbonyl)-2-formyl-4-(2-methylpropyl)-6-trifluoromethylpyridine (VII).

Similar degradates and percentage recoveries were obtained with the humic acid-sensitized solutions. MON-15151 was stable in all control solutions maintained in the dark.

Degradation - Photodegradation on Soil

Ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-15151 (radiochemical purity >99%), at 1 lb/A on silt loam soil, was slightly degraded to 2-(difluoromethyl)-4-(2-methyl-

propyl)-5-[(methylthio)carbonyl]-6-(trifluoromethyl)3-pyridinecarboxylic acid (normal acid; II), which comprised ~5% of the applied radioactivity after continuous exposure to 118 hours of artificial light (one 500-watt xenon arc lamp). MON-15151 was stable in the dark control soil.

DISCUSSION:

Degradation - Photodegradation in Water

1. The absorption spectra of MON-15151 in buffer or buffer plus humic acids was not provided.
2. Emission spectra for the xenon arc lamp with two types of filtering devices were provided as was a spectrum for sunlight. However, it was not possible to make a realistic comparison of the wavelength-energy distribution between the artificial light sources and sunlight because of large differences in scale values on the figures depicting the spectra.
3. Intensity measurements were provided for the light system used, but it could not be determined what the relationship was between the values provided and the actual intensity within the photolysis chamber.
4. Although the temperature of the solutions within the photolysis chamber was apparently predetermined to be 25°C with a coolant temperature of 19.8°C (the protocol specified 5°C) and the temperature of the solutions was measured prior to analyses, sample data were not provided to substantiate that the photolysis experiments were actually conducted at 25°C.
5. Filter-sterilized deionized water was used in these experiments, but it could not be determined whether the humic acid solutions were sterilized after addition of the humic acids.
6. A study was also conducted using filter-sterilized rice paddy water (pH 8.0); the half-life calculations for MON-15151 differed by less than 1 day between pH 7 buffered water and rice paddy water. The data were not evaluated in detail because they are not required by current EPA Guidelines for Registering Pesticides.

Degradation - Photodegradation in Soil

1. The photolytic degradation of MON-15151 on soil was stated as having been conducted at 25°C. However, this value appears to have been based upon a personal communication that a coolant temperature of 10°C would maintain the soil surface at 25°C rather than upon actual soil temperature measurements.
2. The registrant is advised that continuous (24 hrs/day) ^{photolytic Exposure} is not an acceptable procedure. Alternating light/dark periods of 12 hours are recommended to allow bacterial recovery. The intensity of the artificial light must be comparable to natural sunlight, not many times greater.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Degradation - Photodegradation in Water

Ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-15151 (radiochemical purity of the mixture was >99% and specific activity was 17.87 mCi/mmol, Monsanto Company) was added at 0.7 ppm to sterile pH 7 phosphate buffer and buffer containing 25.8 mg/L humic acids as photosensitizers. The solutions were irradiated in a cylindrical Pyrex chamber at 25°C under continuous light from a xenon arc lamp with a light intensity of 198 mW/cm² between 300 and 750 nm. Samples were removed at intervals from 0.5 to ca. 128 hours for degradate analysis, pH, and temperature measurements. A foam plug used to trap volatiles was removed from the photolysis chamber for analysis at each sampling period. A control solution was kept in the dark at 25°C and sampled at an unspecified time period for degradate formation.

Total radioactivity of the test solution and foam plug was determined at each sampling period by ISC. Solutions were then extracted with ether, acidified, extracted with ethyl acetate, and concentrated. The fractions from the extraction of the nonphotosensitized solutions were analyzed by HPLC with a radioactive flow detector, identification of degradates was based upon comparison of retention times with standards, and structure confirmation was determined by MS. Degradates in extracts of the sensitized solutions were identified by comparison of their retention times with those of the identified degradates from the nonsensitized solutions.

Degradation - Photodegradation on Soil

A slurry of sieved (2 mm) silt loam soil (24% sand, 68% silt, 8% clay, pH 7.5, and CEC 10.7 meq/100 g) was added to petri dishes to a depth of 1 mm and air-dried overnight. Ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-15151 (properties as previously described for photodegradation in water) in hexane was added to the soil at 1 lb/A, allowed to air dry for evaporation of the hexane, and continuously exposed to the xenon arc lamp source (radiant properties as previously described) in an enclosed cylindrical Pyrex chamber at ca. 25°C for intervals varying from 3.6 to ca. 118 hours. Comparable control dishes of soil were sealed and maintained in the dark for 2.4 and ca. 118 hours prior to sampling. The Pyrex chamber was vented through two ports fitted with foam plugs to trap volatiles.

At each sampling period, the chamber was flushed with air and the foam plugs and one dish of soil were removed for extraction and analysis. The plugs were extracted with ether and the soil was extracted with water:acetonitrile (50:50). Radioactivity was determined by both combustion and ISC; analysis and confirmation was based upon HPLC with radioactive flow monitoring and comparisons to known standards.

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Pages 32 through 47 are not included in this copy.

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DATA EVALUATION RECORD

STUDY 3

SHAUGHNESSY NO. 128994

MON-15151

Sec. 162-1

FORMULATION—00—ACTIVE INGREDIENT

MRID NO. 40638629

Pantano, L.K. and Y.L. Sing. 1988. The aerobic soil metabolism of MON-7200/15100, 3,5-pyridine-dicarbothioic acid, 2-(difluoromethyl)-4-(2-methyl-propyl)-6-(trifluoromethyl)-,S,S-dimethyl ester. Laboratory Project No. MSL-7781. R.D. No. 866. Prepared and submitted by Monsanto Agricultural Company, St. Louis, MO.

DIRECT REVIEW TIME = 12

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: J. Harlin TITLE: Staff Scientist

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Allen J. Reiter
8-30-88

SIGNATURE:

CONCLUSIONS:

Metabolism - Aerobic Soil

This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the metabolism of ring-labeled [$^{13}\text{C}/^{14}\text{C}$]MON-7200/15100 in aerobic sandy loam, silt loam, loam, and clay soils.

SUMMARY OF DATA BY REVIEWER:

[$^{13}\text{C}/^{14}\text{C}$]MON-7200/15100 (labeled in the 4 position of the pyridine ring; radiochemical purity >99.0%), at ~1 ppm (50 ug/50 g dry soil), degraded with registrant-calculated half-lives of 336 days in Dupo silt loam soil, 418 days in Sarpy sandy loam soil, 490 days in Sharkey clay soil, 904 days in "volcanic ash upland" loam soil, and 946 days in "volcanic ash paddy" loam soil. The treated soils were incubated in the dark at 25°C

and 75% of 0.33 atmosphere moisture for 6 months (sandy loam, clay, and loam soils) or 12 months (silt loam soil only) posttreatment. Nonvolatile degradates, each comprising $\leq 3.3\%$ of the applied in the five soils at intervals up to 6 months posttreatment, were . . .

2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-3,5-pyridinedicarboxylic acid (diacid; IV);

6-(difluoromethyl)-4-(2-methylpropyl)-5-[(methylthio)carbonyl]-2-(trifluoromethyl)-3-pyridinecarboxylic acid (reverse acid; III); and

2-(difluoromethyl)-4-(2-methylpropyl)-5-[(methylthio)carbonyl]-6-(trifluoromethyl)-3-pyridinecarboxylic acid (normal acid; II).

After 6 months of incubation, between 4.9 and 16.0% of the applied had been volatilized as undegraded MON-15151 and 0.06-0.71% had been volatilized as $^{14}\text{CO}_2$; unextractable [^{14}C]residues ranged from 3.99 to 11.10% of the applied. In the Dupo silt loam soil, the maximum concentration of the nonvolatile degradates occurred at 9 months posttreatment; the diacid accumulated to a maximum concentration of 6.9% of the applied, the reverse acid accumulated to 3.0% of the applied, and the normal acid to 5.9% of the applied. In the Dupo silt loam soil at 12 months posttreatment, $\approx 19.6\%$ had been volatilized as MON-15151, 0.43% was $^{14}\text{CO}_2$, and 7.08% was unextractable. The material balances in all soils throughout the study ranged from 92.49 to 102.06% of the applied.

DISCUSSION:

1. Although it was stated that additional flasks of soil were autoclaved and treated with [$^{13}\text{C}/^{14}\text{C}$]MON-7200/15100 to serve as sterile controls, no data were provided.
2. The volcanic ash soils are not typical of soils in the United States. They were included in this study because MON-15151 is to be registered in Japan.

MATERIALS AND METHODS

MATERIALS AND METHODS:

[¹³C/¹⁴C]MON-7200/15100 (labeled in the 4 position of the pyridine ring; radiochemical purity >99.0%, specific activity 18.32-26.21 mCi/mmol, Monsanto Chemical Company) was applied at ≈1 ppm (50 µg/50 g dry soil) to flasks containing sieved (2 mm) moist (75% of 0.33 atmosphere) Sarpy sandy loam, Dupo silt loam, Sharkey clay, volcanic ash paddy loam, or volcanic ash upland loam soil (Table 5). After treatment, the soil was "lightly shaken" and the flasks were sealed with an aerobic trapping tower that was constructed of two foam plugs (in the neck of the flask), a layer of Drierite, a foam plug, Ascarite II (NaOH fused to a silicate carrier), a foam plug, Ascarite II, a foam plug, Drierite, and a foam plug (at the top of the column). All flasks of soil were incubated in the dark at 25°C; the soils were remoistened once or twice each week. The trapping towers were replaced weekly. Duplicate flasks were removed for analysis at intervals up to 6 months (Sarpy sandy loam, Sharkey clay, volcanic ash paddy loam, and volcanic ash upland loam soils) or 12 months (Dupo silt loam soil) posttreatment.

Soil samples were transferred to centrifuge tubes. The sample flasks were rinsed with acetonitrile:water (50:50), and the rinses were transferred to the appropriate centrifuge tubes. The soils were extracted by shaking for 20 minutes on a wrist action shaker, then centrifuged. The acetonitrile:water extracts were decanted and fresh acetonitrile:water was added to the soil. The soils were extracted with acetonitrile:water a total of five times; the extracts were combined and an aliquot was analyzed by LSC. Additional aliquots were analyzed by HPLC with radioactivity detection, and [¹⁴C]compounds were tentatively identified by comparison to reference standards of MON-7200/15100, Normal acid, Reverse acid, and diacid. The extracted soils were analyzed for unextractable radioactivity by LSC following combustion.

In order to confirm the identity of [¹⁴C]compounds, the acetonitrile:water extract of the 6-month Dupo silt loam soil sample was made basic with sodium bicarbonate and partitioned three times with hexane. The aqueous fraction was acidified and partitioned three times with ether. The hexane, ether, and aqueous fractions were analyzed by LSC; the hexane and aqueous fractions were analyzed by HPLC with radioactivity detection. [¹⁴C]Compounds isolated by HPLC were identified by HPLC/MS (negative ion mode).

The foam plugs from the trapping towers were analyzed by LSC. Foam plugs from the Dupo silt loam (week 28) and volcanic ash upland loam (week 22) soil were extracted with ether; aliquots of the ether extracts and the extracted foam plugs were analyzed by LSC. Additional aliquots of the ether extracts were concentrated, dissolved in acetonitrile:water (50:50), and analyzed by HPLC and LSC. The Ascarite samples were mixed with distilled water, then sulfuric acid was added dropwise to the solution. Escaping CO₂ was trapped in a phenethylamine solution; the phenethylamine solution was analyzed by LSC.

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DATA EVALUATION RECORD

STUDY 4

SHAUGHNESSY NO. 128994

MON-15151

Sec. 163-1

MRID NO. 40638630

Sing, Y.L. 1988. Adsorption/desorption of MON-7200/15100. Laboratory Project Identification MSL-7778; R.D. No. 866. Unpublished study prepared and submitted by Monsanto Agricultural Company, St. Louis, MO.

DIRECT REVIEW TIME = 24

REVIEWED BY: W. Hurtt

TITLE: Staff Scientist

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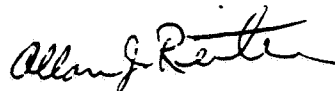
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TEL: 557-7709



SIGNATURE:

September 1, 1988

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

For the purposes of an EUP this study satisfies the requirement for leaching by providing information on the mobility (batch equilibrium) of unaged MON-7200/15100 in six soils ranging from a sandy loam to a clay. An additional study is needed to establish the mobility of aged (30 days) MON-7200/15100 residues. For full registration, a mobility study (batch equilibrium) of the three major degradates (two monoacids and one diacid) must be conducted.

SUMMARY OF DATA BY REVIEWER:

Using batch equilibrium techniques, ring-labeled [^{14}C]MON-7200/15100 (radiochemical purity >99%), at 0.01-0.3 ug/mL, was determined to be slightly mobile to mobile in soil:0.01 M CaSO_4 solution slurries. Freundlich K values were 6.59 in a Sarpy sandy loam soil, 7.91 in a Dupont silt loam soil, 10.93 in a Drummer silt loam soil, 26.92 in a Sharkey clay soil, 34.74 in a "volcanic ash upland" loam soil, and 64.77 in a "volcanic ash paddy" loam soil; 1/n values were 0.80-0.93. The three

highest Freundlich K values ($\approx 27-65$) correlated with the soils having the highest CEC values ($\approx 27-40$) and organic matter content ($\approx 2-5\%$). Similarly, the sandy loam soil which had the lowest K value (6.59) had the lowest CEC value and percentage organic matter content (≈ 10 and <1 , respectively). The K_{oc} values varied from 1141 to 3748. The percentage of MON-7200/15100 desorbed from the soils following adsorption ranged from a low of 5.5% in the "volcanic ash paddy" loam soil to a high of $\approx 62\%$ in the Sarpy sandy loam soil.

DISCUSSION:

1. The reviewer has classified the soils as slightly mobile to mobile based on the definitions of mobility reported in the Federal Register 44(53), dated March 16, 1979. Soils with K values of ≈ 10 are considered mobile and those with K values of ≈ 100 are slightly mobile. The reviewer is uncertain how the registrant determined that the K values obtained in this study determined MON-7200/15100 to be immobile; if the determination was based on the K_{oc} values rather than the K values, it should be noted that mobility scales were not designed for use with K_{oc} values.
2. The mobility of aged [^{14}C]MON-7200/15100 was not addressed. Based on 30-day posttreatment data from the aerobic soil metabolism study (Study 3), aged residues would be $>86.4\%$ parent MON-7200/15100 and three degradates (the normal acid, reverse acid, and diacid) would each comprise $<2\%$ of the applied.
3. The registrant calculated K_{oc} using the equation: $100 \times K'/\text{organic C}$; however, the equation is reported as $K'/\text{organic C}$ in the definitions listed under each table. $K'/\text{organic C}$ is the equation encountered most frequently.
4. It should be noted that an apparent typographical error exists in Table 3 (properties of the soils used) where the sand content of the Drummer silt loam is listed as 1.11%. If this value is used, the sum of sand, silt, and clay would be only $\approx 77.8\%$.

MATERIALS AND METHODS

-4.3-

MATERIALS AND METHODS:

In a batch equilibrium study, six soils (two loams, a clay loam, two silty loams, and a sandy loam; described in Table 3) were air-dried for 1-2 days and sieved through a 2-mm sieve. A preliminary equilibrium test was conducted on each of the soils to determine a valid shaking time for conducting the adsorption experiment. Sixteen hours was selected for all subsequent studies based upon the equilibria obtained (Figure 4). The adsorption studies were conducted with duplicate samples of each soil which were shaken for 16 hours at 25-26°C with 0.01 M CaSO_4 solutions containing ring-labeled [^{14}C]MON-7200/-15100 (radiochemical purity was >99%, specific activity was 26.21 mCi/mmol, Monsanto Agricultural Company) at 0.01, 0.05, 0.1, and 0.3 ppm. After the shaking period, the containers of soil and solution were centrifuged, and aliquots of the supernatant were analyzed for total radioactivity by LSC. An acetonitrile wash was used to minimize loss due to adsorption of the pesticide on the container walls. Identification was by HPLC/LSC and structure confirmation was by GC/MS. A chemical stability test to confirm that MON-7200/15100 was not degraded on the soil or during the analytical steps indicated that degradation was negligible.

Desorption of [^{14}C]MON-7200/15100 was determined by adding back to the centrifuged soils (used in the adsorption experiments) a volume of 0.01 M CaSO_4 equal to that previously decanted, followed by 16 hours of shaking, and a repeat of the decanting and aliquot-counting steps. These desorption steps were repeated three times, the remaining [^{14}C]MON-7200/15100 on the soil was determined by combustion, and all recoveries were totaled to determine the mass balance.

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