5-23-86

DYNAMAC CORPORATION

CHLOROPICRIN

Final Report

Task 1: Review and Evaluation of Individual Studies

Task 2: Environmental Fate and Exposure Assessment

Contract No. 68-02-4250

MAY 23, 1986

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
11140 Rockville Pike
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CHLOROPICRIN

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INTRODUCTION

This report is a scientific evaluation of environmental fate data submitted to EPA by the Great Lakes Chemical Corporation as an addendum to the application for reregistration of chloropicrin. Chloropicrin is a nonspecific preplant soil fumigant registered for use on terrestrial food crop ("all crops", including field, vegetable, and orchard), aquatic food crop, terrestrial nonfood crop (tobacco and ornamentals), greenhouse, and domestic outdoor use sites. Application rates range from 150 to 1076 lb ai/A, and the maximum preplant aeration period is 7 days. Outdoor applications generally are accomplished through the use of mechanical equipment such as chisels and plow soles attached to tractors; small indoor and outdoor soil beds may be injected using hand-held equipment. The injected soil must be immediately sealed by tamping the soil, wetting the top layer of soil, and/or covering the soil with polyethylene plastic tarpaulins.

In addition to the five studies reviewed herein, chloropicrin studies have been previously reviewed by EAB and Dynamac. The contribution of all studies that have been reviewed to date toward fulfillment of data requirements is considered under Recommendations. The structure of chloropicrin is illustrated in the Appendix.

CASE GS -- CHLOROPICRIN STUDY 1 PM --

CHEM 081501 Chloropicrin

BRANCH EAB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID None CONTENT CAT 01

Craine, E.M. 1985a. A hydrolysis study with chloropicrin. Research Report, Analytical 85:6. Project: WIL-48003. WIL Research Laboratories, Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

SUBST. CLASS = S.

DIRECT RVW TIME = 5 (MH) START-DATE END DATE

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CONCLUSION:

Degradation - Hydrolysis

This study is scientifically invalid because the data were too variable to assess the decline of chloropicrin in water. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because the test substance was not characterized and the material balance was incomplete.

MATERIALS AND METHODS:

Sterile aqueous solutions, buffered at pH 5, 7, and 9, were treated with chloropicrin (test substance uncharacterized) at 110.28 ppm (pH 5 solution), 42.10 ppm (pH 7 solution), or 204.73 ppm (pH 9 solution) and transferred to amber-colored flasks. The flasks were sealed and incubated at 25°C. Solution and flask headspace (air) samples were taken at days 0, 2, 4, 9, 14, 21, and 29.

The solution samples were diluted with methanol and analyzed by GC with electron-capture detection. Air samples were analyzed directly by GC.

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REPORTED RESULTS:

The concentration of chloropicrin in solutions of pH 5, 7, and 9 decreased during 29 days of incubation, while the concentration of chloropicrin in the air above the solutions varied from 0.00 to 17.43 mg/bottle (Table 1). Chloroform, a chloropicrin degradate, was detected at up to 0.04 mg/bottle in solution and 4.50 mg/bottle in the air.

DISCUSSION:

1. In the pH 5 solution, the concentration of chloropicrin in solution decreased between 0 and 2 days, then appeared to stabilize. A similar pattern was observed in the pH 9 solution, except that a rapid decrease occurred between 21 and 29 days. In the pH 7 solution, concentrations varied between 7 and 14 mg with no discernible pattern. The concentrations of chloropicrin in the air and chloroform in the solution and air, added to the concentration of chloropicrin in solution do not provide a complete material balance. According to the study authors, chloropicrin is readily adsorbed to glass surfaces; the incubation flasks were glass reagent bottles.

2. The test substance was not characterized.

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Table 1. Chloropicrin and chloroform (mg/bottle) in sterile buffered solutions (pH 5, 7, and 9) treated with chloropicrin and incubated at 25°C.[a]

Sampling interval (days)	Solution		Headspace air[b]	
	Chloropicrin	Chloroform	Chloropicrin	Chloroform
		pH 5		
0	27.57	--[c]	--	--
2	23.57	0.01	2.02	ND[d]
4	23.70	0.01	2.27	ND
9	21.13	0.01	0.96	0.03
14	22.01	0.01	0.97	ND
21	19.32	0.01	1.09	0.81
29	21.11	0.01	1.29	--
		pH 7		
0	10.53	--	--	--
2	14.24	0.02	1.37	0.01
4	10.39	0.02	1.56	0.11
9	7.25	0.02	0.63	0.09
14	11.26	0.02	2.18	0.01
21	10.40	0.02	0.64	ND
29	8.58	0.02	0.76	ND
		pH 9		
0	51.182	--	--	--
2	39.98	0.02	3.01	0.01
4	40.07	0.02	17.43	0.01
9	39.39	0.01	3.65	0.73
14	40.81	0.04	2.06	4.50
21	40.70	0.02	2.59	0.01
29	10.24	0.01	0.70	ND

[a] The solutions were treated at 110.28 ppm (pH 5), 42.12 ppm (pH 7) and 204.72 ppm (pH 9).

[b] Air samples collected from the air space in the flasks.

[c] Not reported.

[d] Not detected; detection limit not reported.

CASE GS -- CHLOROPICRIN STUDY 2 PM --

CHEM 081501 Chloropicrin

BRANCH EAB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID None CONTENT CAT 01

Castro, C.E. and N.O. Belser. 1981. Photohydrolysis of methyl bromide and chloropicrin. J. Agric. Food Chem. 29(5):1005-1008. Acc. No. 260211.

SUBST. CLASS = S.

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CONCLUSION:

Degradation - Photodegradation in Water

This study cannot be validated because the procedures were insufficiently described and the reported results were not supported with quantitative data. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because the test substance was uncharacterized, the test solutions were incompletely characterized, sunlight and the artificial light were incompletely characterized, degradates were not identified, and there was no material balance.

MATERIALS AND METHODS:

Neutral aqueous solutions (solutions not further characterized) contained in quartz cuvettes or Pyrex flasks, were treated with chloropicrin (test substance uncharacterized) at 1 ppm. The majority of the flasks were irradiated with natural August sunlight or a 150-W floodlamp (light sources not further characterized); the remainder were kept under "ambient" conditions as a control. Samples were taken 25, 50, and 75 hours posttreatment and analyzed directly using GC.

REPORTED RESULTS:

Chloropicrin, at 1 ppm, degraded with a half-life of 60-80 hours in neutral aqueous solutions irradiated with either natural sunlight or the floodlamp (Figure 1). Chloropicrin was stable for 240 hrs in solution under ambient conditions.

DISCUSSION:

1. The description of the experiment was too general to assess whether the study was adequate. The test substance and test solutions were uncharacterized, temperatures were not reported, and the light sources were incompletely characterized.

2. Data were reported in graphical form only. No tabular quantitative data and no raw data were provided to support the conclusions.

3. Degradates were not identified. There was no material balance.

4. "Ambient" conditions in the dark control were not adequately defined.

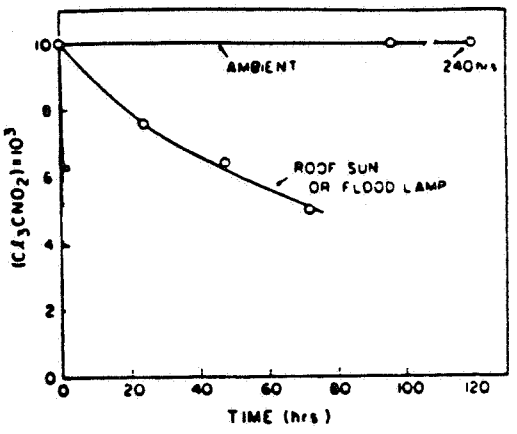

Figure 1. The decline of chloropicrin in water treated at 1.0 ppm and irradiated with sunlight, a 150-W floodlamp, or "ambient" light.

CASE GS -- CHLOROPICRIN STUDY 3 PM --

CHEM 081501 Chloropicrin

BRANCH EAB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID None CONTENT CAT 01

Craine, E.M. 1985c. An aerobic soil metabolism study with chloropicrin. Research Report, Analytical 85:12. Project: WIL-48004. WIL Research Laboratories Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

SUBST. CLASS = S.

DIRECT RVW TIME = 5 (MH) START-DATE END DATE

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CONCLUSIONS:

Metabolism - Aerobic Soil

1. This study is scientifically valid.

2. Chloropicrin (test substance uncharacterized), at 11.35 mg/100 g of soil, dissipated with a half-life of <1 hr in sandy loam soil incubated in the dark under aerobic conditions at 25°C. Inorganic chloride increased by 7.30 mg/100 g of soil during the 24-hour study. Five additional degradates were isolated but neither quantified nor identified.

3. This study does not fulfill EPA Data Requirements for Registering Pesticides because the test substance was uncharacterized, the degradates were not quantified or identified, complete soil characteristics were not reported, and the material balance was incomplete.

MATERIALS AND METHODS:

Samples (100 g) of Wooster sandy loam soil (59% sand, 34% silt, 7% clay, 7.2% organic matter) were flushed with oxygen and treated with 11.35 mg of chloropicrin (test substance uncharacterized). The flasks were stoppered and incubated in the dark at 25°C. Soil samples were taken at 1, 2, 4, 8, 12, 16, and 24 hours posttreatment.

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The soil samples were distilled with water and isooctane, after which the distillate was combined with sodium chloride and shaken. The solution was allowed to separate into aqueous and solvent phases. The iosoctane phase was dried with anhydrous sodium sulfate, diluted, and analyzed by GC with electron-capture detection. Recovery from fortified samples averaged 98-104%.

REPORTED RESULTS:

Chloropicrin dissipated with a half-life of <1 hour (Table 1). Five degradates were isolated but not identified; inorganic chloride in the soil increased by 7.30 mg/100 g during 24 hours of incubation.

DISCUSSION:

1. The test substance was uncharacterized.

2. The degradates were not quantified or identified.

3. The test soil was reported to be a silt loam, but is a sandy loam based on the USDA Soil Textural Classification System. In addition, complete soil characteristics, such as CEC and pH were not reported.

4. The material balance was incomplete.

5. From the data, it appears the initial rapid decline of chloropicrin in the soil is the result of chloropicrin volatilizing from the soil and reaching an equilibrium between the soil and the air in the headspace. Once equilibrium is established, at ~1 hour, the remaining decline is due to degradation (chemical and/or microbial). Since the concentration of chloropicrin in the headspace was not measured and the material balance was incomplete, the mechanism of chloropicrin dissipation is uncertain.

Table 1. Chloropicrin and inorganic chloride (mg/100 g) in aerobic sandy loam soil treated with chloropicrin at 11.35 mg/100 g and incubated in the dark at 25°C.[ab]

Sampling interval (hours)	Chloropicrin	Inorganic chloride
0	11.35	--
1	5.51	0.95
2	5.44	2.11
4	4.23	2.82
8	3.02	4.04
12	2.63	3.65
16	2.01	5.87
24	1.34	7.30

[a] Average of duplicate samples.

[b] Five degradates were isolated from the soil, but were neither identified nor quantified.

CASE GS -- CHLOROPICRIN STUDY 4 PM --

CHEM 081501 Chloropicrin

BRANCH EAB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID None CONTENT CAT 01

Craine, E.M. 1985. An anaerobic soil metabolism study with chloropicrin. Research Report, Analytical 85:13. Project: WIL-48005. WIL Research Laboratories Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

SUBST. CLASS = S.

DIRECT RVW TIME = 5 (MH) START-DATE END DATE

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CONCLUSIONS:

Metabolism - Anaerobic Soil

1. This study is scientifically valid.

2. Chloropicrin (test substance uncharacterized), at 9.96 mg/100 g of soil, dissipated with a half-life of 8-24 hours in anaerobic sandy loam soil incubated in the dark at 25°C. Inorganic chloride increased by 3.79 mg/100 g of soil during the 48-hour study. Five degradates were isolated but not identified or quantified.

3. This study does not fulfill EPA Data Requirements for Registering Pesticides because the soil was not completely characterized, the degradates were not identified or quantified, the test substance was uncharacterized, and the material balance was incomplete.

MATERIALS AND METHODS:

Samples (100 g) of Wooster sandy loam soil (59% sand, 34% silt, 7% clay, 7.2% organic matter) were treated with 9.96 mg of chloropicrin (test substance uncharacterized). The flasks were immediately flooded with water, sealed, and incubated in the dark at 25°C. Samples were taken at 1, 2, 4, 8, 24, and 48 hours.

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The soil samples were distilled and analyzed by GC as described in Study 3.

REPORTED RESULTS:

Chloropicrin dissipated with a half-life of 8-27 hours (Table 1). Five degradates were detected but not quantified or identified.

DISCUSSION:

1. Complete soil characteristics, such as pH and CEC, were not reported. In addition, the test soil, reported to be a silt loam, is a sandy loam according to the USDA Soil Textural Classification System.

2. The test substance was uncharacterized.

3. Five degradates were detected, but not quantified or identified.

4. The material balance was incomplete.

5. Since the concentration of chloropicrin in the headspace was not measured and the material balance was incomplete, the mechanism of chloropicrin dissipation is uncertain.

Table 1. Chloropicrin and inorganic chloride (mg/100 g soil) in an anaerobic sandy loam soil treated with chloropicrin at 9.69 mg/100 g and incubated in the dark at 25°C.[a]

Sampling interval (hours)	Chloropicrin	Inorganic chloride
0	8.84	0.28
1	9.90	1.40
2	6.04	1.61
4	5.27	1.90
8	4.69	2.34
24	3.20	3.37
48	2.57	3.79

[a] Five degradates were isolated from the soil, but were neither identified nor quantified.

CASE GS -- CHLOROPICRIN STUDY 5 PM --

CHEM 081501 Chloropicrin

BRANCH EAB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID None CONTENT CAT 01

Craine, E.M. 1985b. An adsorption study with soil and chloropicrin. Research Report, Analytical 85:14. Project: WIL-48002. WIL Research Laboratories Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

SUBST. CLASS = S.

DIRECT RVW TIME = 5 (MH) START-DATE END DATE

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SIGNATURE: DATE:

CONCLUSION:

Mobility - Leaching and Adsorption/Desorption

This study is scientifically invalid because the experimental method was inadequate to demonstrate the mobility of the test substance in soil. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because the test substance was not characterized, complete soil characteristics were not provided, K_d values were not reported, the study was not conducted in a 0.01 N calcium ion solution, the temperature at which the study was conducted was not specified, and desorption of the pesticide was not studied.

MATERIALS AND METHODS:

Chloropicrin (test substance uncharacterized) was added to 50-g samples of one sand and three sandy loam soils at 446, 1218, 2255, 4440, or 6335 µg/50 g of soil and mixed throughly for 1 hour (Table 1). Then, 200 ml of water were added to each sample (4:1 slurry) and the slurries were agitated for 1 hour. Control samples, which contained water and chloropicrin but no soil, were studied simultaneously. The samples were centrifuged and the supernatant was decanted, mixed with sodium chloride, and extracted with isooctane. The isooctane extract was analyzed for chloropicrin and its degradates using GC with ^{63}Ni electron-capture detection.

The centrifuged soil was extracted with isooctane by distillation; the extract was analyzed using GC with ^{63}Ni electron-capture detection. Recovery of chloropicrin from fortified soil samples ranged from 93 to 111%.

REPORTED RESULTS:

Chloropicrin in the soil phase of soil:water systems ranged from 2.8 to 16.2% of the recovered for the four soils tested (Table 2).

DISCUSSION:

1. Chloropicrin is a fumigant which is highly volatile and which has a short half-life in soil; it is impossible to determine whether losses of chloropicrin were due to volatility, degradation, or poor methods. The registrant should provide a material balance with the mobility study.

2. The test substance was not characterized.

3. Complete soil characteristics, such as CEC and pH, were not provided.

4. K_d values were not reported.

5. The study was not conducted in a 0.01 N calcium ion solution.

6. The temperature at which the study was conducted was not specified.

7. Desorption of the pesticide was not studied.

8. The soils (except the agricultural sand) were reported to be silt loam soils. According to the USDA Soil Textural Classification system, they are sandy loam soils.

9. No evidence was provided to indicate that chloropicrin would reach equilibrium in the soil slurry in only 1 hour.

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Table 1. Soil characteristics.

Soil type	Sand	Silt	Clay	Organic matter
	%			
Agricultural sand	100	0	0	0.3
Canfield sandy loam	56	36	8	5.5
Wooster sandy loam	59	34	9	7.2
Holly sandy loam	69	28	3	7.4

Table 2. Chloropicrin (µg/bottle) detected in the water and soil phases of four soil:water systems treated with chloropicrin at 446-6335 µg and equilibrated for 1 hour.

Initial concentration	Water	Soil	Total	Adsorbed (% of recovered)
		Canfield sandy loam		
446	100	4.1	104.1	3.9
1218	392.6	28.8	421.4	6.8
2255	729.3	73.3	802.6	9.1
4440	1585.4	165.8	1751.2	9.5
6335	2087	265	2352	11.3
		Holly sandy loam		
446	12.7	0.5	13.2	3.8
1218	209.9	10.8	220.7	4.9
2255	684.2	77.9	762.1	10.2
4440	1557.8	257.2	1815.0	14.2
6335	2121	409	2530	16.2
		Wooster sandy loam		
446	6.9	0.2	7.1	2.8
1218	153.7	6.7	160.4	4.2
2255	541.6	47.1	588.7	8.0
4440	1262.7	153.8	1416.5	10.9
6335	1715	241	1956	12.3
		Agricultural sand		
446	164.5	10.2	174.7	5.8
1218	384.4	31.9	416.3	7.7
2255	693.8	44.9	738.7	6.1
4440	1412.4	105.2	1517.6	6.9
6335	1849	125	1974	6.3
		Control[a]		
446	221.7	--	221.7	--
1218	909.4	--	909.4	--
2255	1600.9	--	1600.9	--
4440	2505.8	--	2505.8	--
6335	4663	--	4663	--

[a] Controls contained no soil.

EXECUTIVE SUMMARY

The data summarized here are scientifically valid data that have been reviewed in this report but do not fulfill data requirements unless noted in the Recommendations section of this report.

Chloropicrin (test substance uncharacterized), at 9.96 mg/100 g of soil, dissipated with a half-life of 8-24 hours in anaerobic sandy loam soil incubated in the dark at 25°C. Inorganic chloride increased by 3.79 mg/100 g of soil during the 48-hour study. Five degradates were isolated but not identified or quantified.

Chloropicrin (test substance uncharacterized), at 11.35 mg/100 g of soil, dissipated with a half-life of <1 hr in sandy loam soil incubated in the dark under aerobic conditions at 25°C. Inorganic chloride increased by 7.30 mg/100 g of soil during the 24-hour study. Five additional degradates were isolated but neither quantified nor identified.

RECOMMENDATIONS

Available data are insufficient to fully assess the environmental fate of, and the exposure of humans and nontarget organisms to chloropicrin. The submission of data relevant to reregistration requirements (Subdivision N) for terrestrial and aquatic food crop, terrestrial nonfood crop, greenhouse, and domestic outdoor use sites is summarized below:

Hydrolysis studies: One study (Craine, 1985a, Acc. No. 260211) was reviewed for this addendum. This study is scientifically invalid because the data were too variable to assess the decline of chloropicrin in water. In addition, this study would not fulfill data requirements because the test substance was not characterized and the material balance was incomplete. All data are required.

Photodegradation studies in water: One study (Castro and Belser, 1981, Acc. No. 260211) was reviewed for this addendum. This study cannot be validated because the procedures were insufficiently described and the reported results were not supported with quantitative data. In addition, this study would not fulfill data requirements because the test substance was uncharacterized, the test solutions were incompletely characterized, sunlight and the artificial light were incompletely characterized, degradates were not identified, and there was no material balance. All data are required.

Photodegradation studies on soil: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, the data requirement is waived for soil injection use.

Photodegradation studies in air: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Aerobic soil metabolism studies: One study (Craine, 1985c, Acc. No. 260211) was reviewed for this addendum. This study does not fulfill data requirements because the soil was not completely characterized, the degradates were not identified or quantified, the test substance was uncharacterized, and the material balance was incomplete. All data are required.

Anaerobic aquatic metabolism studies: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Aerobic aquatic metabolism studies: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Leaching and adsorption/desorption studies: One study (Craine, 1985b, Acc. No. 260211) was reviewed for this addendum. This study is scientifically invalid because the experimental method was inadequate to demonstrate the mobility of the test substgance in soil. In addition, this study would not fulfill data requirements because the test substance was not characterized, complete soil characteristics were not provided, K_d values were not reported, the study was not conducted in a 0.01 N calcium ion solution, the temperature at which the study was conducted was not specified, and desorption of the pesticides was not studied. An acceptable adsorption/desorption study is required.

Laboratory volatility studies: No data were submitted for this addendum, but all data are required.

Field volatility studies: No data were submitted for this addendum. The data requirement is deferred pending the receipt of acceptable laboratory volatility data.

Terrestrial field dissipation studies: No data were submitted for this addendum. The data requirement is deferred pending the receipt of acceptable hydrolysis and soil metabolism studies.

Aquatic field dissipation studies: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Forestry dissipation studies: No data were submitted for this addendum, but no data are required because chloropicrin has no registered forestry use.

Dissipation studies for combination products and tank mix uses: No data were submitted for this addendum. Data requirements for combination products and tank mix uses are currently not being imposed for this Standard.

Long term field dissipation studies: No data were submitted for this addendum. The data requirement has been deferred pending the receipt of acceptable soil metabolism and (if required) field dissipation studies.

Confined accumulation studies on rotational crops: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Field accumulation studies on rotational crops: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Accumulation studies on irrigated crops: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, no additional data are required.

Laboratory studies of pesticide accumulation in fish: No data were submitted for this addendum. Based on the Chloropicrin Registration Standard, data will not be required if the registrant can provide acceptable evidence that chloropicrin will not reach water, will not persist in water, and/or has properties that suggest it will not accumulate in fish, mammals, and birds.

Field accumulation studies on aquatic nontarget organisms: No data were submitted for this addendum. The data requirement has been deferred pending the receipt of acceptable soil metabolism and mobility data.

Reentry studies: No data were submitted for this addendum, but all data are required.

REFERENCES

Castro, C.E. and N.O. Belser. 1981. Photohydrolysis of methyl bromide and chloropicrin. J. Agric. Food Chem. 29(5):1005-1008. Acc. No. 260211.

Craine, E.M. 1985a. A hydrolysis study with chloropicrin. Research Report, Analytical 85:6. Project: WIL-48003. WIL Research Laboratories, Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

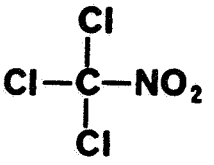
Craine, E.M. 1985b. An adsorption study with soil and chloropicrin. Research Report, Analytical 85:14. Project: WIL-48002. WIL Research Laboratories Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

Craine, E.M. 1985c. An aerobic soil metabolism study with chloropicrin. Research Report, Analytical 85:12. Project: WIL-48004. WIL Research Laboratories Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

Craine, E.M. 1985d. An anaerobic soil metabolism study with chloropicrin. Research Report, Analytical 85:13. Project: WIL-48005. WIL Research Laboratories Inc., Great Lakes Chemical Corp., Ashland, OH. Acc. No. 260211.

APPENDIX - STRUCTURE

ACQUINITE, CHLOR-O-PIC, DOJYOPICRIN,
DOLOCHLOR, LARVACIDE, PIC-CHLOR,
PICFUME, PROFUME A, TRI-CLOR



Trichloronitromethane