816380
Record No.

Review No.

111601
Shaughnessey No.

EEB REVIEW

72-7 a/b

DATE: IN 12/03/91 OUT 3/03/92

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PETITION OR EXP. NO.

DATE OF SUBMISSION 12/04/91

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RD ACTION CODE/TYPE OF REVIEW 620 3(C)2B 90 Day Response

TYPE PRODUCTS(S): I, D, H, F, N, R, S Herbicide

MRID NO(S). 420480-01; 420480-02; 420480-03

PRODUCT MANAGER NO. B. Sidwell (53)

PRODUCT NAME(S) Goal 1.6E (Oxyfluoren)

COMPANY NAME Rohm and Haas Company

SUBMISSION PURPOSE 3 (C) 2 B 90 Day Response/Misc. data

SHAUGHNESSEY NO.	CHEMICAL AND FORMULATION	% A.I.
111601	Oxyfluoren	19.5%

(1)

3/9/1992

MRID No. 420480-01

DATA EVALUATION RECORD

1. **CHEMICAL:** Oxyflurofen.
Shaughnessey No. 111601.

2. **TEST MATERIAL:** 1) Goal 1.6E; Lot No. 2102225; 19.5% active ingredient; a black liquid.
2) ^{14}C-Goal 1.6E; Number 568.0103; 0.504 mCi; 99% radiopurity; a yellow powder.

3. **STUDY TYPE:** Freshwater Invertebrate Static Acute Toxicity Test. Species Tested: Midge (*Chironomus tentans*).

4. **CITATION:** Swigert, J.P. 1989. Acute Toxicity of Soil-Incorporated ^{14}C-Goal® 1.6E to Midge Larvae, (*Chironomus tentans*). Final Report No. 37582. Rohm and Haas Report No. 88RC-0080. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO. Submitted by Rohm and Haas Company, Spring House, PA. EPA MRID No. 420480-01.

5. **REVIEWED BY:**

Louis M. Rifici, M.S.
Associate Scientist
KBN Engineering and
Applied Sciences, Inc.

Signature: Louis M Rifici
Date: 2/25/92

6. **APPROVED BY:**

Pim Kosalwat, Ph.D.
Senior Scientist
KBN Engineering and
Applied Sciences, Inc.

Signature: P. Kosalwat TB 3/9/92
Date: 2/25/92

Henry T. Craven, M.S.
Supervisor, EEB/EFED
USEPA

Signature: Henry T. Craven
Date: 3/4/92

7. **CONCLUSIONS:** The study using HOM soil is not scientifically sound. Mortality in control replicate A of the HOM soil test was 47%. The study using LOM soil is scientifically sound but does not meet the guideline requirements for a sediment toxicity test using midge larvae. The concentrations tested were not high enough to produce an EC_{50} value but were less than 100 ppm. The 96-hour EC_{50} was >7.8 ppm (mg/kg), the highest concentration tested. A no-observed-effect concentration was not generated in the test.

8. **RECOMMENDATIONS:** N/A.

02

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9. **BACKGROUND:**

10. **DISCUSSION OF INDIVIDUAL TESTS:** N/A.

11. **MATERIALS AND METHODS:**

A. **Test Animals:** Midge larvae (*Chironomus tentans*) were obtained from in-house cultures. The cultures were fed a suspension of Tetramin® and cereal leaves, *ad libitum*. Two egg masses were collected and placed in glass dishes (18 x 30 cm). The larvae were maintained in the dishes until they developed to the desired stage. Third-instar midge larvae (17 days post-hatch) were used in the test.

B. **Test System:** Vessels used in the test were 1-l glass jars having a cross-sectional area of approximately 57 cm^2. Each jar contained 100 g of dosed sediment (sediment depth 2.5 cm) and 500 ml of clean water. The jars were placed in a temperature-controlled water bath (20 ±2°C).

The working stock for the test was prepared by mixing 0.1988 g of Goal 1.6E formulation with 0.75 ml of ^{14}C-Goal 1.6E study stock solution (prepared in acetone) and diluting to 25 ml with acetone. The concentration of the working stock was determined using gas chromatography as 1.80 mg/ml.

Low organic matter (LOM) and high organic matter (HOM) soils were used. The composition of the soils, as determined by the Rohm and Haas Company, was presented in Table 1 (attached). The soil was dosed with the working stock using a pressurized sprayer. The dosed soil was aged for approximately 24 hours then sprayed with pressurized water to simulate one-quarter inch of rain. The soil was allowed to dry for 24 hours or to dryness. Formulation blank soil samples were dosed with an amount of carrier ingredients equal to that applied to the highest test concentration.

The dilution water used was soft blended water with a hardness of 40-48 mg/l, an alkalinity of 44-56 mg/l, and a pH of 7.1-7.9. The conductivity of the 100-160 µmhos/cm.

C. **Dosage:** Ninety-six-hour static test. Based on preliminary studies, three nominal soil concentrations (0.35, 1.75, and 7.0 mg/kg dry weight), a formulation

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blank, and a negative (undosed) control were used per soil type.

D. **Design:** Twenty-four hours after the addition of the dilution water, midge larvae were added to the test containers one or two at a time. Two jars were used for each concentration and 15 midge larvae were used per jar. Three jars per concentration were set-up containing no larvae so that analytical measurements could be made without disturbing the test larvae. Slow aeration was provided from beginning of the second day through the end of the test. After 96 hours, the sediment was sieved and the number of live larvae determined. Missing larvae were counted as dead.

Soil samples from each test level were collected from the dry treated soil before the test and the "flooded" soil at test initiation and termination. The samples were filtered to dryness to exclude interstitial water. Water samples were collected at the beginning and end of the test. The concentration of ^{14}C-Goal 1.6E in all samples was determined by sample combustion followed by liquid scintillation counting or by direct liquid scintillation counting. The actual test concentrations in ppm were calculated by a computer.

E. **Statistics:** Survival of the exposed larvae were compared to control survival using t-tests.

12. **REPORTED RESULTS:** The concentrations of the test material measured during the test are presented in Table 10 (attached). Mean measured soil concentrations for the HOM soil were 0.26, 1.23, and 7.78 mg/kg. Mean measured soil concentrations for the LOM soils were 0.24, 1.28, and 5.1 mg/kg. The HOM soil bound appreciably more test material than the LOM soil. The concentration of ^{14}C-Goal 1.6E in the HOM soil was fairly constant throughout the 4-day exposure. No test material was detected in the water overlying the HOM loaded test jars. A decline in ^{14}C-Goal 1.6E concentrations in LOM soil was observed after the addition of "flood" water. Measurable amounts of ^{14}C-Goal 1.6E were detected in the "flood" water at the two highest test levels.

The responses of midge larvae are given in Table 12 (attached). Control survival in the HOM and LOM soils was 63 and 93%, respectively. The survival of midge larvae from the formulation blanks was not significantly different from the survival of their respective negative controls. "The investigation showed that soil-incorporated Goal 1.6E was not acutely lethal to midge larvae. In both high and low

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organic matter soil, concentrations of Goal 1.6E applied at up to 20 times the clear water LC_{50} had no observed effect on survival. Either Goal 1.6E is less biologically available to midge larvae when incorporated into the soil or it is not available in a toxic form."

Water quality data for the test are summarized in Table 11 (attached).

13. **STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES:** The authors presented no conclusions other than those previously mentioned.

Quality Assurance and Study Compliance Statements were included in the report, indicating that the study was conducted in accordance with FIFRA Good Laboratory Practice Standards set forth in 40 CFR Part 160.

14. **REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS:**

A. **Test Procedure:** Presently, no SEP exists for sediment toxicity tests using freshwater invertebrates. An ASTM (1990) Standard Guide for conducting sediment toxicity tests with freshwater invertebrates was used in conjunction with the SEP for acute freshwater invertebrate tests to evaluate the study. The test procedures were generally in accordance with the above protocols but deviated as follows:

Survival in one of the HOM control replicates was 53%. ASTM states that tests with <70% control survival are unacceptable.

The concentrations tested were not high enough to produce an EC_{50} value but were less than 100 ppm.

The dissolved oxygen concentration at the end of the first day ranged from 2.1 to 5.0 mg/l or 23.9 to 56.8% of saturation at 22°C. ASTM states that the dissolved oxygen should remain >40 and ≤100% of saturation throughout the test. As recommended by ASTM, aeration of the overlying water should have been maintained throughout the test.

ASTM states that test larvae must be collected from at least 3 separate egg masses. The larvae used in this test were collected from 2 egg masses.

The test chambers were not covered as recommended.

ASTM recommends that the conductivity, alkalinity, and hardness of the overlying water be measured at the beginning and end of the test. These parameters were not measured during the test.

The midge larvae used in this test were 17 days old. ASTM states that only midge larvae <16 days old should be used to initiate the test.

No acclimation to the overlying water was described in the report.

The photoperiod used in the test was not given in the report. A 16-hour light/8-hour dark photoperiod is recommended.

B. **Statistical Analysis:** The reviewer used Tukey's test and the Kruskal-Wallace test (Toxstat Version 3.3) to compare the survival of the controls to the survival of the exposed midges. There was no statistically significant affect on the exposed midges (see attached printouts 1-3).

C. **Discussion/Results:** The data presented in this report provides valuable information on the fate of soil-incorporated Goal 1.6E in a sediment-water system. Goal 1.6E appears to have a high affinity for sediment which is enhanced by the clay/silt/organic material content. It should be noted that the author's classification of the two soils based on organic material content is simplistic. It is quite possible that the differences in clay and silt content between the two soils, which as suggested by the breakdowns in Table 1 (attached) is significant, leads to the slightly different affinities for the test material.

The laboratory did not measure the concentration of Goal 1.6E in the interstitial water of the sediment (sediment samples were evaporated to dryness prior to analysis). Adams et al. (1985) determined that kepone in the interstitial water was the most likely route of exposure, and therefore toxicity, in *Chironomus tentans*. In the present test system, the LOM soil continually leached Goal 1.6E into the overlying water but the HOM soil did not. Greater mortality (7-40%) was observed in the test using HOM soil compared to the LOM soil test (7-20%). The HOM soil composition may have unduly stressed the midge larvae. However, no strong conclusions can be drawn regarding the effect of Goal 1.6E in the interstitial water because of high

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control mortality in the HOM soil test and the concentration of Goal 1.6E in interstitial water was not measured.

The study using HOM soil is not scientifically sound. Mortality in control replicate A of the HOM soil test was 47%. The study using LOM soil is scientifically sound but does not meet the guideline requirements for a sediment toxicity test using midge larvae. The concentrations tested were not high enough to produce an EC_{50} value but were less than 100 ppm. The 96-hour EC_{50} was >7.8 ppm (mg/kg), the highest concentration tested. A no-observed-effect concentration was not generated in the test.

D. **Adequacy of the Study:**

(1) **Classification:** Invalid for the test using HOM soil. Supplemental for the test using LOM soil.

(2) **Rationale:** HOM soil test: Mortality in control replicate A of the HOM soil test was 47%. LOM soil test: The concentrations tested were not high enough to produce an EC_{50} value but were less than 100 ppm.

(3) **Repairability:** No.

15. **COMPLETION OF ONE-LINER FOR STUDY:** Yes, 01-21-92.

REFERENCES:

ASTM. 1990. Standard Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates. E 1383-90.

Adams, W.J., R.A. Kimerle, and R.G. Mosher. 1985. Aquatic Safety Assessment of Chemicals Sorbed to Sediments. ASTM STP 854. pp. 429-453.

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RIN 0637-00

EFED Review - Oxyfluorfen

Page____ is not included in this copy.

Pages 8 through 12 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of 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.

TITLE: 420480-01, GOAL 1.6E, HOM SOIL, MIDGE SURVIVAL
FILE: A:42048001.DT1
TRANSFORM: ARC SINE(SQUARE ROOT(Y)) NUMBER OF GROUPS: 5

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	BLANK CONTROL	1	0.5300	0.8154
1	BLANK CONTROL	2	0.7300	1.0244
2	SOLVENT CONTROL	1	0.8700	1.2019
2	SOLVENT CONTROL	2	0.7300	1.0244
3	1X	1	0.8700	1.2019
3	1X	2	0.8000	1.1071
4	5X	1	0.9300	1.3030
4	5X	2	0.8000	1.1071
5	20X	1	0.6700	0.9589
5	20X	2	0.6000	0.8861

Shapiro Wilks test for normality
Data PASS normality test at P=0.01 level. Continue analysis.

Bartletts test for homogeneity of variance
Data PASS homogeneity test at 0.01 level. Continue analysis.

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	4	0.143	0.036	2.789
Within (Error)	5	0.064	0.013	
Total	9	0.207		

Critical F value = 5.19 (0.05,4,5)
Since F < Critical F FAIL TO REJECT Ho:All groups equal

TUKEY method of multiple comparisons

GROUP	IDENTIFICATION	TRANSFORMED MEAN	ORIGINAL MEAN	GROUP 0 0 0 0 0 1 5 2 3 4
1	BLANK CONTROL	0.920	0.630	\
5	20X	0.922	0.635	. \
2	SOLVENT CONTROL	1.113	0.800	. . \
3	1X	1.155	0.835	. . . \
4	5X	1.205	0.865	. . . . \

* = significant difference (p=0.05) . = no significant difference
Tukey value (5,5) = 5.67 s = 0.013

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TITLE: 420480-01, GOAL 1.6E, LOM SOIL, MIDGE SURVIVAL
FILE: A:42048001.DT2
TRANSFORM: ARC SINE(SQUARE ROOT(Y)) NUMBER OF GROUPS: 5

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	BLANK CONTROL	1	1.0000	1.4413
1	BLANK CONTROL	2	0.8700	1.2019
2	SOLVENT CONTROL	1	0.6700	0.9589
2	SOLVENT CONTROL	2	0.8000	1.1071
3	1X	1	0.9300	1.3030
3	1X	2	0.8700	1.2019
4	5X	1	0.8700	1.2019
4	5X	2	0.8000	1.1071
5	20X	1	0.9300	1.3030
5	20X	2	0.9300	1.3030

Shapiro Wilks test for normality
Data PASS normality test at P=0.01 level. Continue analysis.

Hartley test for homogeneity of variance
Bartletts test for homogeneity of variance
These two tests can not be performed because at least one group has zero variance.

Data FAIL to meet homogeneity of variance assumption.

KRUSKAL-WALLIS ANOVA BY RANKS - TABLE 1 OF 2 (p=0.05)

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	RANK SUM
1	BLANK CONTROL	1.322	0.935	15.000
2	SOLVENT CONTROL	1.033	0.735	3.500
3	1X	1.252	0.900	13.000
4	5X	1.155	0.835	7.500
5	20X	1.303	0.930	16.000

Calculated H Value = 6.548 Critical H Value Table = 7.418
Since Calc H < Crit H FAIL TO REJECT Ho:All groups are equal.

DUNNS MULTIPLE COMPARISON - KRUSKAL-WALLIS - TABLE 2 OF 2 (p=0.05)

GROUP	IDENTIFICATION	TRANSFORMED MEAN	ORIGINAL MEAN	GROUP 0 2	0 4	0 3	0 5	0 1
2	SOLVENT CONTROL	1.033	0.735	\				
4	5X	1.155	0.835	.	\			
3	1X	1.252	0.900	.	.	\		
5	20X	1.303	0.930	.	.	.	\	
1	BLANK CONTROL	1.322	0.935	.	.	.	.	\

* = significant difference (p=0.05) . = no significant difference
Table q value (0.05,5) = 2.807 SE = 2.944

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420480-01, GOAL 1.6E, LOM SOIL, MIDGE SURVIVAL
File: A:42048001.DT2 Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	4	0.115	0.029	2.907
Within (Error)	5	0.049	0.010	
Total	9	0.164		

Critical F value = 5.19 (0.05,4,5)
Since F < Critical F FAIL TO REJECT Ho:All groups equal

TUKEY method of multiple comparisons

GROUP	IDENTIFICATION	TRANSFORMED MEAN	ORIGINAL MEAN	GROUP 0 2	0 4	0 3	0 5	0 1
2	SOLVENT CONTROL	1.033	0.735	\				
4	5X	1.155	0.835	.	\			
3	1X	1.252	0.900	.	.	\		
5	20X	1.303	0.930	.	.	.	\	
1	BLANK CONTROL	1.322	0.935	.	.	.	.	\

* = significant difference (p=0.05) . = no significant difference
Tukey value (5,5) = 5.67 s = 0.010

Shaughnessey # 111601 Chemical Name Oxyfluorofen Chemical Class _____ Page 1 of 1

Study/Species/Lab/ MRID #	Chemical % a.i.	Results	Reviewer/ Date	Validation Status
48-Hour EC_{50}	_____	EC_{50} = pp (95% C.L.) Control Mortality (%) = Solvent Control Mortality (%) =		
Species:		Slope = # Animals/Level = Temperature =		
Lab:		48-Hour Dose Level pp /(% Effect) ( ), ( ), ( ), ( ), ( )	_____	_____
MRID #		Comments:		
96-Hour LC_{50}	19.5	LC_{50} = >7.8 ppm* (95% C.L. N/A) Control Mortality (%) = 0-47% Solvent Control Mortality (%) = 13-33%		
Species: Chironomus tentans		Slope = N/A # Animals/Level = 15 Temperature = 20±2°C	LMR 1/21/92	Testing Hom Soil Invalid
Lab: Analytical Bio-chemistry		96-Hour Dose Level ppm* /(% Mortality) Hom 0.26 (17), 1.23 (13), 7.78 (37), 0.27 (10), 1.28 (17), 5.1 (7)		test using LOM Soil: Supplemental
MRID # 420480-01		Comments: * Mean measured Concentration LOM		

OF 6

DATA EVALUATION RECORD

1. **CHEMICAL:** Oxyflurofen.
Shaughnessey No. 111601.

2. **TEST MATERIAL:** 1) Goal® 1.6E (6-2757); Lot No. K6066; 19.5% active ingredient; a brown liquid.
2) ^{14}C-Goal® 1.6E; No. 568.0103; 0.504 mCi; a yellow powder.

3. **STUDY TYPE:** Freshwater Invertebrate Static Acute Toxicity Test. Species Tested: Midge (*Chironomus tentans*).

4. **CITATION:** Forbis, A.D. 1986. Acute Toxicity of ^{14}C-Goal® 1.6E Herbicide to Midge Larvae (*Chironomus tentans*). Final Study No. 34971. Rohm and Haas Report No. 87RC-0003. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO. Submitted by Rohm and Haas Company, Spring House, PA. EPA MRID No. 420480-02.

5. **REVIEWED BY:**

Louis M. Rifici, M.S.
Associate Scientist
KBN Engineering and
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Signature: Louis M. Rifici
Date: 2/19/92

6. **APPROVED BY:**

Pim Kosalwat, Ph.D.
Senior Scientist
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Signature: P. Kosalwat
Date: 2/19/92

Henry T. Craven, M.S.
Supervisor, EEB/EFED
USEPA

Signature: Henry T. Craven
Date: 3/4/92

3/4/92

7. **CONCLUSIONS:** This study is not scientifically sound and does not meet the guideline requirements for a static-acute toxicity test using the midge larvae, *Chironomus tentans*. The midge larvae were not in the same developmental stage (reported as third-fourth instar) and were not approximately the same age. The test procedures and culture conditions were not clearly outlined in the test report. The 48-hour LC_{50} value of 0.27 mg/l (mean measured concentration) classifies Goal 1.6E as highly toxic to midge larvae. The NOEC was 0.085 mg/l mean measured concentration.

8. **RECOMMENDATIONS:** N/A.

9. **BACKGROUND:**

10. **DISCUSSION OF INDIVIDUAL TESTS:** N/A.

11. **MATERIALS AND METHODS:**

A. **Test Animals:** Midge larvae (*Chironomus tentans*) were obtained from in-house cultures. The cultures were fed a suspension of Tetramin® and cereal leaves.

B. **Test System:** Vessels used in the test were 250-ml glass beakers containing 200 ml of test solution.

The working stock for the test was prepared by mixing 16.5 µl of Goal 1.6E formulation with 13 µl of ^{14}C-Goal 1.6E primary stock solution (prepared in acetone) and diluting to 5 ml in deionized water. The preparation of the test solutions was not described in the report. The solvent blank chambers received an aliquot of solvent blank equivalent to that used in the highest test concentration (0.074 ml).

The dilution water used was aged well water with a hardness of 250-258 mg/l, an alkalinity of 154-160 mg/l, a conductivity of 700 µmhos/cm, and a pH of 8.2. The characteristics of the well water source are given in Table 1 (attached).

Lighting was maintained at 70-100 ft-candles on a 16-hour light photoperiod with 30-minute dawn and dusk simulation periods. The vessels were held in a temperature-controlled area (20 ±2.0°C).

C. **Dosage:** Forty-eight-hour static test. Based on a preliminary test, seven nominal concentrations (0.054, 0.10, 0.18, 0.32, 0.54, 1.0, and 1.8 mg/l), a solvent blank, and a dilution water control were used.

D. **Design:** Two beakers were used for each concentration and ten midge larvae were used per beaker. All beakers were observed once every 24 hours for mortality and abnormal effects. The temperature, dissolved oxygen (DO), and pH were measured in the control, solvent blank, and low and high concentrations containing live midge larvae at the beginning and end of the test.

The concentration of ^{14}C-Goal in water samples from all replicates was determined by liquid scintillation counting at test initiation and termination. The

actual test concentrations in ppm were calculated by a computer.

E. **Statistics:** The 48-hour LC_{50} value and associated confidence interval was determined using a computer program developed by Stephan et al. (1978).

12. **REPORTED RESULTS:** The mean measured concentrations were 0.049, 0.085, 0.15, 0.26, 0.39, 0.65, and 1.23 mg/l (Table 4, attached). These values represent 65-91% of nominal concentrations. Measured concentrations decreased slightly between sampling times with the change being marked in the two highest concentrations.

The responses of midge larvae are given in Table A3 (attached). The 48-hour LC_{50} was determined as 0.35 mg/l nominal concentration (95% C.I. = 0.28-0.43 mg/l nominal concentrations) using the probit method. The no-observed-effect concentration (NOEC), based on the lack of mortality and abnormal effects, was 0.10 mg/l nominal concentration after 48 hours.

The DO ranged from 6.9 to 8.8 mg/l or 75 to 96% of saturation at 20°C. The pH values ranged from 8.2 to 8.4. The temperature remained 20°C throughout the test.

13. **STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES:** The authors presented no conclusions other than those already presented.

Quality Assurance and Study Compliance Statements were included in the report, indicating that the study was conducted in accordance with FIFRA Good Laboratory Practice Standards set forth in 40 CFR Part 160.

14. **REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS:**

A. **Test Procedure:** The test procedures were generally in accordance with protocols recommended by the guidelines, but deviated from the SEP as follows:

The hardness of the dilution water (250-258 mg/l) was higher than recommended (<200 mg/l).

The midge larvae were in the third or fourth instar at test initiation. The age of the midge larvae at test initiation was not given in the report. Midge larvae used in the test should be the same size, age, and in their second or third instar.

The culture conditions and acclimation period used were not described in the report.

A description of the methods used to prepare the test solutions was not provided in the report. The test solutions should be prepared within 30 minutes of test initiation.

The temperature of the test solutions was determined at 0 and 48 hours. The SEP states that the temperature should be monitored continuously in at least one test vessel during the test.

B. **Statistical Analysis:** The reviewer used EPA's Toxanal program and mean measured concentrations to determine the LC_{50} value. The 48-hour LC_{50} was determined as 0.27 mg/l (95% C.I. = 0.22-0.33 mg/l) using the probit method. The slope of the concentration-response curve was 3.63. Using mean measured concentrations, the NOEC was 0.085 mg/l.

C. **Discussion/Results:** The author used the nominal concentrations to determine the LC_{50} value. In the reviewer's opinion, the mean measured concentrations adequately reflect the actual test concentrations for most of the levels tested (the highest two levels being the exceptions). Using mean measured concentrations in determining the LC_{50} value represents a more conservative approach than the author's.

Although not specified in the SEP, the study would have probably benefitted from the addition of a substrate to the test chambers. Glass beads about the size of sand grains or glass tubes have commonly been used. Midge larvae (*Chironomus* sp.) are benthic animals which build burrows in culture when substrates are provided. Whether the larvae were stressed by the lack of substrate in this test is unknown.

The alkalinity of the dilution water reported in the test summary section (154-160 mg/l; p. 7) was much different from that reported in Table 1 (325-375 mg/l). This discrepancy is not explained by the author.

The test procedures and culture conditions were not clearly outlined in the test report. Test animals were not in the same developmental stage (reported as third-fourth instar). The response or sensitivity of animals at different stages of development to test chemicals may not be the same. All midge larvae must be in the

same instar and approximately the same age. This study is not scientifically sound and does not meet the guideline requirements for a static-acute toxicity test using the midge larvae, *Chironomus tentans*. The 48-hour LC_{50} value of 0.27 mg/l (mean measured concentration) classifies Goal 1.6E as highly toxic to midge larvae. The NOEC was 0.085 mg/l mean measured concentration.

D. **Adequacy of the Study:**

(1) **Classification:** Invalid.

(2) **Rationale:** The midge larvae were not in the same developmental stage and were not of approximately the same age. The test procedures and culture conditions were not clearly outlined in the test report.

(3) **Repairability:** No.

15. **COMPLETION OF ONE-LINER FOR STUDY:** Yes, 01-16-92.

RIN 0637-00

EFED Review - Oxyfluorfen

Page____ is not included in this copy.

Pages 22 through 24 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of 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.

RIFICI OXYFLUOROFEN CHIRONOMUS TENTANS 1-16-92

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CONC.	NUMBER EXPOSED	NUMBER DEAD	PERCENT DEAD	BINOMIAL PROB. (PERCENT)
1.23	20	20	100	9.536742E-05
.65	20	17	85	.1288414
.39	20	16	80	.5908966
.26	20	9	45	41.19014
.15	20	5	25	2.069473
.085	20	0	0	9.536742E-05
.049	20	0	0	9.536742E-05

THE BINOMIAL TEST SHOWS THAT .15 AND .39 CAN BE USED AS STATISTICALLY SOUND CONSERVATIVE 95 PERCENT CONFIDENCE LIMITS, BECAUSE THE ACTUAL CONFIDENCE LEVEL ASSOCIATED WITH THESE LIMITS IS GREATER THAN 95 PERCENT.

AN APPROXIMATE LC50 FOR THIS SET OF DATA IS .2746804

RESULTS CALCULATED USING THE MOVING AVERAGE METHOD

SPAN	G	LC50	95 PERCENT CONFIDENCE LIMITS	
5	.1259553	.2684319	.227041	.3208317

RESULTS CALCULATED USING THE PROBIT METHOD

ITERATIONS	G	H	GOODNESS OF FIT PROBABILITY
4	8.172968E-02	1	.6133946

SLOPE = 3.630965

95 PERCENT CONFIDENCE LIMITS = 2.59293 AND 4.669

LC50 = .2682826

95 PERCENT CONFIDENCE LIMITS = .2207918 AND .3254159

LC10 = .1199011

95 PERCENT CONFIDENCE LIMITS = 8.101996E-02 AND .1534721

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Shaughnessey # 111601 Chemical Name Oxyflurofen Chemical Class _____ Page 1 of 1

Study/Species/Lab/ MRID #	Chemical % a.i.	Results	Reviewer/ Date	Validation Status

48-Hour EC_{50}	19.5	EC_{50} = 0.27 ppm* (95% C.L. 0.22 - 0.33) Probit
		Control Mortality (%) = 0
		Solvent Control Mortality (%) = 0
		Slope = 3.63 # Animals/Level = 20
		Temperature = 20
		48-Hour Dose Level ppm* /(% Effect)
		0.049 (0), 0.085 (0), 0.15 (25), 0.26 (45), 0.39 (80), 0.65 (85), 1.23 (100).
		Comments: * mean measured concentration
			LMR 1/16/92	"INVALID"

96-Hour LC_{50}		LC_{50} = ppm (95% C.L.) Control Mortality (%) = Solvent Control Mortality (%) = Slope = # Animals/Level = Temperature = 96-Hour Dose Level ppm /(% Mortality) ( ), ( ), ( ), ( ), ( ) Comments:		

Species: Chironomus tentans

Lab: Analytical Bio-Chemistry

MRID # 420480-02

Species:

Lab:

MRID #

DATA EVALUATION RECORD

1. **CHEMICAL:** Oxyflurofen.
Shaughnessey No. 111601.

2. **TEST MATERIAL:** 1) Goal® 1.6E (6-2757); Lot No. K6066; 19.5% active ingredient; a brown liquid.
2) ^{14}C-Goal® 1.6E; No. 568.0103; 0.504 mCi; a yellow powder.

3. **STUDY TYPE:** Freshwater Invertebrate Static Acute Toxicity Test. Species Tested: Mayfly (*Hexagenia* sp.)

4. **CITATION:** Swigert, J.P. 1986. Acute Toxicity of ^{14}C-Goal® 1.6E Herbicide to the Mayfly, *Hexagenia* sp. Final Report No. 34972. Rohm and Haas Report No. 87RC-0008. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO. Submitted by Rohm and Haas Company, Spring House, PA. EPA MRID No. 420480-03.

5. **REVIEWED BY:**

Louis M. Rifici, M.S.
Associate Scientist
KBN Engineering and
Applied Sciences, Inc.

Signature: Louis M Rifici
Date: 2/19/92

6. **APPROVED BY:**

Pim Kosalwat, Ph.D.
Senior Scientist
KBN Engineering and
Applied Sciences, Inc.

Signature: P. Kosalwat TB 3/4/92
Date: 2/19/92

Henry T. Craven, M.S.
Supervisor, EEB/EFED
USEPA

Signature: Henry T. Craven
Date: 3/4/92

7. **CONCLUSIONS:** This study is not scientifically sound. The measured concentrations greatly decreased during the test period indicating that the actual concentrations the mayflies were exposed to are unknown. The 48-hour EC_{50} value of 0.11 mg/l (mean measured concentration) classifies Goal 1.6E as highly toxic to mayfly nymphs. An NOEC value could not be determined in the test due to mortality at all levels.

8. **RECOMMENDATIONS:** N/A.

9. **BACKGROUND:**

27

6.5

10. **DISCUSSION OF INDIVIDUAL TESTS:** N/A.

11. **MATERIALS AND METHODS:**

A. **Test Animals:** Mayfly nymphs (*Hexagenia* sp.) were obtained from a commercial supplier and maintained in laboratory aquaria. The mayflies were fed a suspension of fish food and cereal leaves. A representative sample from the test group had a mean weight of 0.27 ±0.085 g and a mean length of 28 ±2.9 mm.

B. **Test System:** The test vessels used were 40-l glass aquaria containing 8 l of test solution. The mayflies were confined in 2-l glass aquaria each containing approximately 1 l of solution. One end of each enclosure was replaced with nytex® screen. Sections of glass tubing were placed in the enclosure aquaria to act as artificial burrows for the mayflies. The test vessels were placed in a temperature-controlled water bath (20 ±2°C).

The working stock for the test was prepared by mixing 284 µl of Goal 1.6E formulation with 1.30 ml of ^{14}C-Goal 1.6E primary stock solution (prepared in acetone) and diluting to 50 ml in deionized water. The preparation of the test solutions was not described in the report. The concentration of the working stock was determined using gas chromatography to be 1.37 mg/ml.

The dilution water used was well water with a hardness of 225-275 mg/l, an alkalinity of 325-375 mg/l, a conductivity of 700 µmhos/cm, and a pH of 7.8-8.3.

C. **Dosage:** Forty-eight-hour static test. Based on a preliminary test, seven nominal concentrations (0.010, 0.022, 0.046, 0.1, 0.22, 0.46, and 1.0 mg/l), a formulation control (0.80 mg formulation blank/l), and a dilution water control were used.

D. **Design:** Twenty mayflies were distributed to each aquarium (5 mayflies per enclosure, 4 enclosures per aquarium). All aquaria were observed once every 24 hours for mortality and abnormal effects. The temperature, dissolved oxygen, and pH were measured in the dilution water control, formulation control, and low and high concentrations at the beginning and end of the test.

The concentration of ^{14}C-Goal in water samples from all test levels (excluding the dilution water control) was

determined by liquid scintillation counting at test initiation and termination. The actual test concentrations in ppm were calculated by a computer.

E. **Statistics:** The 24 and 48-hour LC_{50} and EC_{50} values and associated confidence intervals were determined using a computer program developed by Stephan et al. (1978).

12. **REPORTED RESULTS:** The measured concentrations were presented in Table 2 (attached). The mean measured concentrations (reviewer calculated) were 0.0055, 0.014, 0.024, 0.055, 0.114, 0.27, and 0.497 mg/l. The concentration of ^{14}C-Goal 1.6E decreased substantially during the test. Measured concentrations averaged 78% of nominal at test initiation but only 32% of nominal at test termination.

The responses of mayfly nymphs are given in Table 4 (attached). Behavioral and sublethal effects (loss of equilibrium and quiescence) and mortality were noted at all levels except the dilution water control. The components in the formulation appeared to affect the nymphs. Mortality in the formulation control was 15% after 48 hours. The lack of a clear dose-response was attributed to the formulation components present in the test solutions.

Based on nominal concentrations, the 48-hour LC_{50} and EC_{50} values were 0.42 mg/l (95% C.I. = 0.24-1.0 mg/l) and 0.18 mg/l (95% C.I. = 0.1-0.46 mg/l), respectively. The author did not report a no-observed-effect concentration (NOEC).

The water quality measurements during the test are presented in Table 3 (attached). The values were considered within acceptable ranges for aquatic tests.

13. **STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES:** The author stated that the LC_{50} and EC_{50} values presented do not reflect the true toxicity of Goal 1.6E because the observed toxicity encompasses both solvent (formulation components) and compound toxicity.

Quality Assurance and Study Compliance Statements were included in the report, indicating that the study was conducted in accordance with FIFRA Good Laboratory Practice Standards set forth in 40 CFR Part 160.

14. **REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS:**

A. **Test Procedure:** The test procedures were generally in accordance with protocols recommended by the SEP and

Subdivision E guidelines. Deviations were noted as follows:

The duration of the test was 48 hours; 96 hours of exposure are required by Subdivision E.

The hardness of the dilution water (225-275 mg/l) was higher than recommended (≤200 mg/l).

The recommended test temperature for *Hexagenia* sp. nymphs is 22°C. The temperature during the test was 20°C.

The age and developmental stage of the mayfly nymphs were not given in the report. Mayfly nymphs used in the test should be the same size, age, and in their second instar.

Based on an average weight of 0.27 g, the loading in the test chambers was approximately 1.35 g/l. The recommended biomass loading for tests performed at 20°C is 0.5 g/l.

The acclimation period used was not described in the report.

Organisms must be randomly assigned to the test vessels. The author does not mention if random assignment was used.

A description of the methods used to prepare the test solutions was not provided in the report. The test solutions should be prepared within 30 minutes of test initiation.

The photoperiod used in the test was not described in the report. A 16-hour light/8-hour dark photoperiod with 15-30 minute dawn and dusk simulations are recommended.

Each selected nominal concentration was 45-48% of the next highest concentration. The SEP recommends that each concentration be at least 60% of the next highest.

The temperature of the test solutions was determined at 0 and 48 hours. The SEP states that the temperature should be monitored at least every six hours in at least one test vessel during the test.

B. **Statistical Analysis:** The reviewer used EPA's Toxanal program and mean measured concentrations to determine the 48-hour EC_{50} value. Using mean measured concentrations in determining the EC_{50} value represents a more conservative approach than the author's. The 48-hour EC_{50} was 0.11 mg/l (95% C.I. = 0.085-0.152 mg/l) using the moving average method. Due to lethal effects at all test levels, an NOEC value was not determined in this test.

C. **Discussion/Results:** The materials and methods used in this test were not clearly defined in the report. Collectively, the missing information, deviations listed in Section 14.A., and the unstable test concentrations cast doubt on the usefulness of the results in pesticide risk assessment.

The formulation components were toxic to the mayflies. The EC_{50} therefore encompasses both solvent (formulation components) and compound toxicity. The results of this test did not accurately represent the toxicity of the active ingredient, oxyflurofen.

One additional point warrants mention, the author called each mayfly enclosure within the test vessels a replicate. The enclosures were clearly not replicates because one test solution was shared among the 4 enclosures.

This study is not scientifically sound. The actual concentrations the mayflies were exposed to are unknown. The 48-hour EC_{50} value of 0.11 mg/l (mean measured concentration) classifies Goal 1.6E as highly toxic to mayfly nymphs. An NOEC value could not be determined in the test.

D. **Adequacy of the Study:**

(1) **Classification:** Invalid.

(2) **Rationale:** The measured concentrations greatly decreased during the test period indicating that the actual concentrations the mayflies were exposed to are unknown. The formulation components were toxic to the mayflies, therefore, the EC_{50} does not reflect the toxicity of the active ingredient, oxyflurofen. The test was too short.

(3) **Repairability:** No.

15. **COMPLETION OF ONE-LINER FOR STUDY:** Yes, 01-29-92.

RIN 0637-00

EFED Review - Oxyfluorfen

Page____ is not included in this copy.

Pages 33 through 35 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of 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.

NOTE: BECAUSE THERE WAS CONTROL MORTALITY, AND NONE
OF THE LOWER CONCENTRATIONS PRODUCED ZERO MORTALITY,
THE DATA HAS BEEN SUBJECTED TO ABBOTT'S CORRECTION.

RIFICI OXYFLUROFEN HEXAGENIA 1-29-92 EC50 calculation

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CONC.	NUMBER EXPOSED	NUMBER DEAD	PERCENT DEAD	BINOMIAL PROB. (PERCENT)
.497	17	16	94.1177	1.373291E-02
.27	17	15	88.2353	.1174927
.114	17	11	64.7059	16.61529
.055	20	1	5	2.002716E-03
.024	17	1	5.8824	1.373291E-02
.014	17	0	0	7.629394E-04
.0055	20	1	5	2.002716E-03

THE BINOMIAL TEST SHOWS THAT .055 AND .27 CAN BE
USED AS STATISTICALLY SOUND CONSERVATIVE 95 PERCENT
CONFIDENCE LIMITS, BECAUSE THE ACTUAL CONFIDENCE LEVEL
ASSOCIATED WITH THESE LIMITS IS GREATER THAN 95 PERCENT.

AN APPROXIMATE LC50 FOR THIS SET OF DATA IS 9.755269E-02

RESULTS CALCULATED USING THE MOVING AVERAGE METHOD

SPAN	G	LC50	95 PERCENT CONFIDENCE LIMITS	
5	4.845236E-02	.1120341	8.536532E-02	0.1519597

LC 1/29/92

RESULTS CALCULATED USING THE PROBIT METHOD

ITERATIONS	G	H	GOODNESS OF FIT PROBABILITY
4	1.042929	6.846185	0

A PROBABILITY OF 0 MEANS THAT IT IS LESS THAN 0.001.

SINCE THE PROBABILITY IS LESS THAN 0.05, RESULTS CALCULATED
USING THE PROBIT METHOD PROBABLY SHOULD NOT BE USED.

SLOPE = 2.281526
95 PERCENT CONFIDENCE LIMITS =-4.845715E-02 AND 4.61151

LC50 = .1062098
95 PERCENT CONFIDENCE LIMITS = 0 AND +INFINITY

LC10 = 2.947947E-02
95 PERCENT CONFIDENCE LIMITS = 0 AND 8.522384E-02

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Shaughnessey # 111601 Chemical Name Oxyfluorfen Chemical Class _____ Page 1 of 1

Study/Species/Lab/ MRID #	Chemical % a.i.	Results	Reviewer/ Date	Validation Status

48-Hour EC_{50}	19.5	EC_{50} = 0.11 ppm* (95% C.L. Moving Average ~~0.85~~ 0.085-0.152) Control Mortality (%) = 0
		Solvent Control Mortality (%) = 15%
		Slope = N/A # Animals/Level = 20
		Temperature = 20
		48-Hour Dose Level ppm* /(% Effect)
		0.0055(5), 0.014 (15), 0.024 (20), 0.055 (5), 0.114 (70), 0.27 (90), 0.497 (95)
			LMR 1/29/92	INVALID

Species: Hexagenia sp.				
Lab: Analytical Bio-Chemistry				
MRID # 420480-03		Comments: * mean measured concentration of active ingredient		
96-Hour LC_{50}		LC_{50} = pp (95% C.L.) Control Mortality (%) = Solvent Control Mortality (%) = Slope = # Animals/Level = Temperature = 96-Hour Dose Level pp /(% Mortality) ( ), ( ), ( ), ( ), ( )		
Species:				
Lab:				
MRID #		Comments:		

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