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ENVIRONMENTAL FACTORS AND THE ACTIVITY OF ETHOFUMESATE (2-ethoxy-2,3-dihydro-3,3-dimethyl-5benzofuranyl methanesulphonate) ON SUBSEQUENT CROPPING SYSTEMS presented by

Dale Alan Aaberg

has been accepted towards fulfillment of the requirements for

PhD degree in Crop and Soil Sciences

<u>V Clian Major professor</u>

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ENVIRONMENTAL FACTORS AND THE ACTIVITY OF ETHOFUMESATE (2-ethoxy-2,3-dihydro-3,3-dimethyl-5benzofuranyl methanesulphonate) ON SUBSEQUENT CROPPING SYSTEMS

Ву

Dale Alan Aaberg

A DISSERTATION

Submitted to Michigan State University in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Department of Crop and Soil Sciences

ABSTRACT

ENVIRONMENTAL FACTORS AND THE ACTIVITY OF ETHOFUMESATE (2-ethoxy-2,3-dihydro-3,3-dimethyl-5benzofuranyl methanesulphonate) ON SUBSEQUENT CROPPING SYSTEMS

By

Dale Alan Aaberg

The residues of ethofumesate (2-ethoxy-2,3-dihydro-3, 3-dimethyl-5-benzofuranyl methanesulphonate), a herbicide presently used in sugarbeets, in soil 8 to 12 months after application at 0.56 to 10.01 kg/ha were evaluated under field conditions with oat (Avena sativa, L.), alfalfa (Medicago sativa, L.), soybean (Glycine max, (L.) Merr.), and cucumber (Cucumis sativus L.) as bioassay crops. The general order of crop sensitivity to ethofumesate was cucumber > soybean > alfalfa > oat; however, there were differences among location and year. Visual injury to bioassay plants indicated that after 8 months 75 percent and after 12 months 90 percent of the ethofumesate had been dissipated. Broadcast application treatments of ethofumesate resulted in greater bioassay crop injury and yield reductions than equivalent band applications. Plowing the treated areas substantially reduced plant injury from ethofumesate by a dilution effect compared to discing which in turn was more effective than use of a field cultivator. Several studies were conducted in the greenhouse to evaluate bioassay crop parameters in response to ethofumesate under various soil conditions. The leaching of ethofumesate from a 25.4 cm surface irrigation volume increased with increasing application rate. However, greatest bioassay crop response came from residue levels at the soil surface. Degradation of ethofumesate increased if the soil was not sterilized and maintained at 32.2 C versus lower temperatures. Visual injury to soybean and wheat was reduced on fine textured soils or if the percent organic matter was 11.5 percent or higher.

Residues of ethofumesate in the soil injured wheat (<u>Triticum aestivum</u>, L.), more than oat, cucumber, and soybean which were injured more than dry edible beans (<u>Phaseolus</u> <u>vulgaris</u> L.). However, cultivar response to ethofumesate varied within crop species. Corn (<u>Zea mays</u>, L.subsp. mays) was not visually injured by ethofumesate at rates to 2.24 kg/ha. Plant site of ethofumesate uptake varied with test crop evaluated. Ethofumesate appeared to act very early during plant growth.

Extracted ethofumesate soil residue levels varied considerably among experimental plots, locations and years. Ethofumesate levels of 0.40 ppm or greater caused significant visual injury when related to ratings from field bioassay experiments. ¹⁴C ring-labeled ethofumesate degradation was decreased under sterile versus non-sterile conditions suggesting microbial decomposition. However, under either condition,

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¹⁴C- ethofumesate was reduced if organic matter was 6.9 percent or greater.

Adsorption of ¹⁴C-ethofumesate on soil occurred within 0.25 h if the soil was agitated. High levels of ¹⁴Cethofumesate adsorption were measured on several Michigan soils. ¹⁴C-ethofumesate adsorption increased with increase in the number of active sites. To my beautiful and loving wife, Karen, for her endless support and patience.

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The author wishes to express his sincere appreciation to his major professor, Dr. William F. Meggitt, for his guidance and more importantly friendship during this course of study. The experience gained from association and involvement with the weed control program at Michigan State University is considered invaluable.

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INTRODUCTION

Todays trend in agricultural production continues to be towards total farm mechanization. Hand in hand with this trend is the common practice of using chemical means for weed control. Sugarbeets are no exception with approximately 98 percent of the Michigan production area receiving a herbicide treatment (53,54). Of the over 36000 ha, almost all receive surface applied herbicides. Furthermore, 12 percent also receive soil incorporated herbicides, and 50 percent of the sugarbeets also receive a foliar herbicide application (53,54). Effective chemical weed control in sugarbeets requires an intense and specific program (50,52).

Ethofumesate (Figure 1) is a registered herbicide¹ that may be either soil or foliar applied for the selective control of several annual grass and broadleaf species in sugarbeets (15,18,19,22,23,50,57,60,73). Researchers (23,39,73) have reported that control of susceptible weed species with ethofumesate may extend for 10 to 12 weeks after soil application. According to Dawson (14), a good stand of sugarbeets will produce a closed canopy after that period to minimize new seedling growth.

¹Registered as Nortron[®] by Fisons Incorporated, 1978.





Ethofumesate (2-ethoxy-2,3-dihydro-3,3-dimethyl-5-benzofuranyl methanesulphonate)

.4

Classified in the 'Miscellaneous' group of chemical herbicide compounds.

However, as documented with other residual herbicides (4,8,11,13,26,28,44,48,49,56,61), extended control may result under certain conditions which in turn may cause adverse effects to susceptable rotational crop species. General problems may be two-fold. Injury may result from ethofumesate carry-over to crops sown the following growing season or complications may result from replanting of abandoned sugarbeet fields the same season.

Spring sown cereals such as wheat and barley are sensitive to ethofumesate (63,65,66,84) and band applied ethofumesate may cause a stand reduction to wheat (65,66). Schweizer reported that pinto beans were more tolerant to ethofumesate residues than corn if replanting followed ethofumesate treated sugarbeets within the same growing season. However, Kampe (41) and Schweizer (64) observed no injury or yield reduction, respectively to corn planted the growing season following ethofumesate application.

Application technique and tillage practice appear to have an effect on reducing ethofumesate carry-over (64,65,66). Microbial activity accounts for much of the ethofumesate degradation (23,39,65,83), thus is greater under warm, moist soil conditions (39,65).

In Michigan, winter wheat does not generally succeed sugarbeets in the rotation. However, casual observation in grower fields has indicated that ethofumesate applied to sugarbeets caused visual symptoms to soybeans the following year. Several management factors may contribute i.e. recent

label expansion allowing ethofumesate to be applied as a foliar treatment, and trends toward less energy utilizing tillage operations. These coupled with the preemergence ethofumesate level and later season ethofumesate application, increase the potential for residual carry-over. Several soil properties and climatic factors have been related to the performance of other herbicides i.e. soil pH, soil organic matter percent, molecular structure, moisture (2-9,11-13, 16-17,20,24-25,29,31-36,39,44-49,56,58,62,70,77-79,81).

The purpose of this study was to: (1) determine the influence of soil environmental factors on residual carryover; (2) determine the effect of soil type, pH and organic matter content on ethofumesate carry-over; (3) determine the influence of tillage practices and application techniques on reducing rotational crop response; and (4) evaluate crop cultivar sensitivity to ethofumesate.

FIELD STUDIES

Materials and Methods

The residue levels of ethofumesate emulsifiable concentrate were experimentally determined in four field studies in Michigan between 1979 and 1981. All broadcast applications of ethofumesate were applied with a tractor mounted sprayer delivering 215 L/ha at 2.1 kg/cm² pressure and band applied with the same application equipment at 121 L/ha. Soil analyses and annual precipitation are listed in Appendices 1 and 2, respectively. All soil chemical and mechanical soil analysis were conducted by the Michigan State University Soil Testing Laboratory, East Lansing.

In the first field study initiated in 1978 at East Lansing, ethofumesate was applied broadcast preemergence to sugarbeet alone and in herbicide combinations (Appendix 3). In 1979 ethofumesate residues on this site were determined by a split-split plot experiment with four replications. The main plot was represented by various bioassay crops. Sub-plots were obtained by dividing the established 12.2 m plot into plowed and disced spring tillage portions. Sub-sub plots were treated the previous season with ethofumesate alone and/or in combination treatments. The

entire experimental site was tilled with a field cultivator and two 71.7 cm rows were planted per bioassay crop per initial cultivation technique. Oat cv. 'Russell' was sown May 7, 1979 at a depth of 5.1 cm and rate of one kernel per 2.5 cm and alfalfa cv. 'Vernal' at 0.6 cm depth and a 1.2 cm seed spacing, each with a modified press-wheel grain drill. Alfalfa rows then received a single pass with a rolling coulter. Soybeans cv. 'Hark' and cucumber cv. 'Marketmore-70' were both sown May 23, 1979 2.5 cm below the soil surface at a rate of one seed per 5.1 cm and 25.4 cm, respectively utilizing a pair of tool-bar mounted 'Planted Jr.' plate planters. Rows were hand weeded throughout the growing season. Cucumber plants were dusted with 'Rotenone' (Cube') insecticide to control cucumber beetles July 3, 1979. Parameters evaluated 6 weeks after planting included visual injury ratings consisting of leaf crinkling, leaf fusion, and plant stunting on a percentage basis with 0 equal to no injury and 100 equal to complete kill, and plant population counts for cucumber and soybeans. Oat plots were hand cut at the soil surface August 17-18, bagged, dried, and weighed prior to seed removal with a stationary thrasher. The alfalfa in all the plots was at full bloom. After harvest the alfalfa was dried and weighed. Soybean yield parameter was obtained with a small plot combine at full maturity. Cucumber vine and fruit weights were measured at the onset of yellowing of the fruit of untreated control plants. Oat and soybean seed germination were tested 3 months after harvest by a blotter method (1).

Two additional field bioassays were conducted in 1980. 'Location 1', at East Lansing, was similar to the initial bioassay study but, with additional parameters. Ethofumesate was banded in 35.6 cm strips on 101.6 cm spray boom spacings and broadcast applied alone and in combination with pyrazon (5-amino-4-chloro-2-phenyl-3(2H)-puridazinone) at 4.48 kg/ha and TCA (trichloroacetic acid) at 6.72 kg/ha May 16, 1979 and a simulated post application June 19, 1979. Broadcast applications of glyphosate (isopropylamine salt of N-(phosphonomethyl)glycine) at 2.24 kg/ha were applied July 10, 1979 and September 5, 1979 to control weed growth.

The 16.2 m plots were evenly sub-divided and underwent the following course of events May 23, 1980: sub-plot 1 was plowed then disced, sub-plot 2 was disced, sub-plot 3 was field cultivated. The entire field was then finished with a 'Triple-K' s-tine cultivator with a rolling basket rear attachment prior to planting two 6.2 cm spaced rows of each bioassay crop per tillage segment May 25, 1980. Oat cv. 'Russell' was sown 5.1 cm deep at a rate of one kernel per 1.9 cm using a modified press-wheel grain drill. Soybean cv. 'Hark' and cucumber cv. 'Marketmore-70' were also seeded 5.1 cm deep at a rate of one seed per 5.1 cm and 20.0 cm, respectively, sown with a pair of tool-bar mounted 'Planet Jr.' plate planters. The site was hand weeded throughout the season. Parameters evaluated of the replicated split-split plot were percent visual injury and plant population.

The Bay County site, 'Location 2', was initiated to simulate residual ethofumesate carry-over levels under field conditions. Ethofumesate was applied broadcast and in 25.4 cm bands over 76.2 cm nozzle spacing May 16, 1980 at 0.14, 0.28, 0.56, 0.84, and 1.12 kg/ha. Again, a replicated split-split plot design was established with oat cv. 'Russell', soybean cv. 'Hark', and cucumber cv. 'Marketmore-70' being the main effect, plowing and field cultivation comprising the subeffect, and ethofumesate rates representing the sub-sub plots. Bioassay crop density and depth were similar to Location 1. However, crop row pairs were in 71.1 cm spacings for each cultivation technique and soybeans and cucumbers were manually sown with a single 'Planet Jr.' plate planter. All plots were kept weed-free throughout the season by hoeing. Evaluations included percent visual injury of oat and cucumber and population counts of cucumber 42 days after planting. Oat plant populations were determined by summation of the number of panicles per plot 21 days prior to the August 18 seed harvest with a small plot combine. Remaining foliage was collected, dried, and weighed. Cucumber vine and fruit harvest was 90 days after planting which coincided with the initial control plot fruit turning yellow. Soybeans were devastated by rodents at emergence, therefore no data was obtained.

The final field bioassay was conducted in 1981 to evaluate the potential interaction between soil pH and ethofumesate residual effects. An experimental site that had previously been adjusted with lime to give a range of soil pH from 4.3

to 7.0 in 4.3 m by 7.6 m plots was utilized. Ethofumesate at 0.56, 1.12, 2.24, and 4.48 kg/ha was broadcast applied to the soil and in 30.5 cm bands with 76.2 cm nozzle spacing across the pH adjusted plots October 7, 1980. Another portion of the field was broadcast and banded similarly with ethofumesate rates of 0.14, 0.28, 0.56, and 1.12 kg/ha May 22, 1981. Herbicide plots were divided May 22, 1981 with one-half plowed and the other portion field cultivated. The entire field was tilled with a 'Triple-K' s-tine cultivator with a rolling basket rear attachment and solid seeded to soybeans cv. 'Harcor' in 25.4 cm rows the same day. Bentazon (3isopropyl-lH-2,l,3-benzothiadiazin-4(3H)-one 2,2-dioxide) plus sulfidide (2-1-(ethoxyimino)-buty1-5-2-(ethy1thio)propyl-3-hydroxy-2-cyclohexene-l-one) was applied as a postemergence tank-mix June 19, 1981 at 1.12 plus 0.56 kg/ha, respectively for weed control. Percent visual injury ratings were obtained 33 days after planting.

Results and Discussion

Visual leaf injury sysmptoms caused by ethofumesate were most obvious at the beginning of the season and became masked with increased plant foliage. Reductions in plant population resulted from death of emerged plants due to ethofumesate injury rather than germination inhibition. Ethofumesate in herbicide combination showed no significant difference in detectable residues, therefore data presented are combined summaries for each of the bioassay crops.

Previous broadcast ethofumesate application of 10.08 kg/ha caused visual oat stunting when the initial spring tillage was either plowing or discing (Figure 2). Visual injury observed 6 weeks after planting of 22 and 33 percent for the plowed and disced portions, respectively, was also reflected in the seed harvest at East Lansing (Figure 2). Visual injury and seed yield showed a direct relationship to ethofumesate levels under both plowing and discing conditions (Figure 3). Panicles per meter of row and dry weights were reduced only at the 10.08 kg/ha ethofumesate level if the initial tillage technique was discing (Appendix 4). Ethofumesate application of 6.72 and 10.08 kg/ha caused visual injury to alfalfa and reduced harvest dry weight regardless of which tillage practice was used (Figure 4). A direct relationship was also noted between visual response and yield to ethofumesate level as in oat (Figure 5). However, a reduction in severity of each of these parameters was observed if the treatment areas was plowed prior to planting.

Observable injury to soybean foliage resulted from 3.36, 6.72, and 10.08 kg/ha ethofumesate applied to the test site in 1978 and disced prior to planting the following spring. If spring plowed, plant injury from the 3.36 kg/ha ethofumesate rate was insignificant (Figure 6). Necrosis and crinkling of soybean leaves increased 50 and 26 percent at 6.72 kg/ha and 67.1 and 43 percent at 10.01 kg/ha ethofumesate, respectively if the initial tillage was disced rather than plowed. Ethofumesate levels evaluated caused no soybean plant population Figure 2. Effect of ethofumesate residue level on oat visual and seed yield under different cultivation techniques. 1979 field bioassay, East Lansing, MI.





Figure 3. Relationship between oat visual injury and yield as affected by ethofumesate soil residue levels. 1979 field bioassay, East Lansing, MI.



Figure 4. Effect of ethofumesate residue level on alfalfa visual injury and dry matter yield under different cultivation techniques. 1979 field bioassay, East Lansing, MI.





Figure 5. Relationship between alfalfa visual injury yield as affected by ethofumesate soil residue levels. 1979 field bioassay, East Lansing, MI.



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Figure 6. Effect of ethofumesate residue level on soybean visual injury and seed yield under different cultivation techniques. 1979 field bioassay, East Lansing, MI.




Figure 7. Relationship between soybean visual injury and yield as affected by ethofumesate soil residue levels. 1979 field bioassay, East Lansing, MI.



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Figure 7. Relationship between soybean visual injury and yield as affected by ethofumesate soil residue levels. 1979 field bioassay, East Lansing, MI.



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reduction when compared to the untreated control plot (Appendix 5). Soybean seed yield was reduced only at an ethofumesate rate of 10.08 kg/ha (Figure 6) indicating the soybean plants compensate for a large portion of the observed foliage injury. Also visual injury correlated with yield existed only under plowed conditions (Figure 7). Germination of oat and soybean seed produced by injured plants was not affected by ethofumesate residue levels. Cucumber yield production parameters were increased if the plot area was plowed rather than disced prior to planting. Residual effects from ethofumesate as low as 3.36 kg/ha caused significant visual injury to cucumber and fruit yield reduction if the area was disced prior to planting (Figure 8). Visual injury from ethofumesate at 10.01 kg/ha reached a level of 75 and 60 percent if disced and plowed, respectively (Figure 8). A direct response of visual injury and yield to ethofumesate was evident if plowed or disced prior to planting (Figure 9). Ethofumesate levels did not affect cucumber plant density (Appendix 6).

Visual injury symptoms from ethofumesate were not observed on any of the bioassay tests crops at East Lansing in 1980. Annual precipitation was similar for the locationyears, however, pH varied from 5.2 to 7.3 for the 1978 and 1979 sites, respectively (Appendix 2). The pH variation did not appear to be totally responsible for the lack of response but warranted evaluation.

The effect of ethofumesate on oat and cucumber was reduced by plowing versus field cultivation primarily as a result of dilution (Figure 10-15; Appendices 7-10). Broadcast and band applications of 0.56 kg/ha ethofumesate or greater prior to field cultivation and bioassaying caused stunting of oats. Visual injury increased from 19 to 20 percent to 60 and 48 percent with broadcast and band ethofumesate application of 0.84 and 1.12 kg/ha, respectively (Figure 10). Ethofumesate broadcast applied at 0.84 and 1.12 kg/ha followed by field cultivation reduced oat panicles per meter of row, seed and straw yields (Appendices 7-8). Plowing following the same ethofumesate rates eliminated detrimental effects on these same parameters (Figure 10-12; Appendices 7-8). Band applied ethofumesate reduced only seed yield which was directly correlated with visual injury (Figure 12).

Cucumber as a bioassay test crop responded similarly to oat in parameters measured but with increased observable foliage injury symptoms. Broadcast applications of ethofumesate increased the level per unit area thus enhancing visually observed injury at lower rates than equivalent band application (Figure 13). Similarly injury from ethofumesate increased if the ethofumesate treated area was field cultivated rather than plowed prior to planting.

Ethofumesate applied in the fall or spring to soil with a pH range of 4.3 to 7.0 showed no observable differences in injury to soybean when compared to equivalent ethofumesate

Figure 8. Effect of ethofumesate residue level on cucumber visual injury and fruit yield under different cultivation techniques. 1979 field bioassay, East Lansing, MI.





Figure 9. Relationship between cucumber visual injury and yield as affected by ethofumesate soil residue levels. 1979 field bioassay, East Lansing, MI.



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Figure 10. Oat visual response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., Mi.



Figure 11. Oat yield response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., Mi.





Figure 12. Relationship between oat visual injury and yield as affected by broadcast and band applications of ethofumesate. 1980 field bioassay, Bay Co., MI.



VISUAL INJURY (%)



Figure 13. Cucumber visual response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., MI.







Figure 14. Cucumber fruit yield response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co. MI.



Figure 15. Relationship between cucumber visual injury and yield as affected by broadcast and band applications of ethofumasate. 1980 field bioassay, Bay Co., MI.



rates on soils with a neutral pH. As previously observed, injury to soybeans was dependent on ethofumesate levels in the soil (Figure 16-17). Field cultivated portions showed significantly more ethofumesate injury to soybeans than plowed areas. A measureable increase in visual injury to soybean was detected when ethofumesate was broadcast applied at 0.56 kg/ha compared to a band application of 1.12 kg/ha in the fall.

Soybean response to ethofumesate broadcast or band applied in the spring increased with ethofumesate levels. Visual injury to soybeans from ethofumesate broadcast applied at 1.12 kg/ha was significantly greater if the area was field cultivated rather than plowed. Based on percent visual injury to soybeans, residue from fall application of ethofumesate were equivalent to approximately one-fourth that of residues from spring application of ethofumesate. Figure 16. Soybean visual response to broadcast and band fall applied ethofumesate treatments under different cultivation techniques. 1981 field bioassay, East Lansing, MI.





Figure 17. Soybean visual response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1981 field bioassay, East Lansing, MI.





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GREENHOUSE STUDIES

Materials and Methods

Experiments were conducted in the greenhouse to evaluate ethofumesate rate simulated residual response to potential rotational crops following sugarbeets. Other parameters used to evaluate ethofumesate levels under controlled environmental conditions included crop varietal response to ethofumesate soil levels, interact: n between soil types and ethofumesate level in response to crop injury, ethofumesate leaching, crop uptake site determination, ethofumesate response to pH, and ethofumesate degradation over time studies. In all studies, ethofumesate emulsifiable concentrate was applied utilizing an experimental spray chamber delivering a volume of 950 L/h at a pressure of 2.1 kg/cm^2 with an 80 degree nozzle. Each study was repeated except the ethofumesate degradation study over time. Supplemental greenhouse lighting was either flourescent tubes delivering $70\mu \text{Em}^{-2} \text{s}^{-1}$ or metal halide lamps delivering $240_{\mu} \text{Em}^{-2} \text{s}^{-1}$ to provide a 16 h day photoperiod.

Ethofumesate residual level study. Aluminum flats were volumetrically filled with greenhouse potting soil (organic matter approximately 8 percent) to a level of

7.62 cm. Ethofumesate equivalent to 0, 0.14, 0.28, 0.56, 0.84, 1.12, and 2.24 kg/ha was applied to the soil surface. Following ethofumesate application, the soil was thoroughly mixed in a mechanical rotational soil mixer. Soft white winter wheat cv. 'Tecumseh', oat cv, 'Korwood', cucumber cv. 'Marketmore-70', dry edible bean cv. 'Seafarer' and soybean cv. 'Harcor' were planted 2.54 cm below the soil surface in 473-ml wax containers that had been volumetrically filled with ethofumesate treated soil. All bioassayed crops were planted at a rate of four seeds per container except wheat and oat which were planted ten seeds per container. The experiment was arranged as a split-plot with main effects being crops and sub-effects represented by ethofumesate rate. All treatments were surface irrigated and supplemented with flourescent lighting in the greenhouse. Visual injury to plants and fresh weight were determined 19 days after planting.

<u>Crop varietal response study</u>. Ethofumesate was applied to greenhouse potting soil at rates of 0, 0.56, and 1.12 kg/ha and incorporated as described in the residual study. Ethofumesate treated soil was volumetrically divided into 473-ml wax containers and sown with common crop cultivars grown in Michigan (Table 1). Soft white and red winter wheat and oat were sown at a rate of ten kernels per container while cucumber, dry edible beans, soybean, and corn were planted four per container. All test crops were planted at a depth of 2.54 cm. Varieties

of each of the crops tested are listed in Table 1. Design was a split-split plot with main plot equal to crop, subplot represented by ethofumesate rate and sub-sub plot comprised of the crop varieties. Treatment containers were surface irrigated and maintained in the greenhouse with supplemental flourescent lighting. Visual injury to plant and shoot fresh weight were ascertained 19 days after planting.

Soil studies. Soils evaluated were natural unaltered soil types collected from various sugarbeet growing areas in Michigan (Appendix 1). Soils were divided into groups for experimentation based on clay content and organic matter. Ethofumesate at 0.56 kg/ha was applied to each soil type in 7.62 cm aluminum flats. Treated soil was thoroughly mixed in a mechanical rotational soil mixer and equally divided into 473-ml wax containers. Bioassay test indicators of ethofumesate level were soft white winter wheat cv. 'Tecumseh' and soybean cv. 'Harcor'. Both crops were planted at a depth of 2.54 cm and rate of ten seeds and four seeds for oat and soybean, respectively. Treatments were surface irrigated and provided supplemental lighting with metal halide lamps. Arrangement in the greenhouse was a split-plot design comprised of ethofumesate rate as the main effect and soil type as sub-effect. Plant visual injury and fresh weights were obtained 20 days after planting.

Soft white winter wheat cvs.	Augusta Genesee Tecumseh Yorkstar
Soft red winter wheat cvs.	Abe Arthur
Oat cvs.	Astro Ausable Korwood MacKinaw Mariner Russell
Cucumber cvs.	Green Star Marketmore-70
Dry edible bean cvs.	Sanilac Seafarer Tuscola
Soybean cvs.	Beeson Corsoy Harcor Hark Hodgson
Corn cvs.	Great Lakes Hybrid MI 4122 Pioneer 3901

Table 1. Bioassay crop cultivars evaluated for response to ethofumesate soil levels in greenhouse studies.

Soil leaching study. Polyvinyl chloride pipe was cut into 61 cm sections and capped at one end. Greenhouse soil mix (organic matter approximately 9 percent) was added to each 18 cm diameter container to 2.54 cm fullness. Ethofumesate at 0, 0.56, 1.12, 2.24, 4.48, and 8.96 kg/ha was applied to an aluminum flat containing 1.27 cm of greenhouse soil. Treated soil was mixed in a mechanical rotational soil mixer. Ethofumesate was added to the top of each column in 1.27 cm of soil, rather than applying ethofumesate directly to the soil surface of each column, to reduce potential water channeling along column walls during irrigation. Water was surface irrigated at the rate of 5.08 cm per h for 5 h. The 25.4 cm level was predetermined as the average rainfall level for April through July in Michigan. Columns were covered with aluminum foil and stored in the greenhouse until planting. One week later, columns were split vertically into equal portions. Metal plates were inserted every 25.4 cm to eliminate lateral bioassay crop root movement. Segments were bioassayed with soft white winter wheat cv. 'Tecumseh' and soybean cv. 'Harcor'. Wheat and soybean were planted in rows 2.54 cm off center and at a depth of 2.54 cm. Wheat emergence was reduced by soil compaction, therefore no data obtained.

A concurrent leaching study was conducted in which 946-ml plastic containers were filled with greenhouse soil mix. Ethofumesate at 2.24 kg/ha was applied to the surface of

each container and leached with 25.4 cm of water applied at a rate of 5.08 cm per h. One week later containers were divided into 5 groups in which soil was removed at 0, 2.54, 5.08, 7.62, and 10.16 cm from the surface. Soil removed was discarded and that remaining was bioassayed with soybeans. Planting was four soybean seeds per container at a depth of 2.54 cm.

Randomized complete block design was maintained in the greenhouse under supplemental flourescent lighting and surface irrigation. Visual foliage injury was evaluated 23 days after planting.

Charcoal barrier study. Greenhouse soil mix (organic matter approximately 8 percent) was measured into 7.62 cm aluminum flats. Ethofumesate at 0, 1.12, 2.24, and 4.48 kg/ha was applied to the soil surface and throughly incorporated with a mechanical rotational soil mixer. Activated charcoal was mechanically mixed with untreated greenhouse soil in a ratio of 2:1 for use as a barrier to prevent herbicide movement. Plastic containers with a capacity of 946-ml were prepared to determine ethofumesate root, shoot, or seed uptake as illustrated in Figure 18. Soft white winter wheat cv. 'Tecumseh' and oat cv. 'Astro' were planted in containers with soil ethofumesate levels of 1.12 kg/ha. Soybean cv. 'Harcor' and cucumber cv. 'Marketmore-70' were planted in soil with an ethofumesate levels of 1.12 kg/ha. Soybean cv. 'Harcor' and cucumber

cv. 'Marketmore-70' were planted in soil with an ethofumesate equivalent to 2.24 kg/ha. Sugarbeet cv. 'US H20', dry edible beans cv. 'Seafarer', and corn cv. 'Pioneer 3901' were planted in containers with ethofumesate applied at 4.48 kg/ha. Ethofumesate levels were pre-determined to give observable injury symptoms to the respective crops. Ten seeds of grass species and four seeds of each broadleaf species were planted per container at a depth of 2.54 cm. Arrangement in the greenhouse was a split-plot with ethofumesate rate and charcoal barrier location comprising the main plot and sub-plots, respectively. All containers were surface irrigated and supplementary lighted with metal halide lamps. Percent visual injury and plant fresh weights were obtained for all species 14 days after planting except cucumber which were determined 20 days after planting.

Ethofumesate pH study. Soft white winter wheat cv. 'Tecumseh' and soybean cv. 'Harcor' were planted in trays containing vermiculite. Trays were maintained in the greenhouse under flourescent tube supplemental lighting to give a 16 h photoperiod. Surface water irrigation was applied daily and Hoaglands nutrient solution (38) added bi-weekly. Two hundred ml of 0.5x concentration Hoaglands solution was measured into 296-ml plastic cups previously covered with aluminum foil. Solutions were adjusted with 1 M KOH or 0.5 M H_2SO_4 to give 0.5 pH intervals between 4.5 and 8.0. Ethofumesate was added to the solution to give a final concentration of 3 ppm.
Figure 18. Charcoal barrier technique for determination of ethofumesate uptake by the root, shoot, or seed.









seed uptake determination

- UT -- untreated soil
- T -- ethofumesate treated soil
- B -- charcoal: greenhouse soil mix (2:1)
- o -- seed placement

Wheat at the two leaf stage and soybeans at the first trifoliate growth stage were transferred from vermiculite into the hydroponic solution. Each cup contained one seedling which was held in place using a 2.54 thick foam puck with radial slit. A completely random design arrangement was maintained in a growth chamber with 15.6 C night temperature and 26.6 C day temperature. Photoperiod was 16 h with flourescent tubes and incandescent bulbs delivering 90 $\mu \text{Em}^{-2} \text{s}^{-1}$. Plant visual injury was determined 14 days after the seedling transferal into the hydroponic solution.

A study was conducted in the greenhouse to evaluate the effect of soil liming on bioassay crop response to ethofumesate levels. Hydrated lime was added to greenhouse soil mix and incorporated at a rate equivalent to 1120 kg/ha. Soils were stored in the dark at 25 C and moisture level of 50 percent field capacity. After a 2 week equilibration period, soil was volumetrically measured into 7.62 cm aluminum flats and treated with ethofumesate equivalent to 0, 0.28, 0.56, 0.84, 1.12, and 2.24 kg/ha. Ethofumesate was incorporated with a mechanical rotational soil mixer and divided equally into 473-ml plastic containers. Soybean cv. 'Harcor' was planted at a rate of four seeds per container and depth of 2.54 cm. Visual injury to soybean was observed 20 days after planting.

Ethofumesate degradation study. Greenhouse soil mix, (organic matter approximately 7 percent) was bulk steam sterilized 4 h. Steam sterilized and unsterilized greenhouse soil mix were placed into separate 7.62 cm aluminum flats and treated with ethofumesate equivalent to 2.24 kg/ha. Ethofumesate was incorporated with a mechanical rotational soil mixer and divided into 473-ml plastic containers. Containers were stored in the dark at 15.6 C, 12 h fluctuating temperatures of 23.9 C to 29.4 C, and 32.3 C. The unsterilized containers were kept at 50 percent field capacity by surface irrigating with distilled H₂0, while the steam sterilized containers received no water for the duration of storage. At weekly intervals, containers were bioassayed. Soybeans cv. 'Harcor' was planted at a rate of four per container and depth of 2.54 cm. Containers were placed in a growth chamber with a day temperature of 26.6 C and night temperature of 15.6 C. Lighting from flourescent tubes and incandescent bulbs at 90 $_{\rm u}$ Em⁻²s⁻¹ provided a 16 h photoperiod. Containers were surface irrigated to 75 percent field capacity with distilled water. Visual evaluation and plant fresh weight harvest was 21 days after planting. Statistical analysis was a split-split plot with main effects being the unsterilized and sterilized greenhouse soils, sub-effects represented by storage temperature, and sub-sub effect equal to degradation time.

Results and Discussion

Ethofumesate residual level study. Crop sensitivity to ethofumesate increased with ethofumesate soil levels (Figure 19). In general, wheat was most susceptible to all ethofumesate levels evaluated. Oat, cucumber, and soybean interacted with rates, but each was still less sensitive than wheat. Dry edible bean and corn were very tolerant.

Wheat, oat, and cucumber were significantly injured from ethofumesate soil levels as low as 0.14 kg/ha. Soybean visual injury of 18 percent was observed from 0.28 kg/ha ethofumesate and increased to 74 percent at 2.24 kg/ha ethofumesate. Ethofumesate soil levels of 2.24 kg/ha caused 10 percent visual injury to dry edible beans and did not visually affect corn when compared to untreated control plants.

Plant fresh weights (Appendix 11) showed similar response to increasing ethofumesate levels as was observed through crop visual ratings. However, a masking effect often occurred at increasing ethofumesate levels. This masking effect occurred in short-term studies resulting in reduced differences when correlated to visual responses.

<u>Crop varietal response study</u>. Crop sensitivity to ethofumesate varied with levels in the soil and cultivars compared. Each crop evaluated showed significant cultivar sensitivity to ethofumesate soil levels of 0.56 kg/ha except

Figure 19. Bioassay crop visual response to ethofumesate soil levels in the greenhouse.



ETHOFUMESATE RATE (kg/ha)

dry edible beans and cucumbers, which responded to 1.12 kg/ha. Cultivar sensitivity to 1.12 kg/ha appeared to be masked within a given crop when compared to cultivar effects from 0.56 kg/ha. Differences existed if crop cultivars were ranked for the two ethofumesate soil levels (Table 2).

Of the crops evaluated, wheat showed the greatest injury from ethofumesate. Soil levels of 0.56 kg/ha caused visual injury between 54 and 89 percent for cvs. 'Yorkstar' and 'Tecumseh', respectively. Soft red winter wheat showed an intermediate cultivar response to 0.56 kg/ha when compared to soft white winter wheat cultivars. Oat visual injury from 0.56 kg/ha ethofumesate soil levels reached 65 percent with cv. 'Ausable'.

Soil studies. Visual response of bioassay crops to ethofumesate soil levels was affected by soil texture, and to a lesser degree by percent organic matter. The explanation for these effects is believed to be the number of active sites available for either short or long term herbicide attraction.

Ethofumesate at 0.56 kg/ha in a Belleville loamy sand or course soil texture caused greater visual injury to soybeans than in other soil textures evaluated. The induced injury response to both soybeans and wheat was reduced in a true clay soil textural type (Table 3).

Crop response to ethofumesate treated soil in the greenhouse was reduced at high percent organic matter (11.5 percent). Visual injury to both soybeans and wheat

	Ethofumesate induced	visual injury (%)
<u>Crop/cultivar</u>	<u>0.56 kg/ha</u>	<u>1.12 kg/ha</u>
Soft winter wheat ¹ Yorkstar Abe Augusta Arthur Cenesee	53.6 60.4 65.7 69.2	78.6 93.3 78.5 95.1
Tecumseh	89.0	93.9
LSD ² 0.05	(2.4))
Vat	22.2	61 0
Machinaw	40.7	01.9 77.2
Russell	40.9	65.0
Astro	42.0	81.8
Korwood	44.3	76.2
Ausable	64.1	84.5
	(0.2)	
Cucumber	(8.2)	,
Greenstar	61.7	72.5
Marketmore-70	63.3	81.7
0.05	(5.6)	
Dry edible bean		
Sanilac	0.8	4.1
Tuscola	1.7	1.7
Seafarer	2.5	5.0
	(2.6)	
Sovbean	(2.0)	,
Beeson	25.8	38.3
Hark	53.3	69.2
Corsoy	54.2	72.5
Harcor	54.2	75.0
2KL-TAA	55.0	61./
	(5.2)	

Table 2.	Bioassay crop cultivar response to ethofumesate
	as determined by visual injury ratings from
	greenhouse studies.

¹Cultivars Abe and Arthur are soft red winter wheat while others evaluated are soft white winter wheat varieties.

²Statistical significance exists if percent visual injury separation of crop cultivars by rate exceeds the 5% Least Significant Difference value.

in the greenhouse	•			
Soil type/texture ^l	Visual In Soybean	jury (%) Wheat	Fresh weight/plant Soybean	(% of control) Wheat
Belleville loamy sand	87.3	88.3	62.4	6.1
Badaxe sandy loam	51.7	91.3	84.1	7.3
Poseyville sandy clay loam	59.6	94.7	90.1	3.3
Sanilac silty clay loam	49.2	98.8	109.8	1.7
Toledo clay loam	42.1	91.8	78.2	19.1
Charity clay	39.2	72.9	81.9	14.3
LSD ² 0.05	(10.3)	(11.8)	(21.0	(6.4)
l Complete soil analyses for	each soil	texture ap	pears in Appendix 1.	

Soybean and wheat response to soil texture and ethofumesate soil level Table 3.

5% ²Statistical significance exists if value separation within a column exceeds the Least Significant Difference based on untreated controls of the respective crop. at 0.56 kg/ha was reduced to 41 and 78 percent, respectively (Table 4). However, visual injury response for soybeans and wheat was only a reduced effect and not eliminated with increasing percent organic matters.

Soil leaching study. The depth to which ethofumesate was leached with 25.4 cm surface irrigation water in greenhouse studies was dependent on rate of active ingredient applied. Ethofumesate at 0.56 and 8.96 kg/ha was leached to 10.16 and 22.86 cm, respectively (Figure 20). Bioassaying 2.54 cm segments within the columns gave an indication that the majority of ethofumesate remained in the uppermost 7.62 cm of soil at rates of 0.56 and 1.12 kg/ha. With increased application rates and leaching depths, visual injury to soybean still remained the greatest in the top 7.62 cm of soil surface.

Visual injury to soybean in soil leached by 25.4 cm surface irrigation confirmed the results of the column leaching study. Observable injury to soybeans from 2.24 kg/ha did not decrease until containers were bioassayed in soil with the upper most 10.16 cm of soil removed (Appendix 12).

Charcoal barrier study. The site of ethofumesate uptake by bioassay crops was compared to untreated controls. Activiated charcoal adhered to the first leaves of soybean, cucumber, and dry edible beans causing a leaf crinkling appearance that was difficult to differentiate from ethofumesate injury alone. Seed uptake determination included

TOŚ	im texture.				
Soil Type ^l	Organic Matter (%)	Visual In Soybean	jury (%) Wheat	Fresh weight/plant Soybean	(% of control) Wheat
Capac	2.7	57.1	93.3	106.1	6.9
Tappan**	3 . 5	62.3	99.2	52.5	2.0
Poseyville	4.0	59.6	94.7	90.1	3.3
Brookston	4.9	53.3	93.3	85.1	6.0
Shebeon	5.3	61.2	92.1	72.2	36.5
Tappan- Belleville	7.1	56.2	92.3	60.5	9.4
Owosso	11.5	40.8	77.9	73.6	52.6
, ISD	2 0.05	(10.3)	(11.8)	(21.0)	(6.4)
l Complete soi	l analysis for	each soil t	ype appears	in Appendix 1.	

and ethofumesate response on soybean All soils evaluated were sandy clay Effect of soil organic matter and wheat in the greenhouse. Table 4.

²Statistical significance exists if value separation within a column exceeds the 5% Least Significant Difference based on untreated controls of the respective crop.

Figure 20. Soybean response to ethofumesate levels leached by 25.4 cm surface irrigation in greenhouse column studies.



early shoot and radicle uptake along with penetration through the seed coat because of the width of treated band.

Uptake of ethofumesate by wheat was greatest at early embryo development (Table 5). However, sensitivity was shown both from shoot and root treated zones. The high level of ethofumesate involved may account for part of the wheat susceptability. Uptake by oat was exclusively by the shoot or coleoptile plant portion.

Uptake by soybean, cucumber, sugarbeet, and dry edible bean was primarily by roots. The high susceptibility level by the seed site concluded that this was early in radicle development. Corn uptake was imbibed into the seed or occurred at a very early embryonic plant stage.

Visual injury consisting of leaf fusion or minimal crinkling was not detected in plant fresh weights (Appendix 13). Therefore, fresh weight per plant determinations are slightly altered from conclusions based on visual injury.

Ethofumesate pH study. Information obtained from the hydroponic study to evaluate plant response to ethofumesate in pH buffered solutions was inconclusive since normal plant growth could not be maintained. Plant response in treated and untreated solutions was similar and plant vigor greater at or near neutral pH rather than under acidic conditions, regardless of ethofumesate application. Complications of chemical sedimentation also occurred in the hydroponic study containers at pH 7.5 or greater.

			ual Injury (8			
Cropl	Untreated control	Untreated control with barrier	Root uptake	Shoot uptake	Seed uptake	LSD ² 0.05
Wheat	0.0	3.8	62.5	62.5	96.0	(6.6)
Dat		2.5	8.8	30.0	10.0	(13.6)
Soybean	0.0	17.5	31.3	11.3	25.0	(14.7)
Cucumber		18.8	70.0	22.5	70.0	(12.5)
Sugarbeet	000	2.5	12.5	2.5	47.5	(11.3)
Dry bean		62.5	70.0	58.8	70.0	(12.3)
Corn		0.0	0.0	5.0	65.0	(9.5)
l <mark>Ethofumesa</mark> kg/ha.	te levels	for the three crop	groups are eq	uivalent to 1.	12, 2.24 and	4.48

Ethofumesate site of crop uptake as determined by visual injury through Table 5. "Statistical significance exists if value separation within a row exceeds the 5% Least Significant Difference based on untreated control of the respective crop.

The effect of soil liming on ethofumesate response to wheat and soybeans was similar to soil that was limed and treated (Table 6).

Ethofumesate degradation study. Ethofumesate level was not reduced if treated soil was stored at 15.6 C for a duration time of 14 weeks. Degradation was reduced by steam sterilization when stored at 23.9 to 29.4 C. Visual injury to soybeans from ethofumesate decreased to levels of 55% and 50% if untreated controls stored at 32.3 C for 14 weeks for sterilized and unsterilized greenhouse soil mix, respectively (Figure 21,22) Appendix 14).

Ethofumesate rate (kg/ha)	V Limed Soybean	'isual I soil Wheat	njury (%) ¹ <u>Unlimed</u> Soybean	soil Wheat
0	0.0	0.0	0.0	0.0
0.28	25.0	72.5	25.0	77.5
0.56	32.5	87.5	36.3	90.0
0.84	42.5	96.0	48.8	93.8
1.12	52.5	96.0	53.8	95.0
2.24	66.3	99.0	67.5	98.0

Table 6	•	Effect	of	soil	liming	on	visu	al	injury	of	soybean
		and whea	at	to e	thofumes	sate	e in 🛛	gre	enhouse	e st	udies.

¹Statistical differences exist among ethofumesate levels only and not for the limed versus unlimed soil fractions. Figure 21. Soybean visual response to bioassays in ethofumesate treated sterilized soil stored at 15.6, 23.9 to 29.4, and 32.2 C for 14 weeks.

STERILIZED SOIL 15.6 C r = 0.11 NS 70-• 60 14 12 2 10 0 6 8 4 90-VISUAL INJURY (%) 80-70-60-23.9-29.4 C 50-0.28 NS r Ξ 10 12 2 6 8 14 0 4 90-80-32.2 C r = 0.87**70-60 50 10 14 0 2 6 8 12 4 STORAGE PERIOD (weeks)

Figure 22. Soybean visual response to bioassays in ethofumesate treated unsterilized soil stored at 15.6, 23.9 to 29.4, and 32.2 C for 14 weeks.



LABORATORY STUDIES

Materials and Methods

Determination of ethofumesate residual in soil. Soil samples were collected from each broadcast applied ethofumesate treatment prior to cultivation techniques and from all plots following tillage. Hand sampled probes of 2.54 x 15.24 cm were obtained at 10 randomly selected sites in each plot. Soil samples were sealed and stored at -25 C until analyzed.

Prior to analysis, samples were allowed to thaw at 20 C. Treatment replications from each site were combined and a subsample was removed and placed in an air flow dryer at 25 C for approximately 15-20 h. Soil was passed through a 1 mm mesh sieve and underwent the following extraction procedure. Reagent grade solvents were used throughout all laboratory procedures.

Twenty grams of dry, sieved soil were equilibrated with 7.5 ml glass distilled water for 15 minutes. Then 67.5 ml of methanol were added to each flask, contents swirled and allowed to set for 30 minutes. After refluxing for 1.5 h, samples were cooled and filtered through Whatman No. 1 filter paper then 20-25 ml methanol were added to give a final filtered volume of 80 ml. The extract was added

to 180 ml of glass distilled water and 10 ml saturated sodium sulfate solution in a 500 ml separatory funnel. Then 200 ml dichloromethane were added to the separatory funnel mixture. The dichloromethane phase was percolated through a anhydrous sodium sulfate bed and rinsed with 25 ml of dichloromethane. Contents were concentrated to approximately 0.5 ml under vacuum on a rotary evaporator at 40 C. Following an addition of 25 ml acetone the solution was transfered to 15 by 110 mm screw cap culture tubes and evaporated just to dryness under a nitrogen gas stream. One-half ml of toluene was added, then tubes were capped and agitated. Short-term storage of tubes was at 3 C prior to detection. Dilutions of the 0.5 ml concentrates were made with toluene prior to electron capture detection by gas liquid chromotography. Separation of a 3 μ L sample was in a 2.0 m long by 2.6 mm diameter column packed with 10% OV-11 with supelcoport support and 100/120 mesh. Injection port temperature was 270 C with internal column temperature maintained at 230 C. Nitrogen flow rate was 40 ml/min. at a pressure of 2.4 kg/cm^2 .

Ethofumesate degradation study. Ethofumesate degradation over time was evaluated by utilizing an enclosed CO₂trap apparatus (Figure 23). Twenty-five grams of Tappan^{**}/ sandy clay loam soil (Appendix 1) was air dried and sifted through a 1 mm metal screen and distilled water added for an equivalent of 60 percent field capacity soil mixture.



One-half of the flasks were stoppered with aluminum foil and autoclaved at 120 C and 20 kg/cm² for 2 h. Flasks were stored in the autoclave for 48 h and autoclaved a second time along with the constructed trap holder and rubber stopper for 1 h. Ethanol volume of 100 μ L containing 0.211 μ Ci/100 μ L ring-labeled 14C-ethofumesate with specific activity of 1.16 mc/mmole was applied via syringe to the soil surface of sterilized and unsterilized flasks in an asceptisized hood. Untreated controls consisted of 100 µL ethanol applications to the soil surface. One ml of 1.0 M KOH was added to each 2 ml disposable beaker and flasks stoppered and stored in the dark at 23 C until analyzed. At weekly intervals, randomly selected replications of traps were added to 15 ml of NEN formula 963 (New England Nuclear) scintillation cocktail with 1 ml glass distilled water. Radioactivity was assayed by liquid scintillation spectroscopy.

Ethofumesate soil movement study. Ethofumesate movement in soil was determined by thin layer chromotopraphy (TLC) technique. Badaxe fine sandy loam, Capac sandy clay loam, Tappan-Belleville sandy clay loam, Capac sandy clay loam, Capac clay loam, and Charity clay soils (Appendix 1) were air dried and sieved through a 0.074 mm or 0.595 mm mesh sieve. Soil slurries were created by adding glass distilled water and 2 ml ethanol/100 ml slurry to remove air bubbles to each soil type. Soil thickness per plate was 0.50 and 0.75 mm for the 0.074 and 0.595 mm fractions, respectively. Soil TLC plates were allowed to air dry for approximately 24 h

prior to applications of radioactive material. The bottom 1 mm of each plate was scraped free of soil and wrapped with a strip of highly adsorbant paper. The adsorbant paper was extended from the end of the plate to the soil and held in position with rubber bands until water adhesion occurred. Two 12 μ L spots containing 0.015 μ Ci/12 μ L activity each of ¹⁴C-ethofumesate, and ¹⁴C-trifluralin (α, α, α -trifluoro-2, 6-dinitro-N,N-dipropyl-p-toluidine) and ¹⁴C-chloramiben (3-amino-2,5,dichlorobenzoic acid) as references were applied per plate. Plates were placed upright at a 60 degree angle in glass developing tanks. Tanks contained 150 ml glass distilled water which was allowed to move approximately 75 percent the height of the plate. Plates were air dried and Rf values of herbicide movement determined by Beta scan and radioautographs.

Ethofumesate soil adsorption studies. Soil adsorption studies consisted of passing air dried soils (Appendix 1) through a 1 mm sieve. Soil allotments of 0.5 gm were placed in 15 ml high speed glass centrifuge tubes. Six ml of glass distilled water was added per tube and amended with 25 μ L of ethanol containing 0.07 μ Ci/25 μ L of ¹⁴Cethofumesate. Tubes were stoppered with aluminum foil covered rubber stoppers and equilibrated 4 h at 20 C on a wristaction shaker at 300 cycles/min. Samples were then centrifuged at 30,000X G for 20 min at 20 C. A 0.5 ml sample of supernatant fluid was removed, placed in 15 ml of NEN

formula 963 (New England Nuclear) scintillation fluid, and assayed for radioactivity 10 min/sample by liquid scintillation spectrscopy. The quantity of herbicide bound was determined by loss of radioactivity from solution compared with tubes not containing soil. A replicated completely randomized design was utilized.

A Tappan**/sandy clay loam soil was studied to determine the length of time required for ethofumesate to equilibrate between the soil and aqueous phase of the suspension. The general procedure was modified by adding 3 ml of glass distilled water to 0.25 gm soil/tube and spiking with 12 μ L of ethanol containing 0.015 μ Ci/12 μ L ¹⁴C-ethofumesate. Samples were equilibrated for 0, 0.25, 0.50, 0.75, 1, 2, 4, 8, 24, 48, and 96 h, centrifuged and radioassayed.

Results and Discussion

Determination of ethofumesate residual in soil. A high degree of variability occurred from the residue analysis of experimental plots from field location-years (Table 7-10). Part of the variability could have resulted from sampling techniques or as a result of non-replicated ethofumesate extractions/treatment. Random selections of treatments were analyzed by the Residue Analysis Department of Fisons Corporation in England and found to be quite representative of results obtained from extractions of a similar subsample. However, certain generalizations can be made which are similar to field bioassay conclusions. Decreasing the

plots	
treatment	
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in so	Jassay
level	79 bid
esate	the 19
Ethofum	within
Table 7.	

Preemergence			Sampling Depth		1
Ethofumesate Rate	Cultivation Techniquel	0-7.62 cm Pre-Tillage	7.62-15.24 cm Pre-Tillage	0-15.24 cm Post-Tillage	ļ
l.l2 kg/ha	д	(ppm) 0.04	(ppm) 0.02	(ppm) 0.04	
	D	8	:	0.01	
3.36	д	0.33	0.47	0.12	
	D	8	-	0.45	
6.72	д	0.53	0.36	0.43	
	D	1	1	0.31	
10.01	д	0.78	0.42	0.45	
	D	!	1	1.21	
					I

¹P denotes plow while D represents disc.

Preemergence				Broadcast Applied	band Applied
Ethofumesate	Cultivation	0-7.62 cm	7.62-15.24 cm	0-15.24 cm	0-15.24 cm
Rate	Technique ¹	Pre-Tillage	Pre-Tillage	Post-Tillage	Post-Tillage
2 24 kg/ha	D	(mdq)	(mdg) 0 27	(mdd)	(mdq)
	۰ D	1 1 1 1	- - - -	0.09	0.01
	FC	;	ł	0.09	0.06
3.36	ሲ	0.23	0.49	0.03	0.01
	Ω	;	1	0.06	0.02
	FC	!	1	0.07	0.01
4.48	ዋ	0.40	0.50	0.02	0.01
	D	1	!	0.04	0.03
	FC	1	1	0.04	0.06
6.72	ሗ	0.39	0.22	0.07	0.05
	D	:	1	0.06	0.10
	FC	!	1	0.11	0.05
2.24 + 1.12	പ	0.06	0.17	0.05	0.05
Postemergence					
	D	1	1	0.02	0.01
	FC	1	t 1	0.05	0.02
2.24 + 2.24	പ	0.44	0.47	0.03	0.04
Postemergence					
	D	1	1	0.04	0.06
	FC	1	1	0.23	0.04

Cultivation <u>Techniquel</u>	Broadcast Applied 0-15.24 cm Post-Till	Band Applied 0-15.24 cm Post-Till
P	(ppm) 0.01	(ppm) 0.01
FC	0.01	0.01
Р	0.01	0.01
FC	0.03	0.01
Р	0.04	0.03
FC	0.15	0.01
Р	0.18	0.02
FC	0.27	0.08
Р	0.25	0.04
FC	0.80	0.11
	Cultivation TechniquelPFCPFCPFCPFCPFCPFCPFCPFCPFCPFCPFCPFC	Cultivation Techniquel Broadcast Applied 0-15.24 cm Post-Till P 0.01 FC 0.01 P 0.01 FC 0.01 P 0.01 FC 0.01 FC 0.03 P 0.04 FC 0.15 P 0.18 FC 0.27 P 0.25 FC 0.80

Table 9.	Ethofumesate level in soil samples collected
	from treatment plots within the 1980 field
	bloassay study, Bay Co., MI.

¹P denotes plow while FC represents field cultivator.

Ethofumesate Rate 0.56 kg/ha	Cultivation Techniquel P	Broadcast Applied 0-15.24 cm Post-Till (ppm) 0.09	Band Applied 0-15.24 cm Post-Till (ppm) 0.04
-	FC	0.05	0.05
1.12	Р	0.10	0.05
	FC	0.10	0.04
2.24	Р	0.64	0.31
	FC	0.74	0.56
4.48	Р	0.46	0.10
	FC	0.84	0.89

Table 10. Ethofumesate level in soil samples collected from treatment plots within the 1981 field bioassay, East Lansing, MI.

 $^{1}\mathrm{P}$ denotes plow while FC represents field cultivator.

ethofumesate application to sampling time increased ethofumesate levels. Plowing reduced ethofumesate levels in soils followed by discing, followed by field cultivation techniques. Less ethofumesate was detected from band versus broadcast applied ethofumesate.

In relating ethofumesate residue extracted to percent visual injury observed in the field, it was noted that for sensitive bioassay species, ethofumesate levels of 0.40 ppm or greater caused significant observable injury. The East Lansing field trial during 1980 did not contain levels greater than 0.23 ppm. Therefore, potential reason for undetected plant symptoms. Apparently, the ethofumesate applied was either leached below the sampled area or conditions were such that decomposition occurred during the period of time in the field.

Ethofumesate degradation study. Since ethofumesate was ring labeled, ${}^{14}\text{CO}_2$ production could only occur with cleavage and oxidation of the rings. The levels of radioactivity detected in the KOH vials were minimal (18.4 dpm/ week or <1% of the total) during the 10 week evaluation period (Figure 24). However, ${}^{14}\text{CO}_2$ levels detected were approximately twice as great if the soil was not steam sterilized prior to ${}^{14}\text{C}$ -ethofumesate application versus sterilized soil. Measurable radioactivity in the vials increased from 116 to 297 dpm and 412 to 610 dpm for sterile and non-sterile conditions, respectively during

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Figure 24. Cumulative ¹⁴CO₂ production from 14C-ethofumesate under sterile and non-sterile soil conditions for 14 weeks.


the 10 weeks. This would substantiate other research, that ethofumesate is primarily microbially decomposed. Major microbial activity appeared to be under aerobic conditions because of the soil environmental state. 14 Cethofumesate recovery efficiency from the soil throughout the experiment ranged from 64 to 67 percent of the initial amount added.

Ethofumesate soil movement study. Slight movement of ¹⁴C-ethofumesate was apparent in the soils evaluated. Rf values up to 0.18 occurred under conditions tested (Table 11). Herbicides used as controls for the various soils were ¹⁴C-trifluralin and ¹⁴C-chloramben, both with well documented soil characteristics. Water solubility of ethofumesate is reported as 110 ppm (23). Under these same conditions chloramben, (77), which is mobile in water at a 700 ppm water solubility level, moved to a Rf value up to 0.74. Trifluralin is believed to be a result of a fairly high vapor pressure rather than water solubility. No difference in ethofumesate movement was noted if the thickness of the soil on the TLC plate or the particle size was altered. Of the soils evaluated 14 C-ethofumesate movement was decreased with organic matter levels of 6.9 and 22.6 percent. Soil texture and ¹⁴C-ethofumesate movement within soil from field experimental sites of 1979 and 1980 showed no difference. Therefore, the similarity in rainfall and environmental conditions between the two years did not explain the highly significant-no effect situation that resulted.

Table 11.	Rf values of ¹ compounds as d of six Michiga	⁴ C-ethofumess etermined fro n soils.	te, ¹⁴ C-trif m soil thin	lluralin, and ¹⁴ (layer chromatog	C-chloramben parent caphy radioautographs
			Soil Sie	ve Size	
Michigan		0.074 mm).595 mm
Soil Type	Ethofumesate	Trifluralin	Chloramben	Ethofumesate T	cifluralin Chloramben
Badaxe fin sandy loam	e 0.09	<0.01	0.62	0.14	<0.01 0.62
Capac sand clay loam ²	y 0.17	<0.01	0.70	0.17	<0.01 0.62
Tappan- Belleville sandy clay loam ³	1	1	1	0.17	<0.01 0.68
Capac sand clay loam ⁴	<i>Y</i> 0.18	<0.01	0.75	0.17	<0.01 0.73
Capac clay loam	0.08	<0.01	0.77	0.06	<0.01 0.74
Charity cl	ay	;	1	0.15	<0.01 0.71
l Complete :	soil analysis i	s located in	Appendix 1.		
² soil type	from 1979 etho	fumesate resi	due field bi	oassay location,	, East Lansing, MI.
³ soil type	from 1980 etho	fumesate resi	due field bi	oassay location,	, Bay County, MI.
⁴ Soil type	from 1980 etho	fumesate resi	due field bi	oassay location,	, East Lansing, MI.

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Ethofumesate soil adsorption studies. Based on the radioactive level added to each tube, ¹⁴C-ethofumesate was highly adsorbed to all types of soils tested (Table 12). The level adsorbed to the soil was further affected by two major factors, percent organic matter and soil texture. The increase in binding sites by both factors caused an apparent unseparable interaction. Binding of ¹⁴C-ethofumesate was greatest to montmorillinite and least with kaolinite. In general, as the number of active adsorption sites increased, ¹⁴C-ethofumesate adsorption also increased.

Adsorption of ¹⁴C-ethofumesate appeared to be bound to the soil almost immediately if agitated. Also, minor increases in adsorption were noted if allowed to agitate for increasing periods of time (Table 13).

Soil type/texture	Superr Radioactiv	natant vity (dpm)
Quartz sand	9863	fg
Belleville loamy sand	7753	cde
Badaxe sandy loam	7441	bcd
Tobico sandy loam	7915	cde
Owosso sandy clay loam	6857	bc
Whitaker sandy clay loam	8010	cde
Tappan sandy clay loam	8333	cdef
Capac sandy clay loam ²	8883	defg
Capac sandy clay loam3	8887	defg
Tappan sandy clay loam	8945	defg
Sanilac-Bach sandy clay loam	8950	defg
Poseyville sandy clay loam	8994	defg
Tappan-Belleville sandy clay loam [*]	8999	defg
Shebeon sandy clay loam	9048	defg
Sanilac sandy clay loam	9288	efg
Shebeon-Badaxe silty clay loam	8301	cdef
Toledo clay loam	8672	defg
Shebeon clay loam	8736	defg
Parkhill clay loam	8862	defg
Kilmanagh clay loam	9095	defg
Charity clay	7980	cde
Montmorillinite #31	6013	ab
Kaolinite #9	10302	g

Table 12. Adsorption of ¹⁴C-ethofumesate by several unaltered Michigan soil texture types, quartz sand, montmorillinite, and kaolinite.

¹Means with similar letters are not significantly different at the 1% level by Duncan's multiple range test.

²Field bioassay location, East Lansing, MI, 1979.

³Field bioassay location, East Lansing, MI, 1980.

⁴Field bioassay location, Bay County, MI, 1980.

Time after treatment (h)	Supernatant radioactivity (dpm) ¹
0.00	10107 c ²
0.25	9190 bc
0.50	9207 bc
0.75	9224 bc
1.00	8976 abc
2.00	9299 bc
4.00	8974 abc
8.00	8495 ab
24.00	8751 abc
48.00	8189 ab
96.00	7627 a

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¹⁴C-ethofumesate adsorption to Tappan**/sandy clay loam after several equilibration time Table 13. interavls.

¹Initial radioactivity applied/vial was equivalent to 70476 dpm.

²Means with similar letters are not significantly different at the 1% level by Duncan's multiple range test. Regression analysis is significant at the 1% level where r = -0.84.

SUMMARY

In conclusion, field bioassay crop response to ethofumesate levels varied among location and years. However, visual injury to bioassay plants indicated that 75 percent and 90 percent of the ethofumesate had dissipated after 8 and 12 months, respectively. Broadcast applications of ethofumesate caused greater bioassay crop injury and yield reductions than equivalent band applications. Plowing the ethofumesate treated areas reduced ethofumesate response to bioassay crops more than discing which was reduced more than field cultivation. A soil pH range between 4.3 and 7.0 did not appear to alter soybean response to ethofumesate residue.

Crop responses to ethofumesate in greenhouse studies were primarily based on immature plant visual evaluations. Plant fresh weights were measured at study termination. However, ethofumesate affects on plant weight lacked significance when compared to untreated controls because of the foliar nature of the injury. Plants with ethofumesate induced foliar symptoms of crinkling and necrosis had similar mass as unaffected plants, unless extreme plant stunting was observed. Concluding studies after short periods reduced plant weight differences that would be enhanced if allowed to continue to maturity. Preliminary experiments

indicated that ethofumesate did not affect germination of any crop species used in greenhouse studies. To eliminate the variability in plant populations per treatment container, fresh weight data is presented on a per plant basis.

Therefore, wheat was visually more susceptable ethofumesate levels than oat, cucumber, and soybean which in turn were more sensitive than dry edible beans. Corn was not affected by levels to 2.24 kg/ha. Common Michigan grown cultivars of each crop evaluated showed varied response to ethofumesate at 0.56 kg/ha. Increasing the soil ethofumesate level to 1.12 kg/ha masked several cultivar differences that were observed at 0.56 kg/ha.

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Table Land

Ethofumesate induced visual injury to soybean and wheat increased if bioassayed in coarse soil texture and low percent organic matter. The depth to which ethofumesate leached increased with amount of active ingredient applied but visual response to soybeans remained greatest at the soil surface.

Uptake of ethofumesate showed varying crop response. In general, site of ethofumesate uptake appeared to be via root for broadleaf test species and shoot for grass species. Often, time of ethofumesate uptake was substantial during early radical or shoot growth.

Ethofumesate degradation over time was greatest in unsterilized soil stored at 32.3 C.

Detectable ethofumesate extracted from field treatment soil samples varied among experimental plots, locations, and

years. However, ethofumesate levels greater than or equal to 0.40 ppm caused significant visual injury when related back to field bioassay studies.

¹⁴C ring-labeled ethofumesate degradation decreased under sterile soil conditions, suggesting possible microbial involvement. ¹⁴CO₂ evolution during a 10-week time span was less than 1 percent if ¹⁴C-ethofumesate was added to either sterile or non-sterile moist soil. ¹⁴C-ethofumesate movement decreased if soil organic matter percent exceeded 6.9

Adsorption of ¹⁴C-ethofumesate to several Michigan soils occurred almost immediately after treatment agitation. However, adsorption increased slightly with increased active sites.

Ethofumesate residual carry-over to subsequent rotational cropping sytems does not appear to be an economic problem with labeled use rates and current production practices in Michigan. Generally, the warm-moist conditions in Michigan enhance microbial activity which accounts for the primary decomposition of ethofumesate. However, with the maximum treatment rates allowed, reduced tillage, and/or extreme environmental conditions, the potential does exist for visual symptoms. A possible solution if a potential problem is suspected, would be to follow sugarbeets in the rotation with dry beans or corn. A second alternative if soybeans are to be planted may be the selection of a more tolerant cultivar coupled with conventional tillage practices. In the case of a sugarbeet crop failure, dry beans or certain

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corn varieties may be replanted without severe damage, especially if soil organic matter levels are high or where an additional tillage practice is utilized.

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APPENDICES

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	Organic matter	Me soil	chanica analysi	1 8 (9)	Coil tovtural	۵	¥	۳	¢,	Water coluble	Nitrate
Hd	(8)	sand	silt	clay	class1	-1	(<u>Ř</u> g/F	la)	21	salts (mmhos)	(mqq)
7.4	10.4	82.1	5.0	12.9	losa	178	251	4002	318	0.47	98.7
5.6	6.9	68.4	14.3	17.3	salo	179	399	2210	186	0.65	143.5
7.7	9.8	70.0	11.0	19.0	salo	15	49	4062	376	!	92.1
7.0	11.5	66.4	10.0	23.6	sacllo	213	466	6451	768	ļ	264.3
7.2	4.9	47.7	22.0	28.3	sacllo	83	224	4898	884	!	113.4
7.0	5.5	48.2	22.0	29.8	sacllo	242	394	6213	768	:	267.9
8.1	5.5	45.1	23.6	31.3	sacllo	227	233	7620	455	1	85.9
7.6	3.5	49.9	16.0	34.1	sacllo	245	246	4898	861	0.34	67.0
5.2	2.7	51.5	23.2	25.3	sacllo	328	305	1672	157	0.65	107.3
7.3	3.2	46.0	24.4	29.6	sacllo	ł	ł	ł	1	0.08	ŀ
7.7	7.1	51.5	16.2	32.3	sacllo	161	197	6331	613	0.29	35.9
7.9	3.5	45.7	21.0	33.3	sacllo	85	233	6374	790	:	41.2
7.6	4.0	61.3	14.0	24.7	sacllo	35	237	3942	498	1	83.6
7.3	5.3	50.1	24.4	25.5	sacllo	187	255	4659	624	:	52.9
8.0	5.0	18.8	52.0	29.2	sicclo	84	255	7181	816	0.58	132.1
7.6	3.2	43.9	24.9	31.2	cllo	36	184	4719	739	;	58.3
7.5	3.8	41.3	24.0	34.7	cllo	52	66	5935	904		40.6
7.7	4.2	22.0	44.0	34.0	cllo	105	372	7057	435	0.35	75.8
7.2	3.3	42.0	19.0	39.0	cllo	176	426	5078	976	1	116.6
7.5	3.9	42.1	20.4	37.5	cllo	208	340	6072	883	ł	85.9
6.8	22.6	37.0	34.0	29.0	cllo'	207	887	14575	1536	ł	389.7
6.6	4.1	1.0	30.0	69.0	cl	60	448	7885	755	;	49.3
	0 0 0 0 0 0 0 0 0 0 0 0 0 0	pH Organic 0rganic organic 7.4 10.4 7.7 10.4 7.7 9.8 7.7 9.8 7.0 11.5 7.0 11.5 7.0 11.5 7.0 11.5 7.0 11.5 7.0 11.5 7.1 3.5 7.1 3.5 7.5 3.6 7.7 3.5 7.7 3.5 7.5 3.6 7.5 3.6 7.5 3.6 7.5 3.6 7.7 3.5 7.7 3.5 7.7 3.5 7.7 3.5 7.7 3.5 7.6 3.5 7.7 3.5 7.7 3.5 7.7 3.2 7.6 3.3 7.7 3.2 7.7 3.2 7.7 3.3 6.6 4.1 6.6 4.1	pH Organic Me 7.4 10.4 82.1 7.7 9.8 70.0 7.7 9.8 70.0 7.0 11.5 68.4 7.0 11.5 68.4 7.0 11.5 68.4 7.0 11.5 68.4 7.0 11.5 68.4 7.0 11.5 68.4 7.0 11.5 68.4 7.0 3.5 48.2 7.3 3.5 48.2 7.3 3.5 48.2 7.3 3.5 48.2 7.3 3.5 48.2 7.5 3.5 45.7 7.5 3.5 45.7 7.5 3.2 45.7 7.5 3.3 41.3 7.5 3.3 41.3 7.5 3.3 42.0 6.6 4.1 1.0 6.6 4.1 1.0	DH Organic Mechanica pH (%) soil analysi 7.4 10.4 82.1 5.0 7.7 9.8 70.0 11.0 7.0 11.5 66.4 14.3 7.0 11.5 66.4 10.0 7.2 4.9 87.7 22.0 7.0 11.5 66.4 10.0 7.0 11.5 66.4 10.0 7.2 4.9 87.7 22.0 7.1 5.5 48.2 22.0 7.6 3.5 49.9 16.0 7.7 3.5 49.9 16.0 7.3 3.5 49.9 16.0 7.3 3.5 49.9 16.0 7.3 3.5 49.9 16.0 7.3 3.5 49.9 16.0 7.6 4.0 31.4 0 7.7 3.5 45.7 21.0 7.7 3.5 45.7 21.0 7.6 3.2 43.9 24.0 7.7 3.3 42.0 19.0 7.6 4.1 1.0 30.0 7.5 3.2 47.1 24.0 7.7	DefinitionCorganic CorganicMechanical analysis (%) 7.4 10.482.1 5.0 12.9 7.7 9.870.011.019.0 7.7 9.870.011.019.0 7.0 11.566.410.028.3 7.0 11.566.410.028.3 7.0 11.566.410.028.3 7.0 11.566.410.028.3 7.0 11.566.410.028.3 7.6 5.5 49.9 16.0 34.1 7.7 3.5 49.9 16.0 34.1 7.7 7.1 51.5 23.2 24.4 7.7 3.5 45.1 23.6 31.3 7.6 4.0 34.1 24.0 34.1 7.7 3.5 45.7 21.0 24.7 7.7 3.5 45.7 21.0 34.0 7.7 3.5 45.0 24.0 34.0 7.7 3.2 44.0 34.0 7.7 3.2 42.0 19.0 34.0 7.7 3.2 42.0 19.0 34.0 7.7 3.2 42.0 10.0 24.7 7.7 4.2 22.0 44.0 34.0 7.7 3.2 42.0 19.0 34.0 7.7 3.2 34.0 34.0 34.0 7.7 3.2 42.0 19.0 34.0 7.7 3.2 34.0	Organic 	Organic 	$\begin{array}{c cccc} \text{Mechanical} \\ \hline \text{DH} & \begin{array}{c} \text{Organic} & \text{Mechanical} \\ \hline \text{matter soil analysis (8)} \\ \hline \text{Soil textural} & \begin{array}{c} \text{Soil textural} & \underline{P} & \underline{K} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{silt}} & \underline{\text{classl}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{(8)} & \begin{array}{c} \text{sand} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} \\ \hline \text{sand} & \begin{array}{c} 179 & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \begin{array}{c} 197 & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} \\ \hline \text{sand} & \underline{\text{sand}} & \text{sand$	$\begin{array}{r c c c c c c c c c c c c c c c c c c c$	Departic organicMechanical matterSoil analysis (%) sindSoil tanalysis (%) 	Definition DefinitionOrganic ConditionMater soluble ($\$)$ Mater soluble ($\$$)Mater soluble sale PH Soil sand siltSoil textural class1PKCaMgMater soluble7.410.488.15.012.9105a17825140023180.477.69.870.011.019.08alo17825140023180.477.011.566.410.021.6sacilo1782514066517687.011.566.410.021.6sacilo1782514066517687.011.566.410.021.6sacilo2134666517687.011.566.410.021.68acilo2423346038647.011.566.410.021.38acilo2423346137687.15.548.223.631.38acilo242334613667.15.548.223.376.249561.30.657.15.548.223.376.24550.657.231.251.332.44998660.657.351.551.523.352.723356.21570.657.351.551.623.38acilo242245

Analyses of several soil types collected in the major sugarbeet production area of Michigan. Sampling depth was equivalent to the upper-most 6.72 cm of the soil profile. All chemical and mechanical analyses were conducted by the Michigan State University Soil Testing Laboratory, East Lansing. Appendix 1.

¹Abbreviation key for soil textural class: sa - sand, lo - loam, cl - clay, si - silt.

²Field bioassay location, East Lansing, MI, 1979.

³Field bioassay location, East Lansing, MI, 1980.

⁴Field bioassay location, Bay County, MI, 1980.

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<u>1978</u>	<u>1979</u>	1980	<u>1981</u>
4.55	8.31	8.61	15.24
4.62	5.28	6.86	9.47
7.19	13.06	11.91	9.02
4.39	6.81	10.34	4.04
8.05	9.93	16.56	6.96
11.13	0.00	9.09	14.45
39.93	43.39	63.37	59.18
69.70	65.38	72.64	
	$ \begin{array}{r} 1978 \\ 4.55 \\ 4.62 \\ 7.19 \\ 4.39 \\ 8.05 \\ 11.13 \\ 39.93 \\ 69.70 \end{array} $	$ \begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

Appendix 2.	Precipitatio	n data col	llected 1	near Ingha	am
	County site	locations	at East	Lansing,	1978-81.

¹Normal yearly precipitation averaged over the past 15 years equivalent to 77.19 cm.

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Appendix 3. Herbicide treatment list for East Lansing experimental field site 1979. Treatment/ combinations were broadcast preemergence applied to surgarbeets May 10, 1978. Post emergence applications were June 9, 1978.

Ethofumesate	3.36
Ethofumesate	6.72
Ethofumesate	10.08
Ethofumesate + TCA	6.72 + 6.72
Pyrazon + TCA	4.48 + 6.72
Ethofumesate + R-25788	3.36 + 0.56
Ethofumesate + R-25788	6.72 + 0.56
Ethofumesate + R-25788	10.08 + 0.56
Ethofumesate + TCA + R-25788	6.72 + 6.72 + 0.56
Pyrazon + TCA + R-25788	4.48 + 6.72 + 0.56
Ethofumesate + R-25788	3.36 + 1.12
Ethofumesate + R-25788	6.72 + 1.12
Ethofumesate + R-25788	10.08 + 1.12
Ethofumesate + TCA + R-25788	6.72 + 6.72 + 1.12
Pyrazon + TCA + R-25788	4.48 + 6.72 + 1.12
(Desmedipham + Phenmedipham + endothall + crop oil concentrate)	(0.56 + 0.56 + 0.56 + 1% v/v)
Ethofumesate + (Desmedipham + crop oil concentrate)	1.12 + (0.84 + 1% v/v)
Ethofumesate + (Desmedipham + endothall + crop oil concentrate)	1.12 + (0.84 + 0.56 + 1% v/v)

() denotes foliar applied portion of treatment combination.

Appendix 4. Effect of ethofumesate residue level on oat plant population and foliage dry weight under different cultivation techniques. 1979 field bioassay, East Lansing, MI.





Appendix 4. Effect of ethofumesate residue level on oat plant population and foliage dry weight under different cultivation techniques. 1979 field bioassay, East Lansing, MI.





Appendix 5. Effect of ethofumesate residue level on soybean plant population under different cultivation techniques. 1979 field bioassay, East Lansing, MI.



Appendix 6. Effect of ethofumesate residue level on cucumber plant population and vine fresh weight under different cultivation techniques. 1979 field bioassay, East Lansing, MI.





Appendix 7. Oat plant population response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., MI.





Appendix 8. Oat foliage dry matter production from broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., MI.





Appendix 9. Cucumber plant population response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., Mi.





Appendix 10. Cucumber vine fresh weight response to broadcast and band spring applied ethofumesate treatments under different cultivation techniques. 1980 field bioassay, Bay Co., MI.

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Appendix ll.	Effect of et from greenhc	thofumesate l ouse studies.	evels on bioa	ssay crop	foliar fresh	weights
Ethofumesate (kg/ha)	Wheat (g/plant)	Oat (g/plant)	Cucumber (g/plant)	Soybean (g/plant)	Dry bean (g/plant)	Corn (g/plant)
0	0.397	0.408	1.001	1.046	1.868	1.377
0.14	0.330	0.408	1.050	1.018	2.036	1.362
0.28	0.127	0.423	0.869	1.257	2.198	1.401
0.56	0.029	0.362	0.896	1.199	1.501	1.265
1.12	0.012	0.169	0.756	1.098	2.512	1.314
2.24	0.002	0.043	0.469	1.024	1.938	1.244
LSD ¹ 0.05	(060.0)	(0.141)	(0.217)	(0.250)	(SN)	(SN)
l Statistical	significance	exists among	ethofumesate	levels if	plant fresh	weight

Appendix	11.	Effect of ethofumesate	levels	d no	ioassay	crop	foliar	fresh	weight
		from greenhouse studies	•						

separation within a given column exceeds the 5% least significant difference value.

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injury of soybean bloa	ssays in the greenhouse.
Soil removed from container surface prior to bioassay initiation (cm)	Soybean visual injury (१)
2.54	84.3
5.08	85.3
7.62	86.7
10.16	52.5
LSD ¹ 0.05	(14.6)

Appendix 12. Ethofumesate leaching depth from 25.4 cm surface irrigation water as determined by percent visual injury of soybean bioassays in the greenhouse.

¹Statistical significance among bioassay depths if percent visual injury rating separation exceeds the 5% least significant difference value.

Appenutx 13.	from char	are site of crop up coal studies in the	greenhouse		TTEST N	argue per prane
		Fresh we	eight/plant	(mg)		
Cropl	Jntreated control	Untreated control with barrier	Root uptake	Shoot uptake	Seed uptake	LSD ² 0.05
Wheat	0.30	0.20	0.08	0.08	0.01	(0.03)
Oat	0.31	0.25	0.26	0.12	0.22	(0.06)
Soybean	1.91	1.48	1.39	1.34	1.51	(0.29)
Cucumber	3.28	2.20	1.01	1.97	1.13	(1.04)
Sugarbeet	2.40	2.57	2.87	2.22	1.05	(0.59)
Dry bean	2.80	0.81	1.24	1.04	2.62	(2.08)
Corn	2.75	2.44	2.37	1.73	1.47	(0.59)
lEthofumesat sugarbeet 2	e levels fo .24; and dr	r wheat and oat are y bean and corn 4.48	equivalent 8 kg/ha.	to 1.12;	soybean,	cucumber, and

nlant ner weight frech γų ned determi U π untako ç しよし Գ C a + i a (4 0 E+hofim č Annendix

²Statistical significance exists if value separation within a row exceeds the 5% least significant difference based on untreated controls of the respective crop.

Appendix 14. Ethofumesate degradation as effected by time and soil storage temperature. Determinations were obtained from foliar fresh weight of soybeans grown in unsterilized and steam sterilized soil.

		Soybean	Fresh W	eight/P	lant (gm)	
	Un	sterilized So	<u>i1</u>	S	terilized Soi	1
	Stora	ge Temperatur	e (C) ¹	Stora	ge Temperatur	e (C) ²
Degradation (weeks)	15.6	23.9 - 29.4	32.2	15.6	23.9 - 29.4	32.2
0	1.18	1.18	1.18	1.15	1.15	1.15
1	1.15	1.20	1.24	1.20	1.10	1.39
2	1.25	1.51	1.30	1.40	1.49	1.19
3	1.52	1.40	1.72	1.45	1.51	1.40
4	1.30	1.63	1.40	1.91	1.74	1.26
5	1.34	1.71	1.69	1.49	1.82	1.42
6	1.24	1.58	1.61	1.44	1.56	1.24
7	1.23	1.54	1.53	1.41	1.56	1.76
8	1.20	1.43	1.55	1.30	1.63	1.40
9	1.88	1.83	1.42	1.57	1.65	1.64
10	1.05	1.37	1.60	1.32	1.19	1.49
11	1.06	1.57	1.64	1.46	1.34	1.61
12	1.13	1.49	1.61	1.36	1.40	1.35
13	1.10	1.51	1.33	1.27	1.55	1.44
14	1.34	1.79	1.79	1.38	1.54	1.62

Statistical significance over time at the 5% least significant difference level exists if plant fresh weight separation within a column exceeds 0.43 and 0.40 for 1 and 2 respectively.

