

8-29-79

Task 1. Review of BLAZER

Contract No. 68-01-5830

Final Report

August 29, 1979

**CONFIDENTIAL BUSINESS INFORMATION - DOES
NOT CONTAIN NATIONAL SECURITY INFORMATION
(E.O. 12065)**

SUBMITTED TO:

**Environmental Protection Agency
Arlington, Virginia 22202**

SUBMITTED BY:

**Enviro Control, Inc.
One Central Plaza
11300 Rockville Pike
Rockville, Maryland 20852**



TABLE OF CONTENTS

<u>STUDY</u>	<u>TITLE</u>
1	A Hydrolysis Study With ^{14}C -RH-6201
2	A Study of the Photolysis of ^{14}C -RH-6201 in Water
3	Long Term Laboratory Photolysis Study of RH-6201 on Soil
4	The Photodecomposition of RH-2512 on Silica Gel Thin Layer Chromatography Plates
5	^{14}C -RH-6201 Activated Sludge Metabolism
6	RH-6201 Laboratory Soil Metabolism Study
7	The Adsorptive and Desorptive Behavior of RH-6201 on Drummer Silt Loam Soil
8	A Residue Decline Study of Soil Treated With ^{14}C -RH-6201 at Newtown, Pa., in 1977
9	Metabolism of ^{14}C -RH-6201 in Rats, Soybeans and Soil
10	A summary of RH-6201 Residue Data for Soybeans and Soil
11	Leaching and Mobility Studies in Soil Treated With RH-6201
12	Laboratory Leaching Study With Soil-Aged RH-6201
13	Translocation Studies With ^{14}C -RH-6201 on Soybeans, Rice, Cocklebur and Nutsedge
14	A Residue Decline Study on Soybeans Treated Preemergence or Postemergence With ^{14}C -RH-6201 at Newtown, Pa.
15	A Residue Decline Study on Soybeans Treated Preemergence or Postemergence With ^{14}C -RH-6201
16	^{14}C Residue Studies on Rotation Crops Grown in Plots Treated With ^{14}C -RH-6201
17	^{14}C Residue Studies on Rotation Crops Grown in Plots Treated with ^{14}C -RH-6201 at Newtown, Pa.
18	^{14}C Residue Studies on Rotation Crops from Plots Treated With ^{14}C -RH-6201 at Newtown, Pa.

STUDY

TITLE

- 19 ¹⁴C Residue Studies on Rotation Crops from Plots Treated With ¹⁴C-RH-6201 at Newtown, Pa., in 1977
- 20 Evaluation of RH-6201 Against Environmentally Important Microorganisms
- 21 Accumulation and Elimination of RH-6201 Residues in Bluegill and Catfish

CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1975). A Hydrolysis Study with ¹⁴C-RH-6201 (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 3423-75-66)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: David Arieti, Staff Scientist, Enviro Control, Inc., Rockville, Md.

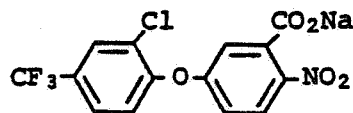
DATE OF REVIEW: August 16, 1979

TEST TYPE: Hydrolysis

CONCLUSION:

This study was performed according to EPA guidelines for registration of pesticides. In the opinion of the reviewer, this study meets the stated objectives, but it is still unclear whether hydrolysis does or does not take place because of the higher concentration measured after 56 days. The concentration of starting material actually rose from 46.8 ppm on day 0 to 60.0 ppm on day 56. The increase is explained by evaporation; thus, there is an experimental error of about 20%, or more if hydrolysis is actually taking place.

MATERIALS AND METHODS:



RH-6201

For the hydrolysis study, RH-6201 labeled in the carboxyl group was used at 1 and 50 ppm. These solutions were incubated for 56 days at 25°, 36°, and 48°C. Aliquots were removed for radioassay and examined with thin layer chromatography on silica gel. The TLC plates were visualized under UV lamps. Radioautography was also performed.

REPORTED RESULTS:A. Hydrolysis Solutions (Refer to Table I)

With nominal concentrations of 1 ppm at the three pH levels, actual concentrations of labeled RH-6201 ranged from 0.95 ppm (pH 4.5, 36°C, day 0) to 1.7 ppm (pH 9.7, 48°C, day 28).

B. Benzene Extractions (Refer to Table II)

At 50 ppm nominal concentrations, the carbon-14 extracted into benzene from the hydrolysis solutions ranged from 16.6% (pH 4.5, 25°C, day 0) to 98.6% (pH 9.7, 48°C, day 7) with most values in the area of 97%.

At 1 ppm nominal concentrations, the carbon-14 extracted into benzene from hydrolysis solutions ranged from 6.8% (pH 7.2, 36°C, day 0) to 98.3% (pH 7.2, 36°C, day 14; pH 9.7, 48°C, day 7). Four values were based on second counts from extracted aqueous solutions. The lowest was 73.1% (pH 7.2, 36°C, day 14); the highest was 98.4% (pH 4.5, 25°C, day 14).

C. Radioautographs of TLC (Refer to Table III)

The only radioactive component present was RH-5781 (the free acid form of RH-6201). Based on these results, it appears that labeled RH-5781 comprises more than 95% of the radioactivity present in the TLC plate. According to the researcher, no water-insoluble or volatile products were formed.

DISCUSSION:

1. Hydrolysis studies are required by EPA guidelines for registration of a pesticide. This study was conducted to meet these guidelines.
2. Only carboxyl-labeled RH-6201 was used. In order to clearly understand what happens to the aromatic portion, ring-labeled RH-6201 should also be used.
3. The actual concentration of labeled RH-6201 at 50 ppm found after treatment intended to cause hydrolysis is greater in most cases than the initial concentration. The researcher suggests that this is due to evaporation. The greatest concentration was 60.0 ppm. This is 20% greater than the initial concentration. Out of 45 samples tested at three pH levels and three temperatures, only 16 were below 50 ppm, and averaged 48.7 ppm. These account for only 33% of the samples. They were counted on day 0 and day 7.

4. The actual concentration of labeled RH-6201 was greater than the nominal 1 ppm by as much as 68% at day 28. Again, this was probably due to evaporation. Only three readings were below 1.0 ppm. The last reading had a gain of 68%. Since this was incubated at 48°C, it is expected that evaporation would be greatest.
5. There are four low extraction efficiencies scattered throughout the tables. These low extraction efficiencies are considered by the researcher to be errors. This appears to be a valid conclusion in this case because all four readings are below 17%.
6. The researcher points out that three values for percentage of carbon-14 had to be done twice because the pH levels were very close to 3, the value recommended in the guidelines. If the analysis was correct, hydrolysis may have indeed taken place. The same error occurred with 1 ppm nominal levels of labeled RH-6201.

Acifluorfen

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Pages 7 through 10 are not included in this copy.

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- Identity of product inert ingredients.
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CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). A Study of the Photolysis of ¹⁴C-RH-6201 in Water (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-14)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M. Edwards, Environmental Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 8, 1979

TEST TYPE: Photolysis Study in Water

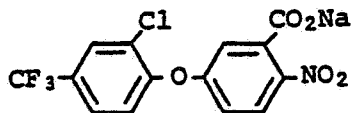
CONCLUSIONS:

1. The author concluded that, based on results of these studies, RH-6201 will be rapidly photodegraded in actual environmental situations. In the opinion of the reviewer, this conclusion is premature (especially in the absence of dark controls) and totally ignores the finding that degradation of carboxyl-labeled RH-6201 did not occur under sunlamp conditions in the absence of a sensitizer. This observation would require further investigation to determine its potential relevance to the degradation of RH-6201 under actual field conditions, and to explain the discrepancies between this finding and the later experiments which did show significant photodegradation.
2. The author also concluded that RH-6201 is initially photochemically decarboxylated to give rise to RH-2512 and unknowns. It was hypothesized that RH-2512 was further degraded through loss of trifluoromethyl as labeled carbon dioxide, with a second possible pathway involving the loss of trifluoromethyl followed by decarboxylation. While the data indicate the formation of RH-2512 from RH-6201, further study would be required to confirm the hypothesis about degradation of RH-2512.

3. In the opinion of the reviewer, the report as a whole was confusing and inconsistent with regard to such factors as test durations, the use of sensitizers, blacklight vs. sunlamp, and the use of the carboxyl labeled RH-6201 preparation vs. the trifluoromethyl group labeled compound. Furthermore, the study failed to conduct follow-up investigations to explain inconsistencies in findings, including the lack of photodegradation in one experiment and observations of polar, water-soluble photoproducts under some conditions but not in others. While investigation of this latter discrepancy would be beyond the scope of the study, investigation into the former finding should have been an integral part of these experiments.
4. Information on artificial light sources, intensities, wavelength distributions, and duration of exposure was provided. However, data on intensities and wavelength distributions were obtained from manufacturers and were apparently not determined under the actual experimental conditions. It also appears that unexposed controls were not run for these experiments, which limits the value of all the data, and means that photodegradation under these conditions is only an assumption.
5. The study did provide half-life estimates and a materials balance for the various studies as required by EPA proposed guidelines, but as a whole the procedures used to arrive at these data were not within EPA recommendations for the conduct of such studies, as discussed above.

MATERIALS AND METHODS:

Two preparations of the herbicide RH-6201 were used in these experiments, one labeled at the trifluoromethyl carbon and the other labeled at the carboxyl carbon with carbon-14.



RH-6201

The photolysis unit (Figure 1) consisted of a pyrex glass cell containing the test solution. A stopcock allowed for the removal of test solution during the experiments, and impinger traps provided for the collection of volatile products and evolved carbon dioxide. These products were carried to the traps by a continuous flow of carbon dioxide-scrubbed air through the system. The light sources used were a 20-watt fluorescent sunlamp (Westinghouse FS20) and a fluorescent blacklight (GE F20T12-BLB-Blacklight). The UV cutoff of the photolysis cell was approximately 290 nm.

Five-ppm aqueous solutions of the RH-6201 preparations (pH adjusted to approximately 8) were used, with and without a sensitizer (2% acetone). Aliquots of the test solution were removed before photolysis was begun, and at regular intervals thereafter (usually 24 hours). Trapping solutions and the Chromosorb 101 traps were changed periodically during photolysis.

Photolysis solutions, carbon dioxide trapping solutions, ethylene glycol trapping solutions, and Chromosorb 101 traps were radioassayed for determinations of carbon-14 concentrations. Solutions were co-chromatographed with known standards on commercial thin layer chromatography (TLC) plates and developed in one of three solvent systems--benzene:methanol, or ethyl ether:petroleum ether:acetic acid, or benzene:hexane.

REPORTED RESULTS:

A. General

Table I presents a summary of the carbon-14 material balance for all experiments. Recovery of carbon-14 exceeded 70% for all experiments, although carbon-14 recovery was consistently lower in the presence of acetone sensitizer.

B. Degradation of Carboxyl-Labeled RH-6201

Sunlamp photolysis of the carboxyl-labeled RH-6201 preparation in the absence of sensitizer failed to degrade the compound after 48 hours of exposure. This was indicated by the lack of labeled carbon dioxide evolution, the constant extractability of carbon-14 from the photolysis

solution by benzene, the slightly higher concentration of RH-6201 in the final photolysis solution than at the start, and the excellent materials balance (101.5% carbon-14). Significant product degradation was observed in the presence of the 2% acetone sensitizer, with the major photodegradative pathway identified as decarboxylation (evidenced by the generation of large amounts of labeled carbon dioxides). A second pathway, hypothesized to produce polar compounds, was indicated by a decreasing extractability of the photolysis solution by benzene.

Blacklight photolysis of the carboxyl-labeled preparation in the absence of sensitizer produced greater degradation than was observed under the sunlamp, with 20% and 55% of the original radioactivity being evolved as labeled carbon dioxide after 48 and 264 hours, respectively.

Photodegradative pathways were considered the same as those observed with the sunlamp photolysis with acetone. Blacklight photolysis in the presence of acetone also indicated decarboxylation as the major photodegradative pathway, but data did not indicate the presence of polar photoproducts observed in the previous studies. None of the photolysis experiments with carboxyl-labeled RH-6201 showed significant levels of carbon-14 in either the ethylene glycol or the Chromosorb 101 traps.

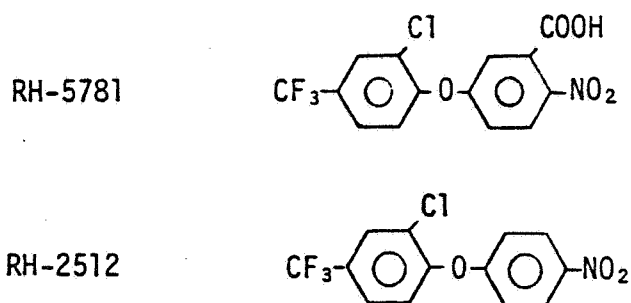
C. Photolysis of Trifluoromethyl Group Labeled RH-6201

Only blacklight photolysis studies in the presence of sensitizer were conducted with the trifluoromethyl-labeled preparation. Data from these two studies (which differed in length of exposure, 215 vs. 264 hours) showed a much smaller amount of labeled carbon dioxide evolution, representing 10% of the original radioactivity. As was the case for the previous studies (with the exception of the sensitized blacklight experiment discussed above), the formation of polar, water-soluble photoproducts was evidenced by the decrease of benzene extractability of aqueous photolysis subsamples. As opposed to the previous studies, however, the trifluoromethyl carbon-14 labeled preparations showed approximately 10% of the total radioactivity in the Chromosorb 101 traps.

D. Identification of Photoproducts

TLC and radioautography of photolysis subsamples of the carboxyl-labeled RH-6201 preparation demonstrated that RH-5781, the free acid form of RH-6201, (see below) was the only radioactive photoproduct. TLC and radioautography

of benzene extracts of the final photolysis solution of trifluoromethyl-labeled RH-6201 showed the presence of as many as eight photoproducts, only one of which was identified. This photoproduct was identified as 2-chloro-1-(4-nitrophenoxyl)-4-trifluoromethylbenzene (RH-2512; see below) and represented approximately 2% of the radioactivity in the benzene extract of the final photolysis solution. The remaining unidentified photoproducts were presumed to be derivatives of RH-2512, and all but one of these unidentified products represented approximately 2% or less of the total radioactivity applied to the TLC plate (one unidentified photoproduct accounted for approximately 7% of the radioactivity). Compound RH-2512 was also found to represent over 80% of the photoproduct collected on the Chromosorb traps in the second trifluoromethyl-labeled experiment.



E. Half-Life Calculations

Table I presents the half-life estimates for the various photolysis experiments. The average half-life of labeled RH-6201 under blacklight photolysis was approximately 139 hours. The sensitizer was not observed to affect the degradation under blacklight conditions, but it greatly increased photodegradation under sunlamp illumination. Photolysis reactions were considered to follow first-order kinetics.

DISCUSSION:

1. This photolysis study used two preparations of RH-6201, one labeled with carbon-14 at the trifluoromethyl carbon and the other at the carboxyl carbon. Additional preparations with carbon-14 labeling of the benzene rings would have been desirable in the reviewer's opinion if further evaluation of degradation products and pathways had been attempted.

2. Material balances in the presence of the sensitizer acetone, which included four of six studies with RH-6201, ranged from approximately 71% to 89%. This recovery was not impressive in the opinion of the reviewer and no explanation was offered as to why the acetone sensitizer lowered the carbon-14 recovery.
3. The reasoning behind the overall study approach and design is unclear. It is uncertain to the reviewer, for example, why some experiments were halted at 48 hours while others proceeded for 264 hours; or why blacklight photolysis studies were not conducted with the trifluoromethyl group labeled RH-6201 preparation in the absence of sensitizer, especially in view of the observation that sunlamp photolysis of the carboxyl-labeled RH-6201 preparation failed to degrade the compound in the absence of sensitizer.
4. Analytical procedures, including thin layer chromatography, radioautography, and radioassays, as well as extraction procedures are considered appropriate, although, as stated above, the low recovery of carbon-14 in the material balances in experiments with the acetone sensitizer may indicate a methodological problem.
5. The lack of polar, water-soluble photoproducts in acetone-sensitized blacklight photolysis of carboxyl-labeled RH-6201 (products that were observed in other experiments) was noted but no attempts were made to explain or to further investigate this difference in findings between experiments. However, it is noted that such an investigation would be beyond the scope of this study.
6. Seven unidentified photoproducts from the blacklight photolysis of trifluoromethyl group labeled RH-6201 in the presence of sensitizer were hypothesized to be derived from RH-2512. Identification of these products would not be required under the EPA guidelines, as none exceeded 10% of the pesticide.
7. The apparent lack of dark control samples is a serious drawback, certainly limiting the credibility of conclusions based on these data.

Acifluorfen

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CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Peirson, W.M., and J.D. Fisher (1978). Long-Term Laboratory Photolysis Study of RH-6201 on Soil (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-78-8)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M. Edwards, Environmental Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 15, 1979

TEST TYPE: Long-Term Laboratory Photolysis Study on Soil

CONCLUSIONS:

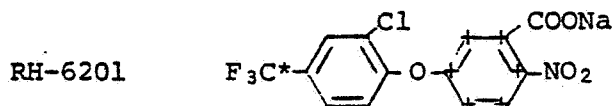
1. RH-6201 is photochemically degraded during 30 days of irradiation, with cleavage of the diphenyl ether bond being a principal route of decomposition. This conclusion is supported by the submitted data.
2. The authors concluded that photolysis, along with microbial degradation (a previous study), would prevent the accumulation of RH-6201 residues in the soil environment. However, studies under actual field conditions and a comparison of the effectiveness of microbial degradation vs. photodegradation appear to be in order to confirm the authors' conclusion. The calculated half-life of RH-6201 under the conditions of this study (57 days), where photodegradation is greatest, is not an especially short period. The results only indicate that photodegradation can play some role (but in reality a potentially small role) in preventing accumulation of RH-6201 residues in the soil.
3. No attempts beyond cochromatography with known standards were made to identify individual soil-extract photoproducts. These photoproducts were less than 10% of the initial herbicide application, and therefore identification would not be required under EPA's registration guidelines. The identification of the volatile photoproduct of the trifluoromethyl-labeled RH-6201 as RH-34800 supports the suggestion that cleavage of the diphenyl ether bond occurs.

-2-

4. This study, as described, is scientifically sound, and was obviously designed to meet the data requirements for photodegradation in support of pesticide registration. The submitted data and experimental design are sufficient to meet these requirements. Information on the design includes light source, intensity, wavelength distributions, and periods of exposure, while the data includes a materials balance and half-life estimate.

MATERIALS AND METHODS:

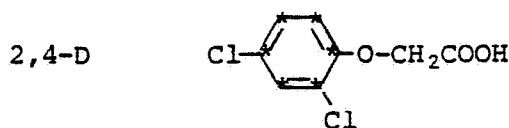
Two preparations of the experimental herbicide RH-6201 and a reference compound, 2,4-D (2,4-Dichlorophenoxyacetic acid), were used. One preparation of RH-6201 was labeled with carbon-14 in the trifluoromethyl group and the other was labeled uniformly in the nitrophenyl ring. The reference compound was uniformly ring labeled.



*Site of trifluoromethyl ^{14}C label

+ Sites of nitrophenyl ring label

Sodium 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoate



*Site of ^{14}C label

2,4-Dichlorophenoxyacetic acid

Fifteen-gram samples of Lawrenceville silt loam soil (pH = 6.9; CEC = 15.0; organic matter = 3.6%; sand = 44%; silt = 46%; clay = 10%) were moistened to the point of clumping and placed in each of 15 borosilicate glass tissue culture flasks. Aqueous solutions of the test compounds were added to the soil surface to provide a level of 5 ppm. Five flasks were prepared for each compound, and designated 3, 10, 20, 30 day and 30-day dark control (wrapped in aluminum foil). Three additional flasks were prepared in the same manner to provide day-0 samples for each compound.

Figure 1 shows the experimental set-up for the prepared flasks. Organic volatiles evolved during photolysis were trapped on segments of Chromosorb^R 101 beads; carbon dioxide was trapped by a 2-methoxyethanol/ethanolamine solution. The flasks were connected to a source of humidified, carbon dioxide-free air, and the atmosphere of the flasks was purged (through the Chromosorb and carbon dioxide traps) for a total of four minutes each hour. Photolysis units consisted of three 40-watt fluorescent sunlamps (Westinghouse FS40) alternating with three 40-watt fluorescent blacklights (General Electric F40BL). The lower surface of the lamps was 4 cm above the flasks, with the average intensity measured at 4.3 watts/m². No significant light was detected below 280 nm or above 500 nm.

Chromosorb and carbon dioxide traps were radioassayed at termination and at 10 and 20 days, and then replaced (for continuing flasks). Radioassays were conducted using liquid scintillation. All samples were counted for a minimum of 10 minutes, and counting efficiencies determined by the channels-ratio method. Soil from all the terminated flasks was extracted, and the extracts and soil radioassayed by liquid scintillation.

Possible degradation products of RH-6201 were investigated by spotting concentrated soil extracts on silica gel TLC plates. Extracts were also cochromatographed against standards to qualitatively evaluate the degradation products. Solvent systems used were hexane/toluene, ethyl ether/petroleum ether/acetic acid, ethyl acetate/isopropanol/water/acetic acid, and isopropanol. Quantification of degradation products was accomplished by autoradiographic location, extraction, and liquid scintillation spectrometry.

REPORTED RESULTS:

A. Radioassay of All Volatiles

The greatest evolution rate of volatile products was observed in photolyzed samples of trifluoromethyl-labeled RH-6201, where labeled volatiles reached a maximum of 6.7% of the original carbon-14 spike in the 20-day sample. Almost no (0.03% maximum) labeled volatiles were found in the nitrophenyl-ring-labeled RH-6201 preparation.

The photolyzed reference compound (2,4-D) reached its maximum rate of evolution of labeled volatiles by the tenth day and a maximum total (4.2%) in the 20-day sample. The carbon dioxide evolution rate generally increased with time, from both photolyzed and dark (control) samples. After 30 days of exposure, labeled carbon dioxide levels reached a maximum of 1.4% and 0.89% for trifluoromethyl and nitrophenyl-ring-labeled RH-6201, respectively. Dark control samples reached 1% and 2.3% for trifluoromethyl-labeled and nitrophenyl-ring-labeled RH-6201, respectively; this was considered indicative of microbial degradation of the test compound.

B. Soil Extraction Results

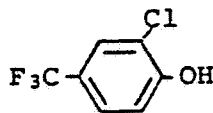
The data showed that the amount of labeled carbon-14 remaining in the soil after extraction generally increased with the duration of photolysis. Radioactivity was found predominantly in the soil extracts, never falling below 81% of the original amount. The distribution of all accountable radioactivity (materials balance) is presented in Table 1. Total accountability ranged from 92 to 103% of the original amount of labeled RH-6201.

C. Thin Layer Chromatography (TLC)

Characterization of the soil extracts showed that the parent compound (the free acid form of RH-6201) declined from 93% at day-0 to 82% at day 20 for trifluoromethyl-labeled RH-6201, and from 87% to 67% for nitrophenyl-ring-labeled RH-6201 (after 20 days). This decline in the parent compound corresponded to increases in polar material. Identification of individual soil extract photoproducts beyond co-chromatography was not considered possible, because of the small amounts available (less than 10% of application).

D. Identification of Volatile Product of RH-6201

Identification of the volatile photoproduct of the trifluoromethyl-labeled RH-6201 that appeared in the Chromosorb volatile trap was accomplished by using a larger sample of trifluoromethyl-labeled RH-6201 (50 ppm) and replacing the Chromosorb trap with XAD-4 resin. After five days of photolysis, the evolved organic volatiles were quantitatively examined by gas-liquid chromatography and mass spectrometry. The data showed that the volatile extract had a retention time similar to RH-34800 (see below), and the mass spectrum of the volatile trifluoromethyl-labeled photoproduct matched the RH-34800 spectrum.



RH-34800

E. Half-Life of RH-6201

The half-life of RH-6201 was determined from all the available data (Table 2): (1) the trapping, which revealed the total volatile labeled products, and (2) the TLC data which revealed the levels of RH-6201 and each product in the soil extract. Computer-generated plots based on these data (Figure 2) showed a half-life of 57 days for the average of both RH-6201 preparations.

DISCUSSION:

1. This study used two preparations of RH-6201 labeled with carbon-14 at two different positions on the molecule (in the trifluoromethyl group and uniformly in the nitrophenyl ring). Labeling at these positions appears to be sufficient for the study. The small quantity of individual soil-extract photoproducts available (less than 10% of the initial application) prevented in-depth studies for identification beyond cochromatography with known standards. In the reviewer's opinion if extensive identification procedures had been undertaken, then a third preparation of RH-6201 with carbon-14 labeling of the ring containing the trifluoromethyl group would have been desirable.
2. Experimental procedures and protocols, including radioassays, TLC, mass spectroscopy, and extraction/cleanup procedures are adequate. Cochromatography and comparisons of spectra with known compounds give support to the identification of the carbon-14 containing photoproduct as RH-34800. Accountability of radioactivity (Table 1) is impressive and indicative of good analytical techniques.
3. Disappearance of the parent compound (the free acid form of RH-6201) in the soil extracts, over time, resulted in increases in polar material and unknowns. Identification of these products was not attempted beyond cochromatography with known standards, and is not required under the EPA guidelines for registering pesticides (being less than 10% of the herbicide).
4. Hypotheses generated by this study were supported by the data, but in general would require further study for verification in the opinion of the reviewer. This includes the suggestion that microbial degrada-

tion was responsible for labeled carbon dioxide evolution in dark (control) samples, and that cleavage of the diphenyl ether linkage was a principal route of degradation.

5. The study was conducted with a single soil type and in the absence of any sensitizers (beyond those that would occur naturally in the soil solution). Temperature conditions were not described, but should not be a critical factor in photodegradation under these laboratory conditions.

Acifluorfen

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CHEMICAL: Experimental RH-2512 (similar to Blazer)

FORMULATION:

CITATION: Spillner, C.J. (1974). The Photodegradation of RH-2512 on Silica Gel Thin Layer Chromatography Plates (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 3923-74-42).

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M.J. Edwards, Environmental Scientist,
Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 2, 1979

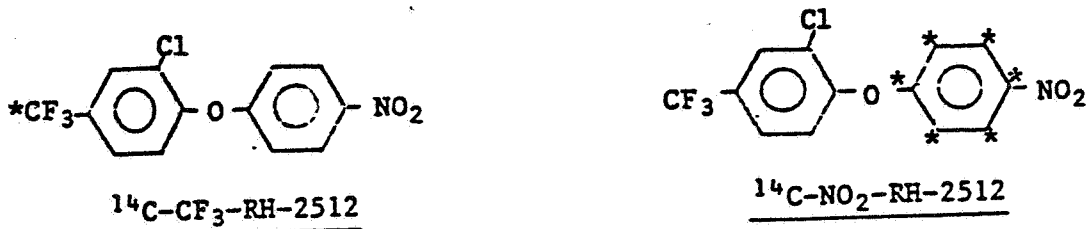
TEST TYPE: Photodegradation: Silica Gel

CONCLUSIONS:

1. Data presented in this study indicate that RH-2512 undergoes rapid photodegradation on silica gel TLC plates, with half-lives of approximately 8 and 16 hours for direct and triplet-sensitized photolysis, respectively.
2. Identified products of photodecomposition (phenols) indicate that cleavage of the ether bond occurs. However, the major photoproduct remains to be identified. Attempts to identify the major photoproduct were undertaken in this study, but were unsuccessful.
3. An unproven hypothesis was put forth that photo-induced nucleophilic substitution by $-SiOH$ is the major degradative pathway.
4. The author concluded that the data indicate the decomposition rate of RH-2512 will not be affected by the presence of natural or synthetic sensitizers. This conclusion does not appear to be well substantiated in the reviewer's opinion, because the photosensitizer anthraquinone retarded the decomposition of RH-2512 (twofold), and no further testing with other sensitizers was conducted.

MATERIALS AND METHODS:

Studies were conducted using two preparations of the test compound (2-chloro-4-trifluoromethyl-4'-nitro diphenyl ether) labeled at different sites with carbon-14. One preparation had the carbon-14 label distributed in the nitrophenyl ring, designated nitrophenyl-labeled RH-2512, and the second preparation had the label in the trifluoromethyl group, and was designated trifluoromethyl-labeled RH-2512:



TLC plates precoated with 0.25 mm silica gel 60-F254 were used. Reagent-grade solvents were used directly from the commercial containers.

Photolysis experiments were conducted under UV lights (three 20-W Westinghouse FS20 fluorescent sunlamps and two 20-W Westinghouse F20T12/BL blacklights), on a rotating disc 7 cm below the lights. Direct photolysis on the silica gel TLC plates using nitrophenyl-labeled RH-2512 (3.9 μg , 0.12 μmole) and trifluoromethyl RH-2512 (5.5 μg , 0.17 μmole) were conducted for 0, 1, 2, 3, 4, 8 and 16 hours. Sensitized photolysis studies were conducted on the same plates, with the test preparations superimposed over a spot of anthraquinone (50 μg , 24 μmole). Product studies were conducted in a similar manner, but exposures were limited to 4 hours. Control plates were prepared in a manner identical to those above, wrapped in foil, and exposed to the UV light for 24 hours.

Exposed plates were spotted with reference compounds, developed in the appropriate solvent and analyzed by radiochemical methods. Two solvent systems, A and B, were used. A was hexane/benzene and B was acetone/benzene. Development was in an unsaturated chamber by the ascending technique to 10-12 cm from the origin. Radioactive spots were determined by radioautography, and TLC spots of standard compounds were located under short-wavelength UV light.

Quantitation of the plates was conducted by scraping the radioactive areas of the silica gel into liquid scintillation vials, and counted by liquid scintillation spectrometry. Background counts were determined using silica gel in the elution zones not containing products. Relative percentages of photoproducts were determined directly from the counts per minute (cpm).

REPORTED RESULTS:

A. Kinetic Studies

Figure 1 (nitrophenyl-labeled RH-2512) and Figure 2 (trifluoromethyl-labeled RH-2512) present the degradation profiles from direct and sensitized photolysis of the two preparations. Sensitized decomposition was significantly slower than direct, with half-lives of approximately 16 and 8 hours for sensitized and direct photolysis, respectively. Loss of approximately 20% of the carbon-14 activity after 16 hours was considered to result from volatility of the parent compound and/or photoproducts.

B. Product Studies

Tables 1 and 2 indicate the formation of at least 10 photodegradation products over the four hours of irradiation, for nitrophenyl-labeled RH-2512 and trifluoromethyl-labeled RH-2512, respectively. The same products were formed in direct and in sensitized photolysis. Cleavage of the ether bond was indicated by observations that photoproduct was unique to trifluoromethyl-labeled RH-2512, whereas photoproducts 5 and 8 were unique to nitrophenyl-labeled RH-2512.

Attempts to identify the products of decomposition were made by TLC comparisons with several compounds considered as likely products, and by further studies with two-dimensional TLC. Identified products were:

Photoproduct 4 = 2-chloro-4-trifluoromethylphenol
Photoproduct 8 = 4-nitrophenol
Photoproduct 2 = RH-2512
Photoproduct 9 = 2-chloro-4-trifluoromethyl-4'-acetamido-diphenyl ether (tentative)

-4-

The major decomposition product was observed to remain at the origin, although there was significantly less photoproduct remaining at the origin from trifluoromethyl-labeled RH-2512 than from nitrophenyl-labeled RH-2512. Attempts were made to identify the photoproducts remaining at the origin through elution with numerous solvent systems and through measurements of the percentage of bound carbon-14 residues at the origin. These latter measurements indicated that the photoproducts remaining at the origin consisted of 3% RH-2512, 2% photoproduct 9, 40% unidentified polar products, and 54% silica-bound material.

DISCUSSION:

1. These experiments on the photodegradation of RH-2512 were conducted using two preparations of the compound, differing with respect to the location of the carbon-14 label on the molecule. A third preparation, with the label on the ring containing the trifluoromethyl group might have been useful in the opinion of the reviewer, especially in providing information on unidentified degradation products.
2. Volatilization of the parent compound or photoproducts was considered to be responsible for the loss of approximately 20% of radioactivity over 16 hours. However, attempts to better define this possibility by comparing losses from irradiated covered and uncovered plates were inconclusive, as there were equal losses from both plates.
3. Discrepancies between the photodegradation rates of nitrophenyl-labeled RH-2512 and trifluoromethyl-labeled RH-2512 were attributed to the larger sample size and increased density of the spot of the latter preparation. This hypothesis was reasonable, but no tests were conducted to confirm or deny this.
4. Initial rates of photodegradation (hours 1-8) approximated first-order kinetics, but observations at 16 hours were removed from this first-order curve. The paper stated that this reflects the unreacted deposit of parent compound and perhaps also unresolved photoproducts. This former belief was not supported by any experimental evidence (which could have been provided by using a smaller sample size); an alternative explanation is that photoproducts were interfering with degradation of the parent compound.
5. The reaction products were considered to support the hypothesis that photodegradation proceeded by cleavage of the ether bond, a step considered essential for complete degradation of the compound. The major photoproduct found, a bound residue at the chromatographic origin, was hypothesized to result from the photoinduced nucleophilic substitution by $-\text{SiOH}$. These hypotheses require further investigation, but based on the study and literature cited, appear to be sound.

Acifluorfen

Page _____ is not included in this copy.

Pages 33 through 36 are not included in this copy.

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- _____ Identity of product inert ingredients.
- _____ Identity of product inert impurities.
- _____ Description of the product manufacturing process.
- _____ Description of product quality control procedures.
- _____ Identity of the source of product ingredients.
- _____ Sales or other commercial/financial information.
- _____ A draft product label.
- _____ The product confidential statement of formula.
- _____ Information about a pending registration action
- X FIFRA registration data.
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- _____ The document is not responsive to the request.

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

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Caplan, J., and K. Schroeder. (1978).
¹⁴C-RH-6201 Activated Sludge Metabolism (unpublished report prepared by Biospherics Inc. for Rohm and Haas Co., Philadelphia, Pa., Laboratory Report No. 7PL-1-1)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M. Edwards, Environmental Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 9, 1979

TEST TYPE: Activated Sludge Metabolism Study

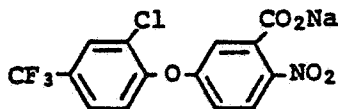
CONCLUSIONS:

1. In this study the authors concluded that RH-6201 exhibited no effects on activated sludge microbial populations, and that little or no degradation of RH-6201 occurred in this activated sludge system.
2. Most added RH-6201 (over 90%) remained in the supernatant, unadsorbed to the solids. Minor amounts (less than 0.5%) of labeled carbon dioxide were evolved, and even lower amounts of carbon-14 volatiles were detected.
3. Except for some reservations noted in the discussion section regarding the nature of the activated sludge, the study was scientifically sound in the opinion of the reviewer, and was obviously designed to satisfy EPA's data requirements for activated sludge metabolism studies. The study succeeds in satisfying these requirements.

MATERIALS AND METHODS:

Two preparations of the test compound RH-6201 were used, one labeled with carbon-14 in the trifluoromethyl group and the other with the label in the nitrophenyl ring. Radiopurities of both preparations were 100% as determined by thin layer chromatography and autoradiography.

-2-



RH-6201

Sodium-5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoate

Activated sludge was obtained from a facility treating primarily industrial wastes (Back River Sewage Treatment Plant, Dundalk, Maryland). Suspended solids were adjusted to 2.5 g/l. Aeration chambers (operating volume = 2,100 ml) were each charged with 700 ml of the activated sludge suspension and 1,400 ml of synthetic sewage solution (nutrient). The activated sludge, nutrient solution, and labeled RH-6201 preparations were aerated for 23 hours per cycle. After each cycle aeration was halted to allow the settling, and 1,400 ml of supernatant were then removed and replaced with fresh nutrient solution and increasing amounts of test compounds (see appended schedule). Prior to discontinuing aeration, suspension samples were taken for plate counts, suspended solids, and to obtain solids and supernatant samples for radiocarbon analysis.

Samples of the suspension taken during aeration were serially diluted, and aliquots of these dilutions were plated on various growth media to determine the number of organisms (bacteria, actinomycetes, and yeasts) per ml of sludge suspension. Sludge solids from the filtration of 100 ml suspensions from each cycle were combusted, and the filtrates and trapping solutions (for volatiles and carbon dioxide) counted for carbon-14 activity by liquid scintillation spectrometry. Filtrates from cycle 10 were acidified, and partitioned by thin layer chromatography (TLC), along with preparations of RH-6201 as standards. Plates were developed in benzene/methanol. Radioactive spots on the developed plates were located by radioautography, and the spots isolated and quantitated by liquid scintillation spectrometry.

REPORTED RESULTS:

A. Effects on Microbial Population

Total plate counts on nutrient agar showed no variation between treated and control flasks, and there was also no variation in actinomycetes counts (on Bacto Actinomycetes Isolation Agar). An increase in yeast (plated on Bacto YM Agar) in treated flasks was attributed to acid conditions on cycle 8 of the treated flasks. Little variation in plate counts was observed in cycle 18-20, when addition of the test compounds was halted.

-3-

B. Carbon-14 Distribution

The study showed that more than 90% of the added labeled RH-6201 remained in the supernatant and was not adsorbed to the solids. This was true for both carbon-14 preparations of the compound. According to the authors, an increase in carbon-14 activity in the solids in cycle 8, when the pH was low, was apparently due to adsorption of the test compound to the solids under acidic conditions. Flasks containing the carbon-14 labeled parent compounds showed little or no evolution of carbon-14 volatiles (from a dry ice-acetone volatiles trap). Evolution of labeled carbon dioxide from flasks containing the trifluoromethyl-labeled compound was approximately 0.5%, and was lower from flasks containing the carbon-14 label in the nitrophenyl ring.

C. Product Identification

Extracts of the supernatants were cochromatographed on TLC plates with preparations of the parent compound. No degradation components were found. The only compound identified in the supernatant was RH-6201.

DISCUSSION:

1. In the reviewer's opinion, the two carbon-14 labeled preparations of RH-6201 appear sufficient for this study.
2. The supernatant (which accounted for more than 90% of added labeled RH-6201) was examined by TLC for the presence of degradation products, but no attempts were made to assay for degradation products that might have been adsorbed to the solids. Even though this fraction would have contained a minor amount of the added carbon-14, the presence of degradation products adsorbed to the solids would be an important finding in attempting to evaluate the potential long-term effects of the compound on an activated sludge system.
3. Physical measurements (pH, temperature, dissolved oxygen, and suspended solids) failed to show any effect of labeled RH-6201 on the activated sludge system. This was confirmed by plate counts for total organisms, yeasts, and actinomycetes.
4. A decreasing pH in the treated flasks at Cycle 8 was noted and this contributed to a rise in yeast populations in the treated flasks, and to an increase in adsorption of the test compounds to the solids as a result of acidic conditions. The low pH was a reasonable explanation for these observations, but some simple additional testing should have been considered to confirm this belief.

-4-

5. Little or no volatile carbon-14 was observed for either RH-6201 preparation, although the data indicate that measurements for these volatiles were not initiated until the 6th cycle or later. This suggests the possibility that some volatiles were evolved in the early stages of the study, but not detected.
6. Experimental protocols and procedures appear satisfactory, although in the reviewer's opinion, some procedures could have been more fully described, such as the method of carbon-14 analysis following combustion of the sludge solids. More information on the nature of the activated sludge used in this study would be desirable, especially in light of the fact that the sludge was obtained from a plant that treats mainly industrial, as opposed to municipal wastes. The high average carbon-14 recoveries, exceeding 92%, indicate good analytical and extraction techniques.

Schedule for Additions for Labeled RH-6201 to Activated Sludge System

Cycle	RH6201 Nominal Concentration in the System (ppm)	Added Amount of Labeled RH-6201 (a.i.) ^a	
		Weight (mg)	dpm x 10 ⁻³
1	0.1	0.21	81.6
2	0.5	1.05	407.4
3	1.0	2.10	814.8
4	5.0	10.5	4,080
5	10.0	21.0	8,158
6	20.0	42.0	16,317
7	40.0	84.0	32,634
8	60.0	126.0	48,951
9	80.0	168.0	65,268
10	100.0	210.0	81,585
11	100.0	210.0	-- ^b
12	100.0	210.0	--
13	100.0	210.0	--
14	100.0	210.0	--
15	100.0	210.0	--
16	100.0	210.0	--
17	100.0	210.0	--
18	0.0	0.0	
19	0.0	0.0	
20	0.0	0.0	

^a Labeled RH-6201 specific activity adjusted to 388 dpm/g for each label position

^b Labeled RH-6201 used in cycles 1-10 and nonradioactive RH-6201 used in cycles 11-17

CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Fisher, J.D., and W.M. Pierson. (1976). RH-6201 Laboratory Soil Metabolism Study (unpublished report for Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-18)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: David F. Arieti, Staff Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 10, 1979

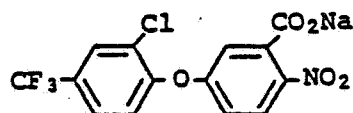
TEST TYPE: Soil Metabolism

CONCLUSION:

Results indicate that RH-6201 degrades in sandy loam and silty loam soils at 1 ppm; however, at 10 ppm, metabolism of RH-6201 is reduced by microbial activity.

The objectives of qualitative and quantitative fate of RH-6201 in soil were met.

MATERIALS AND METHODS:



RH-6201

Sandy loam and silty loam soils (for characteristics see Table I) and sterilized soil were treated at 1 and 10 ppm with labeled RH-6201. RH-6201 was labeled at the trifluoromethyl group for one batch, and the other batch was labeled at the carboxyl group. Ring-labeled 2,4-D was used as a reference compound. Aerobic flasks were connected in series with an oxygen line. Anaerobic flasks were maintained by alternately evacuating the flasks and introducing pure nitrogen.

Total carbon dioxide was measured by titration, while trapped carbon dioxide was measured by counting aliquots in a scintillation counter.

Aerobic and anaerobic soil samples were taken on days 10, 15, 30, 60, 107, and 180 (Table III) or 182 (Tables V onward, probably correct). Sterilized soils were sampled only until day 60. Samples were analyzed for carbon-14.

-2-

Characterization of breakdown products was made by extraction and thin layer chromatography.

Bound residues were characterized by soxhlet extraction and thin layer chromatography.

REPORTED RESULTS:

A. Effects on Total Soil Microbial Activity

Results from the nonsterilized samples (Table III) indicate that most of the carbon dioxide was liberated by day 10 (silt loam) or by day 15 (sandy loam). Recoveries diminished sharply after day 15 and then steadily until the last sampling. Recoveries from sterilized samples were insignificant (Table IV).

These data suggest that soil microorganisms were not inhibited significantly by RH-6201 under these conditions.

B. Soil Residues (Tables V and VI)

Degradation of carboxyl- or trifluoromethyl-labeled RH-6201 and of 2,4-D was measured in sandy or silt loam soil samples after treatment at 1 or 10 ppm. Degradation of RH-6201 at 10 ppm was insignificant in either soil type. Values for carboxyl-labeled RH-2601 at 1 ppm were halved after 182 days in either soil but were unchanged after 60 days in sterilized soil, so far as one can tell from the data (Table V). Values for trifluoromethyl-labeled RH-6201 at 1 ppm were probably diminished but questions of margins-of-error and significance remain. Values for 2,4-D were diminished at both levels in both soils. Again, sterilized samples showed no apparent change.

The anaerobic samples indicated a different picture. RH-6201 residues increased in both soils after 10-ppm treatment but remained constant after 1-ppm treatment. 2,4-D remained constant in either soil after either treatment, so far as one can tell from the data (Table VI). All three compounds diminished promptly in water except 2,4-D at 1 ppm which diminished rather slowly.

C. Extraction of Soil Metabolites

Identification of possible metabolites is given in Table XVI.

- 1) Aerobic Soils (Refer to Table XI): For sandy loam soil, it appears that the acid form of RH-6201, which is coded RH-5781, is the major compound. Results were similar for carboxyl- and trifluoromethyl-labeled RH-6201 in both soil types.
- 2) Anaerobic Soils (Refer to Table XIII): For carboxyl-labeled RH-6201 the most abundant metabolite was RH-5781 at 81.2% initially, but this dropped to 4.8% at day 182. The most abundant metabolite at day 182 was RH-4514.

-3-

For trifluoromethyl-labeled RH-6201, results were similar. At day 0 RH-5781 was 76.8%, and at day 182 it was 7.4%. The metabolite RH-4514 increased from 2.3% at day 0 to 20.6% at day 182.

3) Sterilized Soils (Refer to Table XIV): The major component was RH-5781.

D. Half-Life Determination (Table XXIV)

The authors concluded that the half-life of RH-6201 was 30 days in sandy soil and 60 days in silty soil under aerobic conditions.

E. Characterization of Soil-Bound Residues

Results of sequential extractions of aerobic and anaerobic soils are summarized in Table XVII. In aerobic soils, most of the radioactivity was removed with the solvents. The authors indicated in the text that about 4-5% and 10% of RH-6201 was tightly bound in aerobic and anaerobic soils, respectively, while the figures were 11-19% for 2,4-D.

DISCUSSION:

1. The authors concluded that RH-6201 would degrade at a rapid rate at both 1 and 10 ppm. In the reviewer's opinion, results do not support this conclusion at 10 ppm. The authors point out that at levels of 10 ppm, microorganisms involved in RH-6201 metabolism were inhibited or that the chemical as a substrate was in excess of what could be utilized by the microbial population.
2. The experiment was conducted in great detail. It seems to be scientifically sound in the reviewer's opinion, and complies with EPA guidelines.

Acifluorfen

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Pages 45 through 56 are not included in this copy.

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- _____ Identity of product inert ingredients.
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- _____ Description of product quality control procedures.
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CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Garstka, T.A. (1978). The Adsorptive and Desorptive Behavior of RH-6201 on Drummer Silt Loam Soil. (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-78-4)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M.C. White, Environmental Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 20, 1979

TEST TYPE: Soil Adsorption and Desorption of a Pesticide

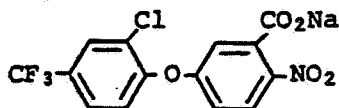
CONCLUSIONS:

1. In this pesticide/soil system, RH-6201's adsorption characteristics are similar to those of 2,4-D, a commonly used model compound. It is adsorbed slightly less than 2,4-D, but desorbs more than 2,4-D.
2. Adsorption of RH-6201 is proportionately greater at lower concentrations than at higher concentrations, indicating that moderate adsorption would occur at the recommended application rate.
3. The samples listed in the raw combustion data do not appear to be related to those listed in the adsorption/desorption sections. Thus, the RH-6201 mass-balance data could not be analyzed unambiguously by the reviewer.
4. In this study, the reported adsorption and desorption coefficients are for intercepts ($C_e = 0$) instead of the standard equilibrium concentration ($C_e = 1$ ppm). However, the coefficients required by EPA guidelines can be easily determined from the reported data. Thus, the protocol and results of this study appear to meet EPA guidelines for pesticide soil adsorption and desorption.

MATERIALS AND METHODS:

Four RH-6201 concentrations (0.1, 1.1, 10, and 123 ppm) were equilibrated (24 hours, 24°C, shaker) with a Drummer silt loam soil (pH 5.2; CEC = 10.1; organic matter = 3.8%; sand = 13%; silt = 65%; clay = 22%) at a four to one solution:soil ratio. RH-6201 was labeled with carbon-14 in the trifluoromethyl group. In addition, 2,4-D ring-labeled with carbon-14 was used as a model compound throughout the study.

-2-



RH-6201

After centrifugation the supernatant was removed and assayed by liquid scintillation counting; counting efficiencies were determined by using the external channels ratio method. Adsorbed RH-6201 was calculated as the difference between initial and final solution concentrations. Following adsorption, each sample was equilibrated with four successive 10-ml portions of distilled water in a shaker bath at 25°C. The desorbing equilibrium times varied between six and 24 hours. After centrifugation the supernatant from each preparation was assayed by liquid scintillation counting. Desorbed RH-6201 was calculated from the difference between the successive residual soil concentrations and that released to the respective equilibrium solutions. After the fourth extraction the soils were combusted to release the remaining carbon-14. Final mass balances were calculated for RH-6201 at each concentration by using the combustion data and the adsorption/desorption data.

Adsorption and desorption isotherms were prepared by using the logarithmic form of the Freundlich equation. The desorption isotherms represent a composite of the four extractions. Adsorption and desorption coefficients (K) were obtained from these graphs at equilibrium solution concentrations (C_e) equal to zero.

REPORTED RESULTS:

RH-6201 was adsorbed and desorbed linearly over the range of equilibrium concentrations (Figures 1 and 3), apparently following the Freundlich relationship.

The proportion of RH-6201 adsorbed to the solid increased with decreasing concentrations (Table VII). Conversely, less RH-6201 was desorbed at the lower concentrations than at the higher concentrations. For example, approximately 89% of the total RH-6201 adsorbed was desorbed at the 123 ppm treatment; whereas only 50% was desorbed at the 0.1 ppm treatment (calculated from Table VII and X).

Total recoverable carbon-14 from adsorption, desorption, and soil combustion, ranged from 92 to 97% for the four RH-6201 concentrations (Table X).

The adsorption and desorption coefficients (K) were determined from logarithmic plots of the Freundlich equation (Table I). Intercept values (antilogs) from RH-6201 were 11.1 and 40.2, respectively. Those for the model compound (2,4-D) were 13.8 and 88.9, respectively.

DISCUSSION:

The experimental procedures used in this study were appropriate for determining the adsorption and desorption behavior of RH-6201 in a typical agricultural soil. However, the combustion data, as presented for carbon-14 mass balance determinations, were confusing to the reviewer. It was not clear if the samples were the same as those used for the adsorption/desorption studies.

Although the reported summary of results stated that the adsorption and desorption coefficients (K) were for $C_e = 1.0$ ppm, this was not the case. Instead the tabular values represent intercept estimates ($C_e = 0$). This is potentially confusing because K values are more appropriately standardized across soils and compounds at equilibrium solution concentrations equal to 1 ppm.

RH-6201's adsorption ($K = 11$) is slightly less than 2,4-D ($K = 14$) for this soil, however considerably more 2,4-D than RH-6201 remains adsorbed after desorption. Therefore, in the reviewer's opinion another model compound would have been helpful in characterizing the complete adsorptive/desorptive behavior of RH-6201. For example, the molecular size of 2,4-D is approximately one-half that of RH-6201. Therefore, preferential movement of 2,4-D into the colloidal clay crystal would be expected, and its desorption would be hindered because of steric rearrangement within the lattice after a wetting and drying cycle. A second model compound would have helped sort out the relative importance of soil organic matter and clay colloids in RH-6201's adsorption and desorption.

FIGURE 1 ADSORPTION⁴⁵²

RH-6201 FREUNDLICH ISOTHERM
DRUMMER SILT LOAM

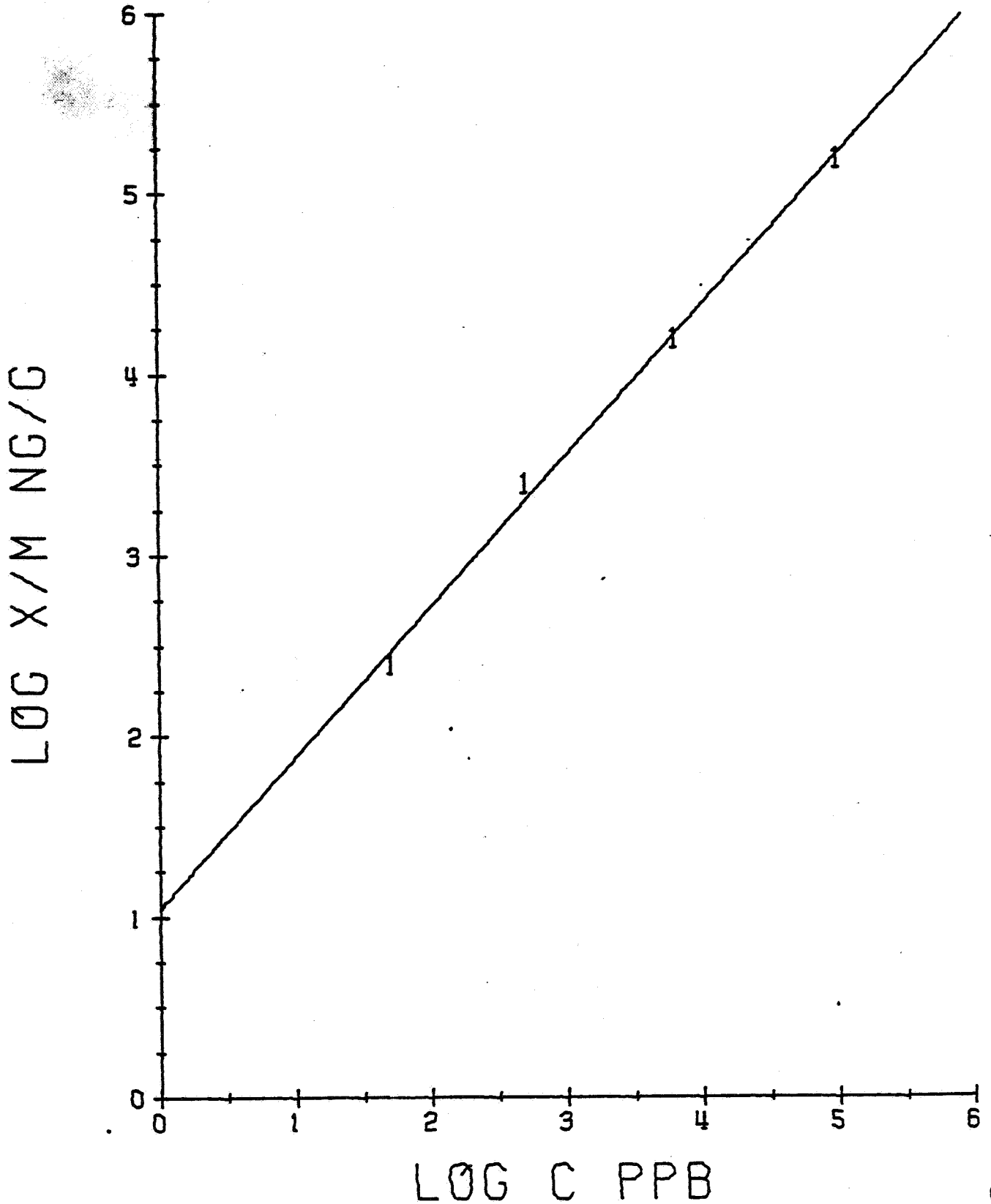
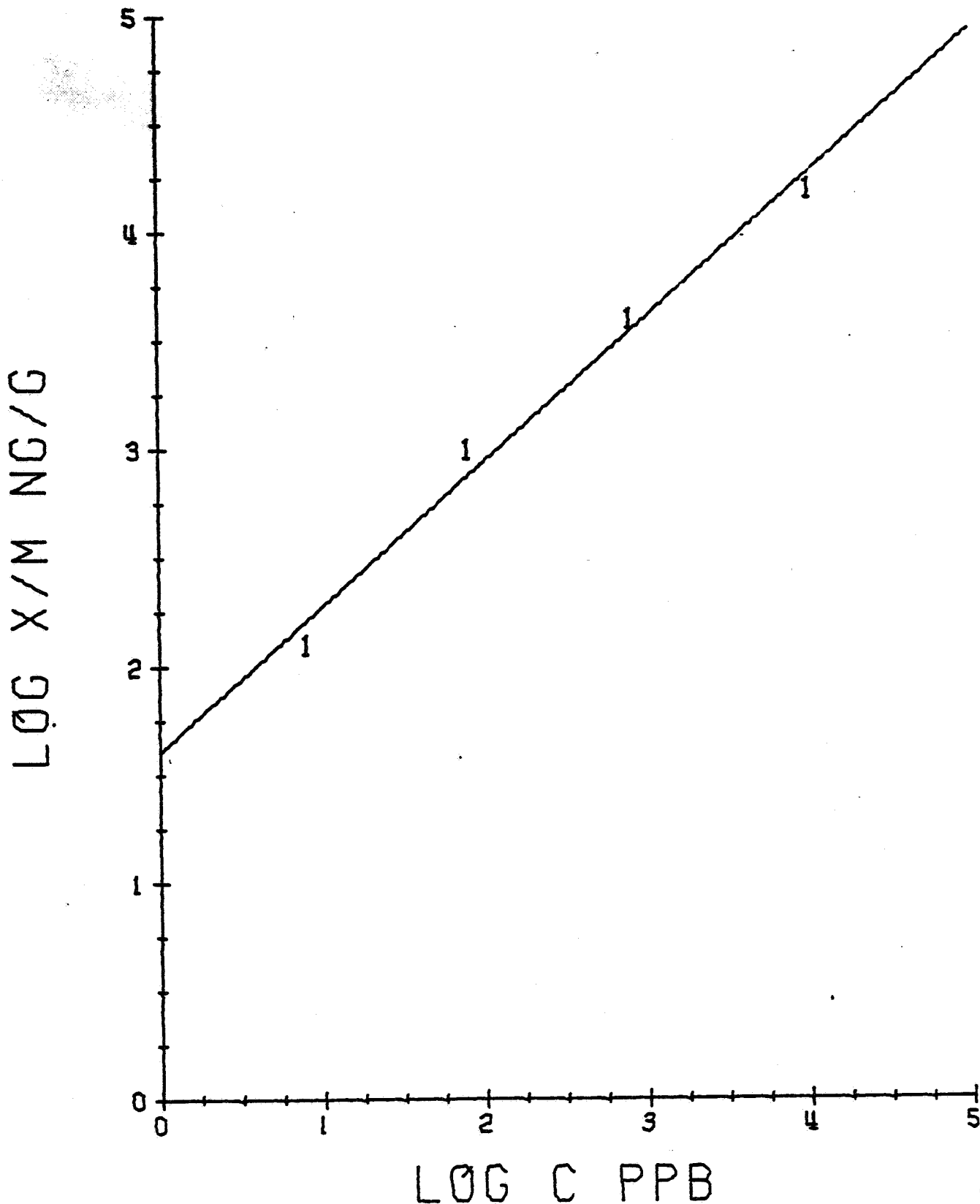


FIGURE 3 DESORPTION RINSES 1-4 454
RH-6201 FREUNDLICH ISOTHERM
DRUMMER SILT LOAM



Acifluorfen

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

CHEMICAL Blazer, sodium salt of acifluorofen, carbofluorofen
(formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Rogerson, T.D. (1978). A Residue Decline Study of
Soil Treated with ¹⁴C-RH-6201 at Newtown, Pa., in
1977 (unpublished report prepared by Rohm and Haas
Co., Philadelphia, Pa., Technical Report No. 34H-78-19)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

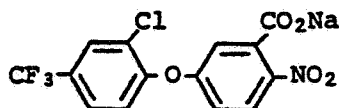
REVIEWED BY: M. Edwards, Environmental Scientist, Enviro Control,
Inc., Rockville, Md.

DATE OF REVIEW: August 17, 1979

TEST TYPE: Residue Decline Study

CONCLUSIONS:

1. The author concluded that the use of RH-6201 as a herbicide will result in low levels of RH-6201-derived residues in the soil at harvest, and that retreatment of the soil each season should not lead to a buildup of such residues. The data support the conclusion that RH-6201-derived residues will decline substantially during the growing season; but, in the reviewer's opinion, it is a subjective judgment on the part of the author to consider the final levels observed (0.12-0.47 ppm in the 0-3 inch soil layer) as being low. It is also the opinion of the reviewer that further study would be necessary prior to concluding that yearly reapplications would not lead to a buildup of soil residues. The data presented in Table I suggest that buildup may occur, since residues in previously treated plots were between two and three times the residues in the previously untreated plot (in the 0-3 inch soil layer) after 110 days.
2. Procedures and protocols in this study were not described well enough to allow the reviewer to conclude that the study was scientifically valid. As presented, the study does not meet EPA's guidelines for reporting data on field studies, with data on precipitation, water table, and grade (slope) not included. In addition, samples for one plot (treated with RH-6201 labeled in the nitrophenyl ring) were not taken immediately after treatment, although this is not seen as a critical omission by the reviewer. The reviewer does not believe that the study succeeded in its objective of determining the nature of total radioactive residues in the treated soils, and the discussion of the chemical residue analysis was weak.

MATERIALS AND METHODS:

RH-6201

Three preparations of RH-6201 were used for this study: one labeled with carbon-14 in the trifluoromethyl group; another labeled in the carboxyl group; and the third uniformly labeled in the nitrophenyl ring. Radiopurity of each preparation exceeded 95%.

Each preparation was dissolved in deionized water (50 ml) and applied to the appropriate plot at a rate equivalent to 1 lb per acre. The plots treated with trifluoromethyl-labeled and carboxyl-labeled preparations had been treated the previous year with the same compound at the same rate of application. The third plot had not been treated previously. Cross-contamination of plots by spray drift during treatment was prevented by covering the remaining plots with polyethylene sheets. Plots were prepared by rototilling and raking. The soil was classified as an Abbottstown silt loam (pH = 7.11; CEC = 1.54 meq/100 g; organic matter = 4.7%; sand = 0.0%; silt = 45.6%; clay = 33.2%).

Soil core samples (0-3, 3-6, and 6-12 inches) were taken five times during the growing season (11, 21, 39, 70, and 110 days after treatment). Pretreatment levels were determined for each plot, and for plots receiving trifluoromethyl-labeled and carboxyl-labeled compounds, values were determined immediately after treatment. Core samples were mixed, and duplicate subsamples were weighed and combusted. Evolved carbon dioxide was trapped and radioassayed by liquid scintillation spectrometry. Efficiencies of combustion, levels of detection (0.001, 0.002, and 0.003 ppm for ring-labeled, carboxyl-labeled and trifluoromethyl-labeled preparations, respectively), and moisture content of soil samples were determined; local climatological data during the test period were presented.

REPORTED RESULTS:

Table I summarizes the radioassay data from the soil-core samples taken before and after treatment (during the growing season). Data from the 0-3 inch core samples were used to calculate half-lives of 28.8, 43.7, and 34.6 days for the nitrophenyl ring -, trifluoromethyl- and carboxyl-labeled compounds, respectively (Figures 1 and 2). Chemical residue analyses of selected soil samples for labeled RH-6201 (Table II) were taken as evidence that labeled RH-6201 rapidly dissipated in the soil.

DISCUSSION:

1. The reviewer considers the three preparations of labeled RH-6201 to be sufficient for the purposes of this study, which were to determine the magnitude, decline rate, and nature of total radioactive residues in soil treated with labeled RH-6201.
2. The data presented in Table I and in Figures 1 and 2 indicate that total radioactive residues declined during the growing season in each treated plot, and that radioactivity in the 3-6 and 6-12 inch samples was low, showing that limited leaching had occurred.
3. Based on the results of chemical residue analysis, the author considered it probable that a portion of the radioactive residue (the unmeasured portion) was associated with humic acid, fulvic acid, and humin fractions of the soil in the form of a bound and/or highly metabolized polar compound. Reasons for this belief were not discussed by the author.
4. In the reviewer's opinion, there was not enough information provided to evaluate the protocols and procedures used in this study. Minimal information was provided on core sampling methods, plot descriptions (slope, drainage, water table) and chemical residue-analysis methods. Rainfall data at the plot sites, if recorded were not included in the report.

Acifluorfen

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CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). Metabolism of ¹⁴C-RH-6201 in Rats, Soybeans and Soil (unpublished report by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-17)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and 2S

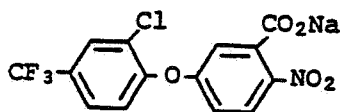
REVIEWED BY: Wan-Shya Chou, Staff Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 20, 1979

TEST TYPE: Field Dissipation Study

CONCLUSIONS:

1. RH-6201 is rapidly metabolized to the amino form (RH-4514) and other unknown compounds in soybean plants.
2. The percentage of bound carbon-14 residues in the soil sample increases as the treatment-to-sampling interval increases.
3. RH-6201 is not metabolized extensively in this soil; more than 70% of the extractable carbon-14 is present as the free acid form (RH-5781) of RH-6201.
4. The results and conclusion reported by the investigator generally meet the very broad objective of this study.
5. This study, in itself, does not meet EPA guidelines for terrestrial field dissipation.

MATERIAL AND METHODS:

RH-6201

Soil, bean, and foliage samples were taken from a labeled RH-6201 residue decline study. The samples contained residues from RH-6201 labeled at either the trifluoromethyl carbon or the carboxyl carbon. The experimental compound was applied to separate soybean plots, either postemergence or preemergence. Soybean foliage was sampled at 14 or 112 days, and the plants were harvested at 112 days. The 0-2 inch layer of soil was sampled at 28, 57 and 112 days after the initial treatment.

Isolation techniques for soybean foliage and beans were as follows:

1. The samples were extracted or digested with either an organic solvent, HCl, NaOH, or a combination of organic solvent and HCl or NaOH.
2. The mixture were filtered after extraction.
3. The aqueous and/or organic phase was subsampled for radioassay.
4. The final organic fraction was concentrated for TLC and radioautographed to identify the metabolites.
5. The filter cake and filter paper were combusted and radioassayed.

Soil samples were extracted with methanol. Aliquots of the extract were assayed for carbon-14 and then concentrated for TLC and radioautography. The methanol-extracted soil was then extracted with 0.5 N NaOH. The supernatant of the mixture was fractionated into fulvic acid and humic acid fractions by using 1 N HCl to precipitate the humic acid. The fulvic acid fraction was extracted with ethyl ether; the solution was concentrated for TCL examination. The activities of the nonextractable and humic acid fractions were determined by combustion and radioassay.

Concentrated solutions were applied to the preabsorbent layer (developed in methanol) of TLC plates and developed in either benzene: methanol; ethyl ether: petroleum ether: acetic acid; or benzene: ethanol.

After the initial TLC and radioautography, radioactive bands of similar Rf values were pooled and rechromatographed for further purification and identification. All filter cakes and filters were combusted. All samples from extraction procedures, TLC plates and combustion were counted in a scintillation spectrometer.

REPORTED RESULTS:A. Soybean Foliage

1. Extractable carbon-14 activity from foliage containing residues from RH-6201 labeled at the trifluoromethyl carbon was always higher than for the foliage treated with RH-6201 labeled at the carboxyl carbon.
2. Seven radioactive metabolites were obtained from the trifluoromethyl-labeled sample. Four radioactive metabolites were obtained from the carboxyl-labeled sample. Of the radioactive bands present, RH-5781 was the confirmed metabolite and the amino form of RH-5781 (RH-4514) was suggested.
3. At final harvest about 30% of the extractable carbon-14 remained as the parent compound (RH-6201). However, fewer metabolites appeared to be present in the final harvested samples.
4. It was postulated that one metabolic pathway for RH-6201 in soybean might involve decarboxylation and incorporation of the carboxyl group into natural products.

B. Beans

1. The amount of carbon-14 extracted from seed containing trifluoromethyl-labeled RH-6201 residues was greater than for carboxyl-labeled residues.
2. RH-5781 and RH-4514, two identified metabolites accounting for 24% of the radioactivity present on the plate, came from plants treated with RH-6201 labeled in the carboxyl group. Three other unknown compounds were also present. One is more polar than RH-5781. Two were less polar than RH-4514.

C. Soils

1. The percentage of bound carbon-14 residues increased as the treatment-to-sampling interval increased (Table VIII).
2. RH-6201 was not extensively metabolized in the soil; more than 70% of the extractable carbon-14 present as labeled RH-5781 (free acid of RH-6201) was in samples collected 57 days after treatment.

3. A radioautograph of a methanol extract of soil collected 57 days after treatment showed more radioactive metabolites in the trifluoromethyl-labeled extract than in the carboxyl group labeled extract. A decarboxylated metabolite was suggested.
4. TLC investigation of the fulvic acid fraction showed that the unaltered parent (RH-5781) was the most prevalent radioactive component in this fraction.

DISCUSSION:

1. According to the EPA guidelines, soil samples for field and vegetable crop uses shall be taken in increments to a depth of 30 cm. However, only the 0-2 inch fractions were used in this study.
2. The reviewer could not confirm the reported results that seven radioactive metabolites came from a trifluoromethyl-labeled sample and four came from a carboxyl-labeled sample, because of the poor quality of the radioautographs presented (Figures 6 and 7). The figures could not be copied, they are the original report.
3. No raw data for the radioassay were presented; the reviewer could therefore not check the results for extractability.
4. Comparison of TLC Rf values was the only identification technique used. Also, the identification of metabolites in soybean was based on one TLC plate.
5. No attempt was made to identify the unknown radioactive metabolites in the fulvic acid fraction from the soil sample.

TABLE VIII

Extractability of ^{14}C -RH-6201 Residues from Soil
(% of ^{14}C in Soil)

Sample Code No: ^{14}C Label Site: Treatment Type: TSI (days) ^a :	75-528-03 CF ₃ Pre-E 28	75-528-04 CF ₃ Pre-E 57	75-528-06 CF ₃ Pre-E 112	75-529-03 COO- Pre-E 28	75-529-04 COO- Pre-E 57	75-529-06 COO- Pre-E 112	75-525-04 CF ₃ Post-E 57	75-525-04 COO- Post-E 57
Methanol Extract	81	56	53	65	65	41	68	65
Total NaOH Extract	11 ^b	26 ^b	26	26	22	29	22	23
Fulvic Acid	7	20	8	13 ^c	10	11	15 ^d	14
Humic Acid	4	6	13	13 ^d	14	18	10	11
Total Bound ^{14}C (Humic)	3 ± 1	19 ± 3	21 ± 3	9 ± 1	18 ± 2	29 ± 7	10 ± 1	12 ± 2

^aTSI = Treatment-to-Sampling Interval

^bThese values were obtained by adding the values found in the fulvic and humic acid fractions.

^cThis value was estimated, based on ^{14}C in total NaOH extract and humic acid fractions. Sample was not radioassayed.

^dThis number is an estimate based on the values for the total NaOH extract and the humic acid. Value obtained from radioassayed was too high, ca. 30%.

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). A Summary of RH-6201 Residue Data for Soybeans and Soil (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-22)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and 2S

REVIEWED BY: David F. Arieti, Staff Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 7, 1979

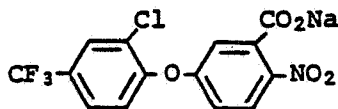
TEST TYPE: Residue Study: Soybean

CONCLUSIONS:

The report objective was to supply residue data needed to establish a temporary tolerance for RH-6201 on soybeans. The title of the report adds residues in soils. In this reviewer's opinion, the data as reported are far from sufficient to achieve either objective. The descriptions of some data are considered to be ambiguous, and other data that are needed and implied cannot be found in the report. This study does not appear to meet EPA guidelines or any accepted standards of validity.

MATERIALS AND METHODS:

Nine soybean cultivars grown on silty loam, clay, and sandy clay loam at 11 locations in the United States were treated preemergence with various doses of RH-6201 and analyzed by methods described in Technical Report No. 34H-76-20.



RH-6201

Recovery factors were calculated from samples enriched with carbon-14 labeled RH-6201 and analyzed for radioactivity.

REPORTED RESULTS:A. Residues in Plants (Table I)

All determinations were 0.0 except for two of 0.010 ppm (1) after RH-6201 had been applied at 1.0 lb per acre, and (2) after no RH-6201 had been applied.

B. Residues in Soil (Table II)

Average determinations after treatment at 1.0 to 4.0 lb per acre ranged from 0.01 to 0.24 ppm.

C. Recoveries from Soybeans (Tables III, IV and V)

Control soybeans were residue-free except for one sample at the detection threshold of 0.01 ppm, mentioned in Table III and in the summary, but not in the body of the report. Corrected (Table V) recoveries from treated soybeans ranged from 56 to 92% (Table IV), but it is not clear that any of these treatments occurred outside the laboratory.

D. Recoveries from Soil (Tables VI, VII and VIII)

No residues were detected in control samples. When control samples were enriched with unstated amounts of labeled or unlabeled RH-6201, recoveries of 61 to 84 additions of "0.4 to 0.36 ppm" (text) or 0.02 to 0.36 ppm (Table VIII) yielded recoveries of 61 to 84% (Table VIII), average 74% (text). Then treated soil residues were corrected to 75% recovery, but the data were not found in the report or tables.

DISCUSSION:

Twenty-one of the 39 determinations in Table I were singles (not replicated), as were 6 of the 12 in Table II, 8 of the 14 in Table III, 9 of the 14 in Table IV, all in Table V, 4 of the 5 in Table VI, 4 of 5 in Table VII, and all in Table VIII. The reliability of the results is therefore uncertain, particularly since no pattern can readily be seen in the Table VI recoveries.

Acifluorfen

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

CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Fisher, J. D. (1978). Leaching and Mobility Studies in Soil Treated with RH-6201 (unpublished report by Rohm and Haas Inc., Philadelphia, Pa., Technical Report No. 34H-78-10)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: David Arieti, Staff Scientist, Enviro Control, Inc., Rockville, Md.

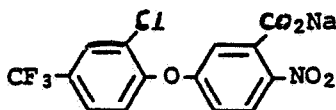
DATE OF REVIEW: August 20, 1979

TEST TYPE: Leaching and Mobility Study

CONCLUSION:

The author concluded that RH-6201 should be classed as moderately mobile but also as a source of persistent residues in soils. These conclusions were extrapolated from observations with soil columns in the laboratory under admittedly extreme conditions. The observations are considered valid so far as they go.

MATERIALS AND METHODS:



RH-6201

In the leaching and mobility study, five types of soil were described (Table I) and four were used. RH-6201 was labeled with carbon-14 either at the trifluoromethyl group or at the nitrophenyl ring. Ring-labeled 2,4-D was used as a reference compound.

Leaching columns were set up for four soil types, and 0.477 mg of labeled RH-6201 was applied to each column, equivalent to 1 lb/acre. Water (2,200 ml) was applied dropwise at a constant rate for 60 hours, equivalent to 20 acre-inches of water. The leachate was collected and radioanalyzed by liquid scintillation spectrometry.

Degradation of RH-6201 was estimated in selected soil and water extracts from serial sections of the columns by TLC and autoradiography, using RH-6201 derivatives (Table VII) 2,4-D and propanil as standards.

REPORTED RESULTS:

A. Leached Soil Residues (Tables II and III)

The column sections yielded radioactivity representing RH-6201 residues ranging between 0.002 ppm for the lowest section of Lakeland soil to 0.092 ppm for the two lowest layers of Pasquotank soil. Recoveries of nitrophenyl ring-labeled RH-6201 tended to be slightly more than of trifluoromethyl-labeled RH-6201. The Pasquotank soil sections yielded more residues than the Lakeland sections. There was a tendency for RH-6201 to collect in the lower column sections. By contrast, 2,4-D residues tended to collect in the upper column sections.

B. Leachate Residues

Radioactivity representing RH-6201 0.009-0.823 ppm was observed in the leachates, with ring-labeled RH-6201 predominating. Differences among soil types appeared insignificant, and residues were concentrated in the middle sections of most columns, both for RH-6201 and for 2,4-D (Tables III and IV).

C. Extraction Recoveries (Table V)

When the radioactivity extracted was compared with the amount unextracted from the top two inches of soil column, from the 6-9 inch section, and from the water, no consistent pattern of results emerged.

D. Thin-Layer Chromatography Tables VII, VIII, IX and X)

Some potential degradation products of RH-6201 were used as standards (Table VII) for TLC of extracts from the top two inches and the 6-9 inch sections of the soil columns. Compounds migrating with RH-5781 and AM 669-1 predominated; traces migrated with RH-883; three unknown compounds were detected in source but not all extracts; and considerable amounts of compounds remained at the origin (Table VIII). In the

Teachates RH-5781 predominated (Table IX). The data from soil extracts appear to be reproducible (Table X).

E. Autoradiographs (Figure 1)

RH-6201 migrated furthest in the sandy soil sample and least in the day soil sample.

DISCUSSION:

1. Soil particle size appears to influence mobility of RH-6201; larger particles, more migration.
2. Added radioactivity was totally recovered within laboratory error ranges.
3. Three possible degradation products were identified tentatively by TLC but no confirmatory studies are reported.

Acifluorfen

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CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Fisher, J.C. (1976), Laboratory Leaching Study with Soil-Aged RH-6201 (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-13)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M. Edwards, Environmental Scientist, Enviro Control, Inc. Rockville, Md.

DATE OF REVIEW: August 14, 1979

TEST TYPE: Laboratory Leaching Study

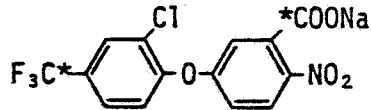
CONCLUSIONS:

1. The author concluded that soil-aged RH-6201 residues are not subject to leaching under extreme laboratory leaching conditions. This conclusion is valid only for residues that contained carbon-14 in the trifluoromethyl or carboxyl group. It is not valid for residues that had lost these groups. For this reason, ring-labeled RH-6201 preparations should have also been used, in the opinion of the reviewer.
2. Explanations of the large amount (approximately 25%) of radioactivity unaccounted for in the combined soil and leachate samples were presented and appeared valid. The research necessary to confirm these explanations, however, was beyond the scope of these experiments.
3. The study, as described, appears scientifically sound and was obviously designed to meet EPA's data requirements for leaching of pesticide residues after aging in soil under aerobic conditions. One deviation from the EPA guidelines was the elution of soil columns with water every weekday as opposed to the recommended daily elution. This could reduce the potential for leaching of the residues, but is not considered a serious deviation by this reviewer. Otherwise, except for the reservations expressed with regard to the lack of ring-labeled RH-6201 preparations, the study appears to meet EPA's guidelines.

MATERIAL AND METHODS:

Two preparations of the experimental herbicide RH-6201 were used, one preparation labeled with carbon-14 in the trifluoromethyl group and the other labeled in the carboxyl group:

-2-



Nixonton sandy loam soil (pH 5.5; CEC 7 meq/100 g; organic matter = 2.59% sand = 71%; silt = 22% and clay = 7%) collected from the top 2-3 inches of a fallow field was air dried, sieved, and moistened to approximately 15% water. Soil (300 g) was fortified to approximately 0.5 ppm with one of the RH-6201 preparations (actual levels were measured as 0.62 and 0.56 ppm for trifluoromethyl and carboxyl label, respectively). Soils were maintained in a greenhouse for 30 days, after which time samples were removed for leaching studies in soil columns, as described below.

Soil columns were filled to a height of approximately 11 inches with Nixonton sandy loam soil, wetted, and overlain with 100 g of each 30-day aged soil preparation. One-half inch of water was added dropwise to each column every weekday for the next 45 days. Leachates were collected daily, and alternate days' collections radioassayed. After 45 days the soil columns were divided into 2-inch segments, and analyzed for radioactivity.

Leachates and subsamples from each soil column segment were analyzed for radioactivity by liquid scintillation spectrometry. Counting efficiencies were determined by the external standard channels ratio (ESCR) method. Limits of detectability for soil samples and leachate samples were 0.002 and 0.001 ppm, respectively.

REPORTED RESULTS:

A. Soil Column Sections

Data from radioassay of soil-column segments taken at the termination of the leachate experiments are summarized in Table II. For both RH-6201 preparations, approximately 70% of the carbon-14 label present in the original aged soil appeared in the top (0-2 inch) segments. No detectable residue was observed beneath the top segment. Radioanalyses of aged soils prior to initiation of soil column studies indicated that total carbon-14 residues had declined approximately 40% during the 30-day aging period.

B. Drainage Water from Columns

Table III summarizes the data from radioanalysis of drainage water leachates, and indicates the absence of radioactivity in all samples.

DISCUSSION:

1. A large amount (approximately 25%) of applied carbon-14 was unaccounted for in the combined soil and leachate radioanalyses. It was considered likely that most of this unaccountable activity was lost through metabolism and/or volatilization during the 45 day leaching period, as indicated by the observation that approximately 40% of the original carbon-14 label added to the soil was lost during the 30-day greenhouse aging period.
2. Neither of the two preparations used were ring labeled, the labels being on the trifluoromethyl and carboxyl groups. Ring-labeled compounds should also have been used, and comparisons made between the results obtained with the side-chain labeled preparations. In the opinion of the reviewer, the absence of leached residues in this study could have been due to the simple unavailability of the compound as a result of adsorption at the soil surface or through volatilization and/or metabolism of the side groups.
3. Procedures and protocols were adequate. Control soil column studies were run, and limits of detectability for analysis of both soil samples and water leachate samples were presented. Triplicate subsamples of each soil column segment were analyzed for radioactivity, and duplicate subsamples were run for leachate analysis.

Acifluorfen

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CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Satterthwaite, S.T. (1976). Translocation Studies with ¹⁴C-RH-6201 on Soybeans, Rice, Cocklebur and Nutsedge (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa. Technical Report No. 3423-76-6)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: David Arieti, Staff Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 10, 1979

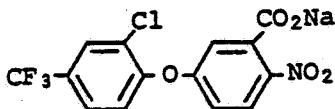
TEST TYPE: Translocation in Rice, Soybean, Cocklebur and Nutsedge

CONCLUSION:

Field test data suggest that RH-6201 adheres to soybean immediately after postemergence exposure and that most of it is lost 28 days later. Some carbon-14 labeled RH-6201 (or residue) was found in preemergence-treated field-grown soybean at 14 days and less at 28 days after treatment. Soil drench treatments of soybean in the greenhouse resulted in some translocation of label.

Weed species appear to translocate more label than crop species 5 days after treatment in greenhouse tests. In the opinion of the reviewer, radioassay data should have been collected 14 days after treatment as radioautography data were collected.

The procedures and protocol used in this study were sufficient in principle for the stated objectives but were not completely followed through.

MATERIALS AND METHODS:

RH-6201

Field Tests

RH-6201 labeled at the trifluoromethyl or carboxyl groups with carbon-14 was applied pre- and postemergence to 'Adelphia' soybean at the rate equivalent to 1 lb active ingredient per acre. A special marl clay-loam soil was used, and a 15-hour day length was maintained with overhead lights.

On days 0, 14 and 28 after application, plant samples were taken, frozen and shipped to a laboratory where they were prepared for radioautography and radioassay.

Greenhouse Tests

'Williams' soybean, 'Blue Bennet' rice, cocklebur and yellow nutsedge were used in the greenhouse study. Labeled RH-6201 was applied as a soil drench at the rate of 1 lb active ingredient per acre (a solution of 0.52 mg of chemical in 8 ml water was applied to the tops of soil in the pots in which the plants were growing). The plants were sampled at five and 14 days after treatment.

REPORTED RESULTS:

A. Field Tests

Results of radioanalyses are summarized in Table I. Postemergence treatment resulted immediately in high residue levels for both labeled compounds. Much lower levels were found 14 and 28 days later.

Radioautographs of a soybean specimen (Figure 2) show apparently complete coverage by the application spray pattern (day 0); by day 14 there is much less activity, and by day 28 almost none.

For preemergence field tests, results indicate that on day 14 there is only very slight carbon-14 translocation to the cotyledons, stems and leaves (Figure 3). On day 28 there are trace amounts of carbon-14 in the cotyledonary leaves (Table II).

B. Greenhouse Tests

Soybean results from soil drench with labeled RH-6201 show moderate carbon-14 translocation to leaves, petioles and stems. Samples at day 14 showed slight translocation (Figure 4-center set).

For rice there was moderate translocation of carbon-14 (Figure 5 center set).

For cocklebur there was a large amount of translocation to all parts of the plant and burs (Figure 6-center set).

Results with nutsedge indicate very strong translocation of carbon-14 throughout the entire shoot (Figure 7-center set).

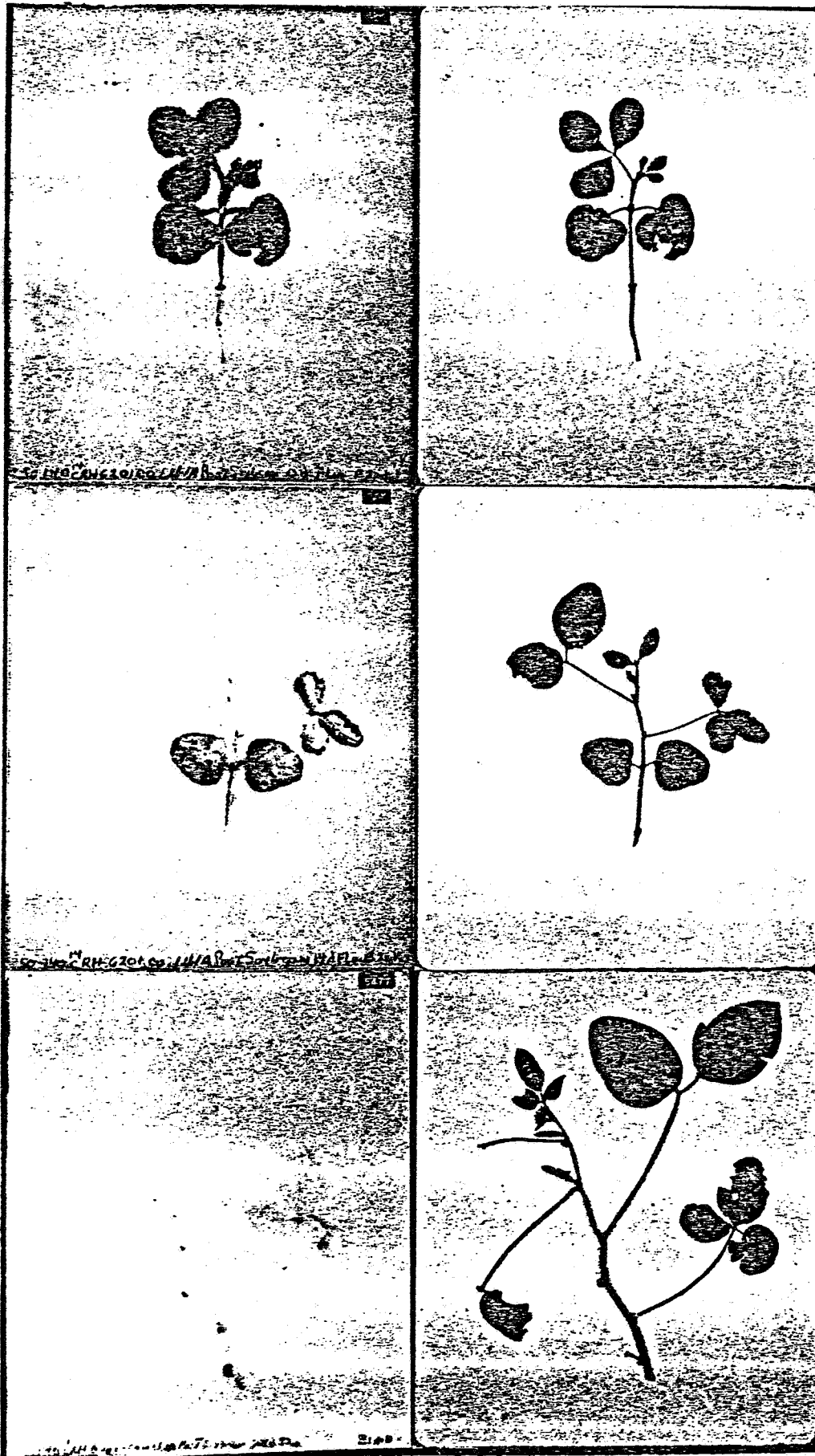
Table VII summarizes the concentrations of trifluoromethyl-labeled RH-6201 in soybean, rice, cocklebur, and nutsedge at day 5 of the experiment.

DISCUSSION:

1. In the study no ring labeled RH-6201 was used.
2. In postemergence field tests using soybeans there was evidence of residual label 14 days after treatment but very little at harvest.
3. All greenhouse data were recorded from day 14 at which time all weeds were dead. In the reviewer's opinion, these tests should have been carried out to day 28 to determine if translocation continued in soybean and rice even after the weeds species died.
4. The author felt that his data suggested greater movement of RH-6201 in susceptible plants than in tolerant plants. Weeds seemed to be more susceptible than the soybean and rice.
5. It is clear from the initially high residues of carbon-14 labeled RH-6201 on postemergence leaves that these were the result of leaf adherence of the labeled compounds (Table I). Only small amounts of residues in soybean shoots were found for the preemergence treatment.
6. The term "Untreated Leaves" in Table VII is not defined by the author and is not clear to the reviewer. If "Untreated Leaves" are controls for the soil drench portion of the study, then the residues on the controls reported in Table VII appear too high.

FIGURE 2. Translocation Data from ¹⁴C-RH-6201 CO Florida Field Residue Test

300



0 Day
Postemergence
¹⁴C-RH-6201 CO
1 lb/A

14 Day
Postemergence
¹⁴C-RH-6201 CO
1 lb/A

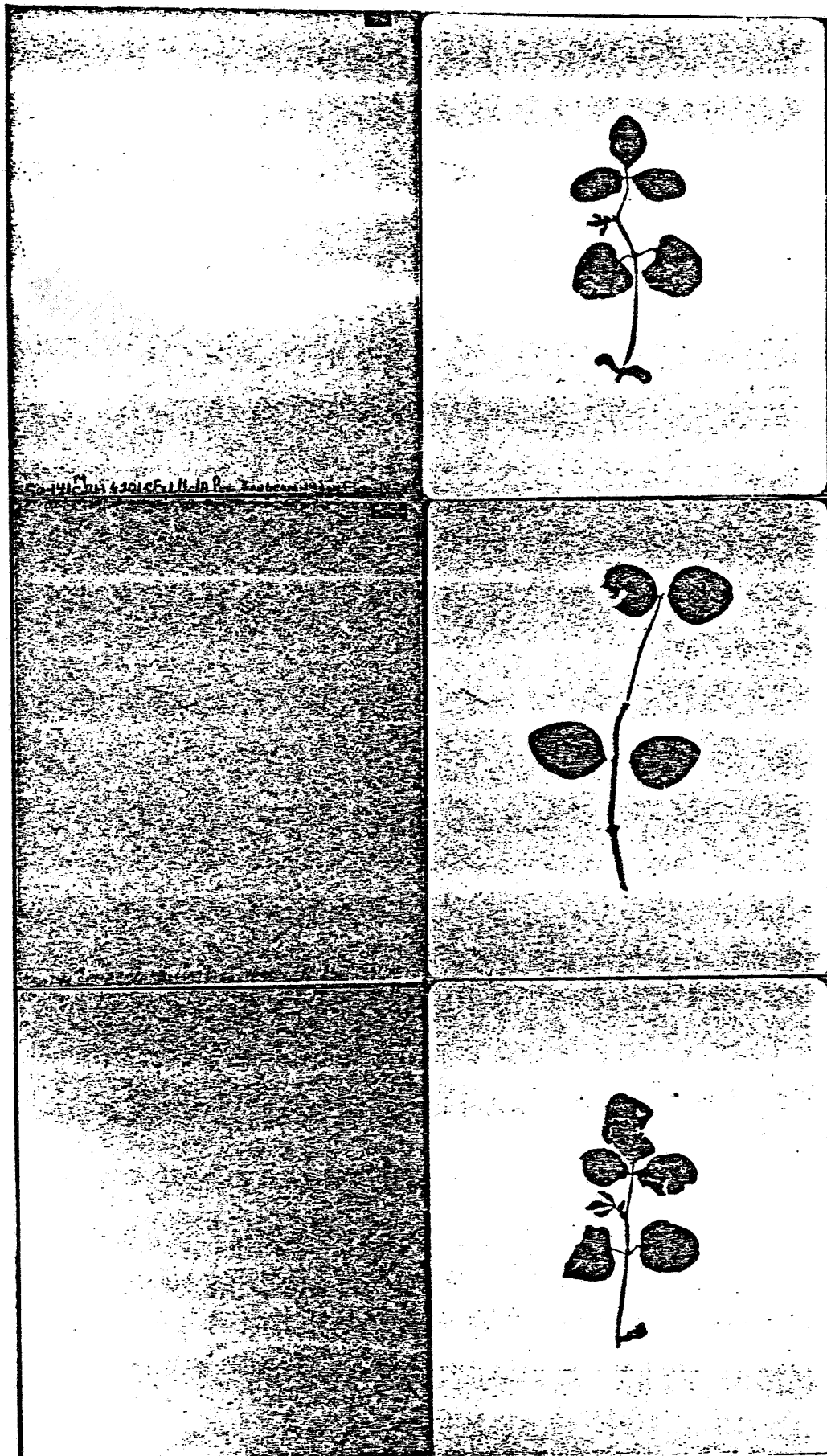
28 Day
Postemergence
¹⁴C-RH-6201 CO
1 lb/A

Radioautographs

Soybean

107

303



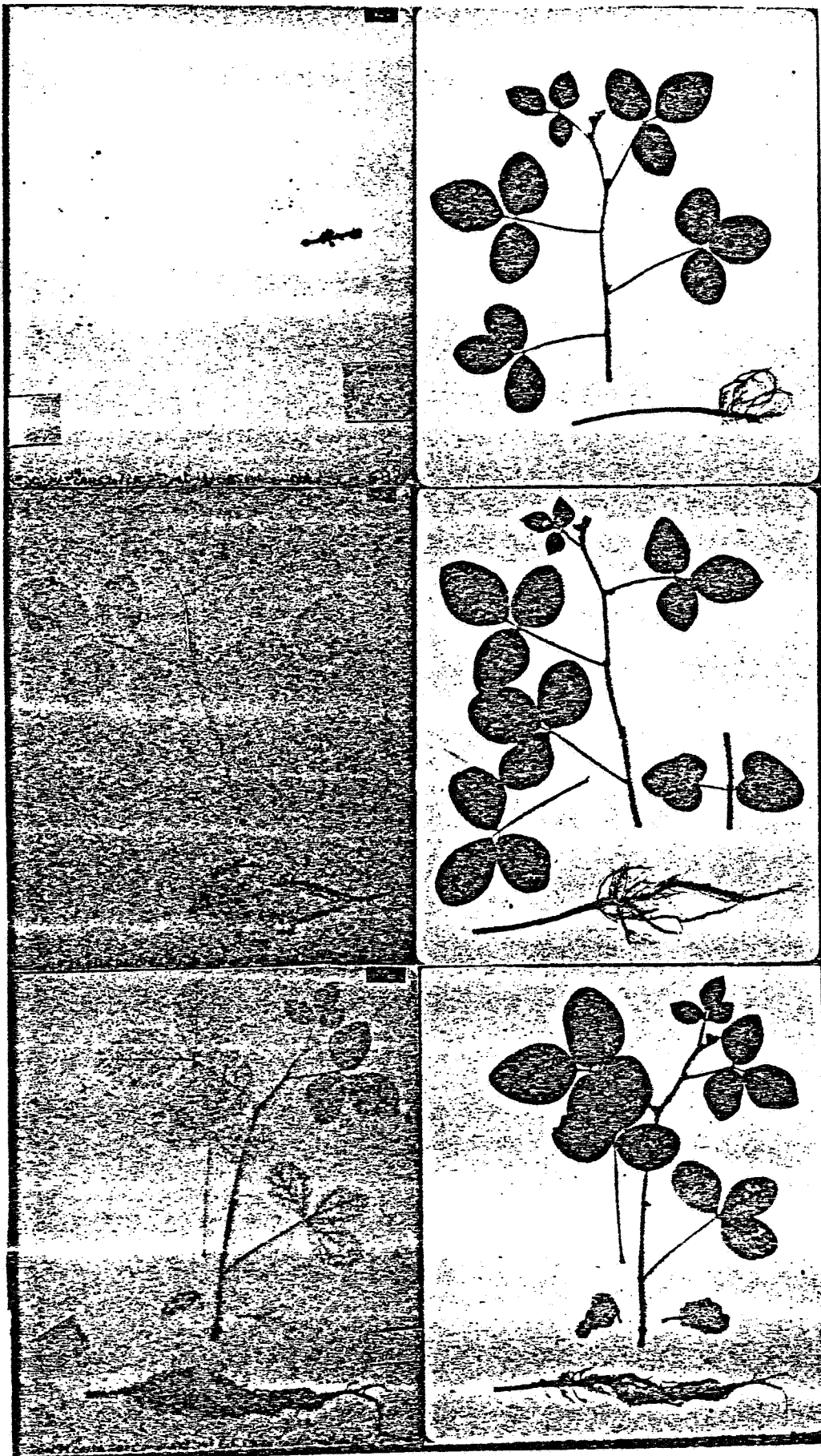
14 Day
Preemergence
 ^{14}C -RH-6201 CF_3
1 lb/A

28 Day
Preemergence
 ^{14}C -RH-6201 CF_3
1 lb/A

14 Day
Preemergence
 ^{14}C -RH-6201 CO
1 lb/A
NOTE: 28 Day, only
trace image of ^{14}C
on stem (not shown)

FIGURE 4. Translocation Studies with ^{14}C -RH-6201 CF_3 , CO 14 Days on Soybeans

307



Leaf Drop
Application
 ^{14}C -RH-6201

5 μg

Soil Drench
 ^{14}C -RH-6201

1 lb/A

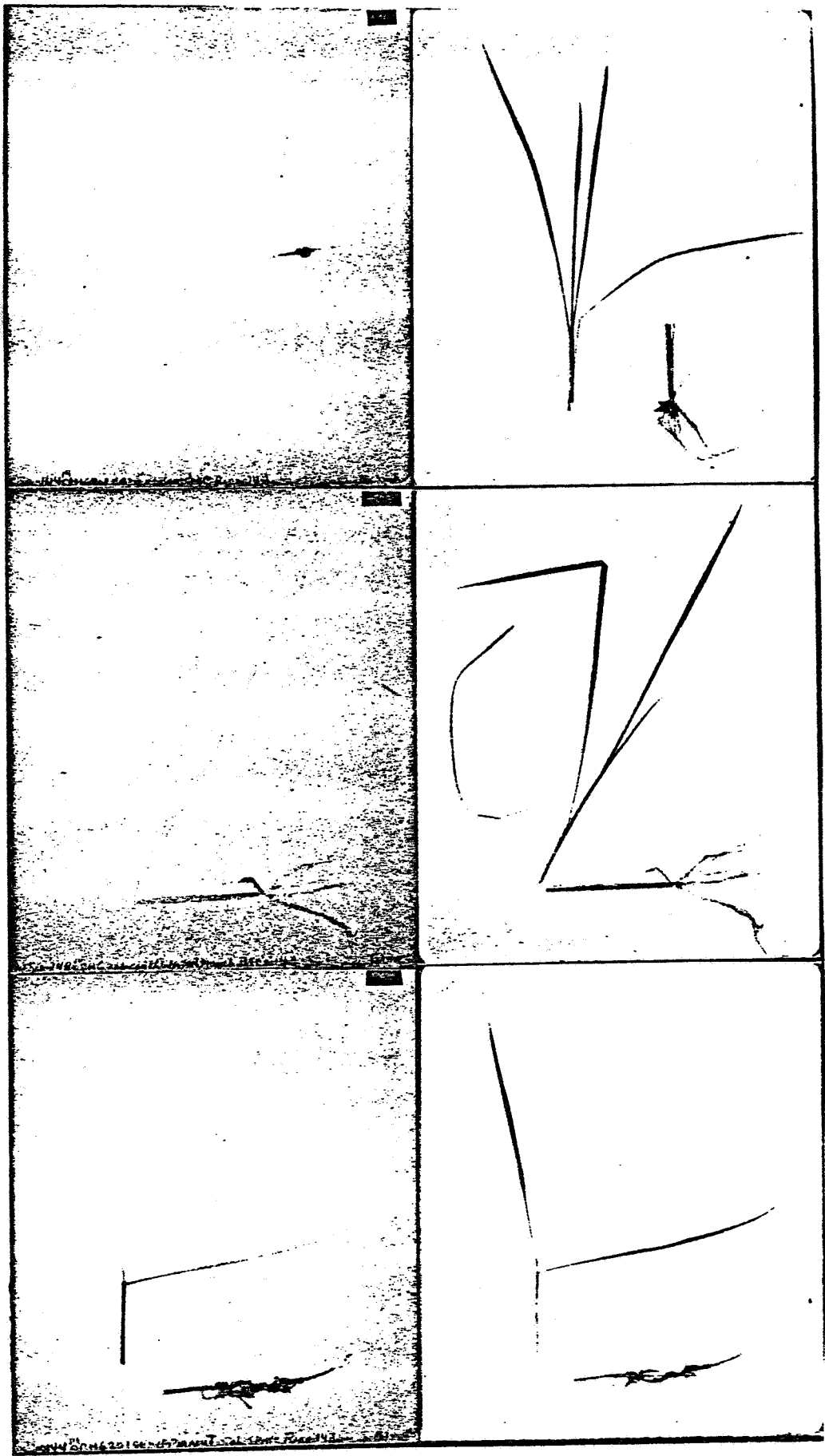
Spiked
Nutrient
Solution
 ^{14}C -RH-6201

1 ppm

Radioautographs

Soybeans

109



Leaf Drop
Application
 ^{14}C -RH-6201
5 μg

Soil Drench
 ^{14}C -RH-6201
1 lb/A

Spiked
Nutrient
Solution
 ^{14}C -RH-6201
1 ppm

Radioautographs

Rice

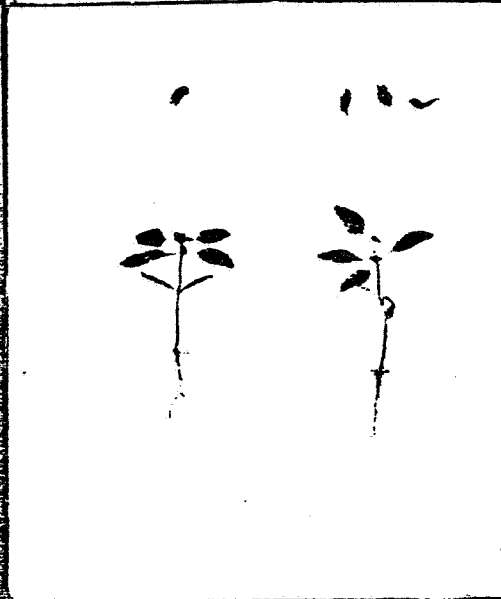
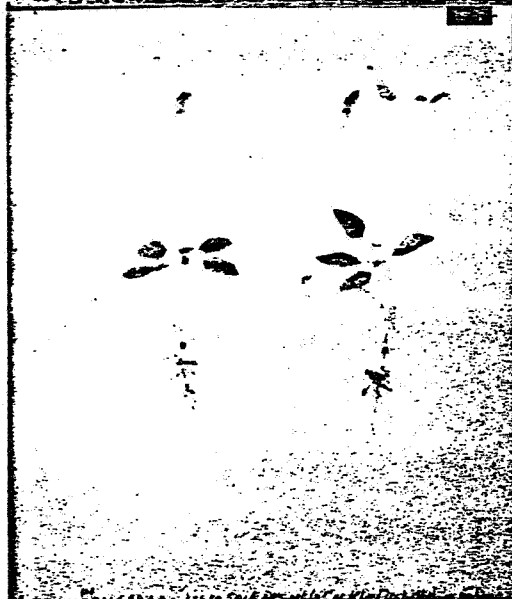
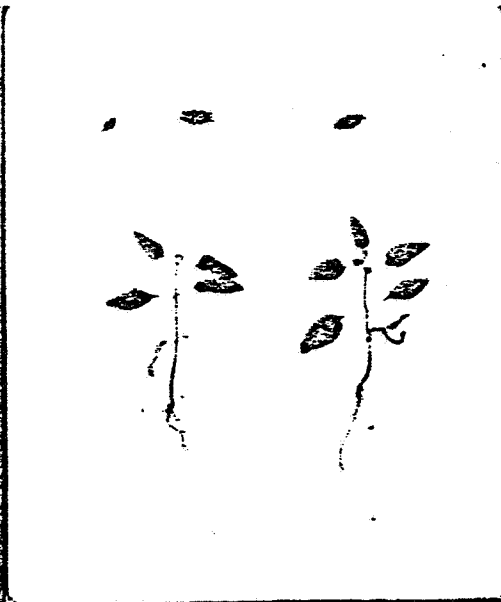
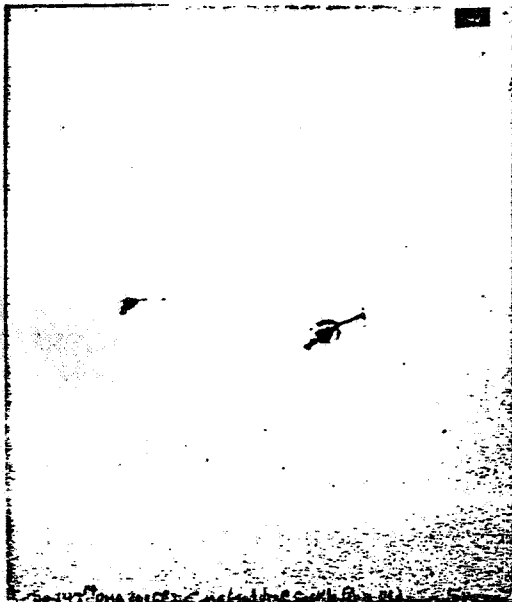
FIGURE 6. Translocation Studies with ^{14}C -RH-6201 CF_3 , CO 14 Days on Cocklebur

312

Leaf Drop
Application

^{14}C -RH-6201

5 μg



Soil Drench

^{14}C -RH-6201

1 lb/A



Spiked
Nutrient
Solution

^{14}C -RH-6201

1 ppm

Radioautographs

Cocklebur

///

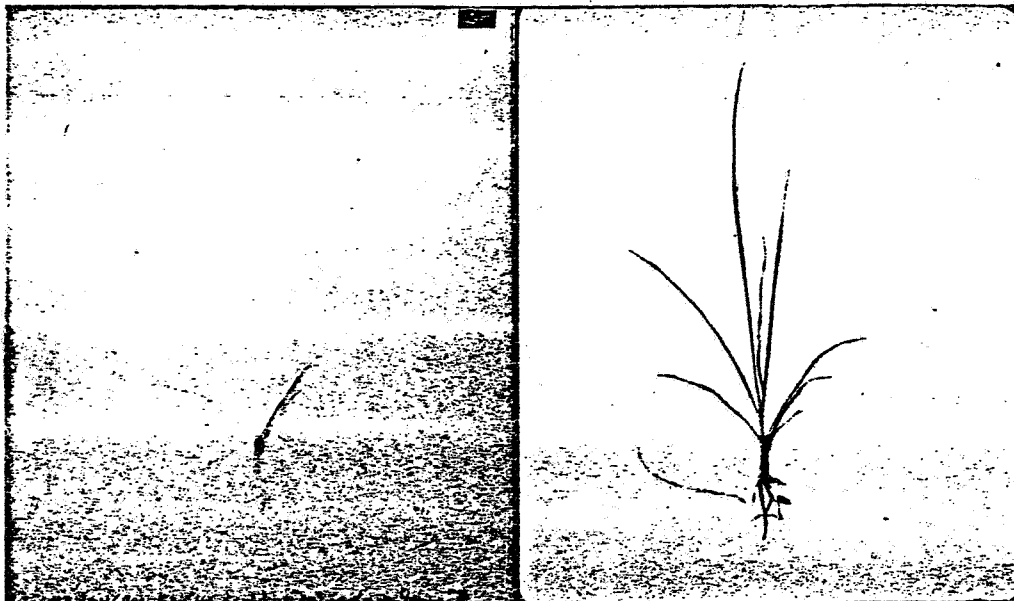
FIGURE 7. Translocation Studies with ^{14}C -RH-6201 CF_3 , CO 14 Days on Nutsedge

314

Leaf Drop
Application

^{14}C -RH-6201

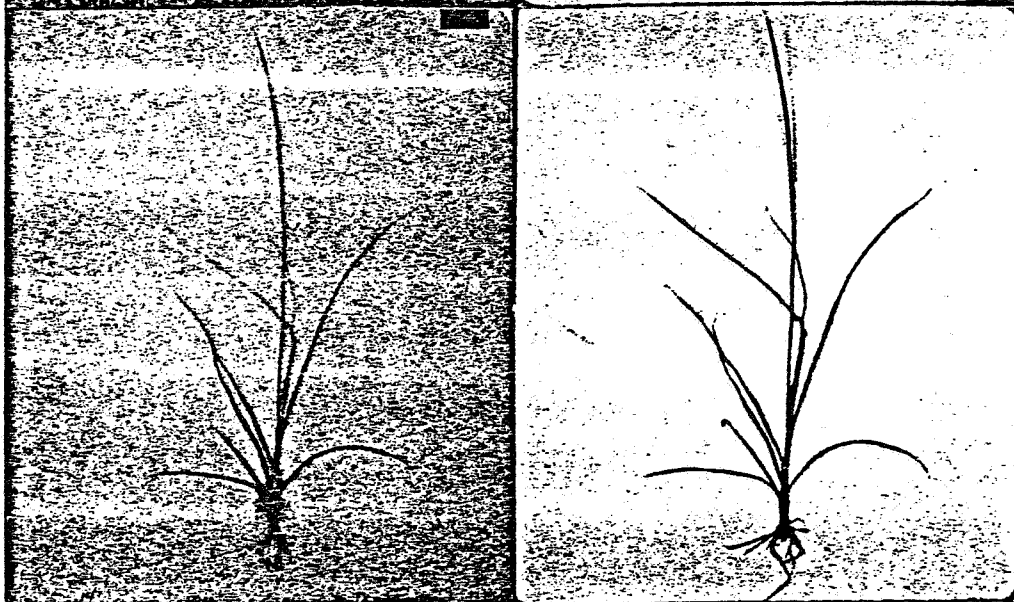
5 μg



Soil Drench

^{14}C -RH-6201

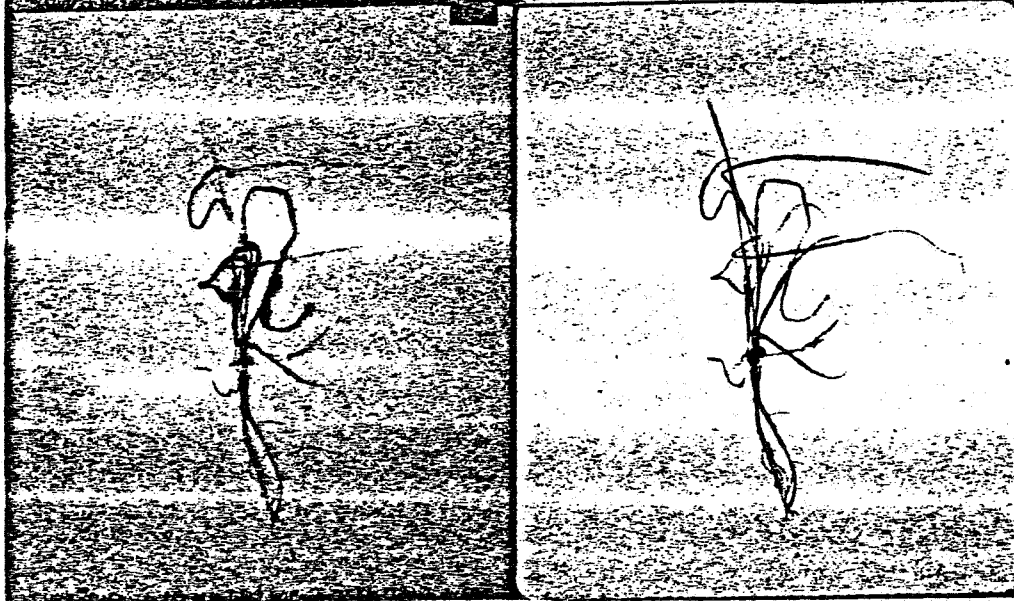
1 lb/A



Spiked
Nutrient
Solution

^{14}C -RH-6201

1 ppm



Radioautographs

Nutsedge

TABLE I¹⁴C-RH-6201 Postemergence Field Soybean Residues

<u>1975 Date</u>	<u>TSI (days)</u>	<u>Sample</u>	<u>CF₃ ppm</u>	<u>CO ppm</u>
October 8	0	Shoot	74	83
October 22	14	Shoot	5.57	6.71
November 5	28	Pod	0.04	0.11
		Shoot	0.24	0.73

TABLE II¹⁴C-RH-6201 Preemergence Field Soybean Residues

<u>1975 Date</u>	<u>TSI (days)</u>	<u>Sample</u>	<u>CF₃ PPM</u>	<u>CO PPM</u>
October 22	14	Shoot	0.13	0.12
November 5	28	Shoot	0.13	0.09

TABLE VII

Summary of ¹⁴C-RH-6201 Greenhouse Translocation Data

	Untreated Leaves ppm ¹⁴ C			Treated Leaves or Roots ¹⁴ C		
	Soybean	Rice	Cocklebur	Soybean	Rice	Cocklebur
Leaf Drop	0.09	0.13	1.51	61.0	77.0	13.0
Soil Drench	1.83	1.21	4.17	32.0	19.0	10.0
Spiked Nutrient	8.43	1.42	13.0	137.0	39.0	34.0

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). A Residue Decline Study of Soybeans Treated Preemergence or Postemergence with ¹⁴C-RH-6201 at Newtown, Pa. (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-19)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: Sandra Gould, Staff Scientist, Enviro Control, Inc. Rockville, Md.

DATE OF REVIEW: August 17, 1979

TEST TYPE: Field Dissipation Study

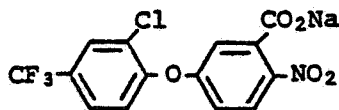
CONCLUSIONS:

The linear regression graphs, in the reviewer's opinion, are inadequate and show poor correlation between day and ppm in several graphs. The investigator should have presented information, especially on his statistical interpretations of the calculated half-lives.

There was an increase in bean and pod residues which was attributed to possible dehydration.

MATERIALS AND METHODS:

Three seedbeds were prepared in each of two enclosures. On May 24, 1976, 'Adelphia' soybean was planted in one seedbed in each enclosure. On June 7, 1976, the same cultivar was planted in the remaining two seedbeds in each enclosure.



RH-6201

On June 8, 1976, RH-6201 labeled at the trifluoromethyl group was applied as a preemergence spray to the row planted on June 7, at a dosage of 2.0 lb per acre, and then was applied as a postemergence spray to the row planted on May 24, at a dosage of 1.0 lb per acre. The other enclosure was treated in a similar manner with RH-6201 labeled in the carboxyl group.

Preemergence-treated plants were sampled four times after planting and postemergence-treated plants were sampled five times after planting. Final harvest samples were taken 125 days after treatment, 126 days and 140 days after planting the preemergence and postemergence-treated plants, respectively. Soil core samples at 0-2, 2-4, and 4-6 inches were taken at each soybean sampling. The soil composition was reported as 4.7% organic matter, 21.1% coarse fraction, 0% sand, 45.6% silt and 33.2% clay. The core depth was not reported.

The samples of plants, pods, beans, and soil were combusted and residues were determined by liquid scintillation counting. Data were corrected for combustion and counting efficiency.

REPORTED RESULTS:

A. Radioactive Residues in Soybean

Means of duplicate samples are reported in Table I. Early samples consisted of soybean foliage while later samples included plants, beans, and pods. A decrease in residues was found for the first 8 weeks of the growing season, from 1.30 ppm to 0.10 ppm, in plants treated with RH-6201 labeled at the trifluoromethyl group as a preemergence spray. An increase in residues occurred at the next sampling and again at harvest, to 2.16 ppm. Plants treated with carboxyl-labeled RH-6201 showed a decline in residues, from 0.51 ppm to 0.20 ppm at 28 days, thereafter residue levels increased with each sampling to 1.02 ppm at harvest.

Postemergence-treated plants showed a more rapid decline in residues than the decrease found with preemergence-treated plants. Residues decreased from 130 ppm at day 0 to 0.05 ppm at day 56 for RH-6201 labeled at the trifluoromethyl group; residues decreased from 78 ppm at day 0 to 0.05 ppm at day 28 for RH-6201 labeled at the carboxyl group. Increases in residues occurred with both labeled groups to date of harvest. However, the final residues were lower than those residues found in preemergence-treated plants. This is attributed to the differences in dosage rate -- the larger residues were found at the higher dosages (2 lb per acre vs. 1 lb per acre). Residues found in beans and pods increased with each sampling to harvest.

B. Radioactive Residues in Soil

Initially, residue levels for the preemergence-treated soil decreased rapidly in the 0-2 inch core (Table II). The listed values are means of duplicate samples. Between day 0 and day 14 the trifluoromethyl-labeled compound declined gradually, whereas the carboxyl-labeled one decreased to 0.56 ppm, and then increased to 1.00 ppm by the last sampling date. However, there was still an overall decrease from the initial residue found. The investigator analyzed the results by linear regression (Figures 1 and 2). The graphs were interpreted by the author to show a rapid and steady decline in the 0.2 inch core.

Postemergence data (Table II) show an initial residue decrease of 46% for the trifluoromethyl-labeled compound, and a 44% decrease for the carboxyl-labeled compound in the 0-2 inch core. Decreases were generally observed by the last sampling day, except for the 1.25 ppm value found for the trifluoromethyl compound. This value was not used to compute the linear regression in Figure 3. Data from Table II indicate a leveling off or increase in the sampling period, with the exception of the decrease found at 0.007 ppm. Linear regression was used by the investigator to demonstrate a gradual residue decline for 0-6 inch core (Figures 3 and 4). Half-lives of 62.5 to 119 days were reported from the graphs.

DISCUSSION:

1. In the reviewer's opinion, EPA data requirements for field and vegetable crop uses in field dissipation studies were not followed. The guidelines state that soil should be sampled to a 30-cm depth from sites in four agricultural-use areas for a maximum of 18 months.
2. The investigator reported half-lives of 62 to 119 days, based on Figures 1 through 4. However, it is the reviewer's opinion that this procedure is questionable. A correlation coefficient and F test are necessary to determine the significance of the regression line before a half-life can be accurately determined from the line. To determine data points for linear regression analysis, more replications may be necessary, as standard deviations from some of the means were higher than 20%.
3. The residues in the 2-4 inch and 4-6 inch cores are apparently caused by leaching, since the residues in the 0-2 inch core show a more steady decline than in the lower cores.
4. The author does not give any explanation for discarding the residue data at 125 days, that is, the 1.25 ppm found with the trifluoromethyl-labeled compound. This point was excluded from the graph in Figure 3.

Acifluorfen

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Pages 119 through 124 are not included in this copy.

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- _____ Identity of product inert ingredients.
- _____ Identity of product inert impurities.
- _____ Description of the product manufacturing process.
- _____ Description of product quality control procedures.
- _____ Identity of the source of product ingredients.
- _____ Sales or other commercial/financial information.
- _____ A draft product label.
- _____ The product confidential statement of formula.
- _____ Information about a pending registration action
- X FIFRA registration data.
- _____ The document is a duplicate of page(s) _____
- _____ The document is not responsive to the request.

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

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). A Residue Decline Study On Soybean Treated Preemergence or Postemergence with ¹⁴C-RH-6201 (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 3423-76-15)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: Wan-Shya Chou, Staff Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 21, 1979

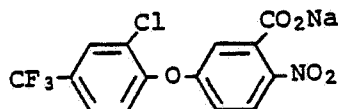
TEST TYPE: Field Dissipation Study

CONCLUSIONS:

1. The use of labeled RH-6201, as either a preemergence or a postemergence herbicide on soybean, resulted in levels of carbon-14 activity in plants which ranged from 0.06 to 0.14 ppm at final harvest.
2. Residues in soil treated (postemergence) with RH-6201 labeled in the carboxyl and trifluoromethyl carbon declined from 0.72 to 0.35 ppm, respectively by the end of the growing season.
3. The reviewer, in general, found it difficult to confirm the study's reported results because of incomplete data. The sampling and analytical methods, however, were well conceived and applied.

MATERIALS AND METHODS:

Two carbon-14 labeled forms of RH-6201 were applied (1.0 lb per acre) to separate rows of soil and/or emerging soybean.



RH-6201

Preemergence-treated soybean was sampled at 16, 30, 59, 84, and 114 days after planting. Postemergence-treated soybean was sampled 13, 27, 41, 70, 95 and 125 days after planting. The last sampling from each treatment was the final harvest. Core samples (0-2 and 2-6 inches) of the soil were taken at each soybean sampling.

Samples of immature plants, beans and pods were homogenized separately with dry ice until a powder-like consistency was attained. Final harvested samples were ground without dry ice added and soil samples were individually mixed by hand. Duplicate subsamples (about 0.4 g) of soybean samples and duplicate subsamples (about 2.5 g) of soil samples were taken for combustion and radioassay. After combustion, all samples were counted by liquid scintillation spectrometry.

REPORTED RESULTS:

A. Radioactive Residues in Soybean (Table 1)

All components of soybean plants with preemergence treatment showed a gradual increase in residues throughout the growing season. The large increase in residues in the plant material at final harvest is probably due to dehydration of the plant.

Plant material from postemergence-treated soybeans showed a rapid decline in carbon-14 content for the first 4-8 weeks after treatment, then the residues gradually increased throughout the remainder of the growing season. It was suggested that the initial decline in residue level could be attributed to growth of the plants, to washoff by rain, and to volatilization.

B. Soil Residue Decline (Figures 1-4)

Linear regression plots of soil residues vs. time showed that the residues in the 0-2 inch samples declined rapidly and steadily throughout the sampling period with half-lives ranging from 54 to 113 days in the top two inches of soil. The residue levels in the entire soil depth samples declined more slowly than in the 0-2 inch section, indicating that some of the decline seen in the 0-2 inch samples was due to leaching of the radioactive material downward through the soil.

DISCUSSION:

1. In general, the sampling and analytic methods used were well conceived and applied. However, data on the moisture content of soybean plants are needed to support the statement made above, that the large increase in residues in the plant material at the final harvest was due to the

dehydration of the plants.

2. Since the actual computations for linear regression plots (soil residues with time) were not reported in the study, the reviewer could not confirm the reported half-lives of residues in the soil.
3. The available residue data for soil samples do not allow the reviewer to confirm the reported results that the decline of the residues in the soil was due to downward leaching and some volatilization of the experimental compound.

Acifluorfen

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- _____ Identity of product inert ingredients.
- _____ Identity of product inert impurities.
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- _____ Description of product quality control procedures.
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- _____ A draft product label.
- _____ The product confidential statement of formula.
- _____ Information about a pending registration action
- X FIFRA registration data.
- _____ The document is a duplicate of page(s) _____
- _____ The document is not responsive to the request.

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

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen,
(formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). ¹⁴C Residue Studies on
Rotation Crops Grown in Plots Treated with ¹⁴C-
RH-6201 (unpublished report prepared by Rohm and
Haas Co., Philadelphia, Pa., Technical Report
No. 34H-76-9)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: Ronald L. Schaefer, Staff Scientist, Enviro Contol, Inc.
Rockville, Md.

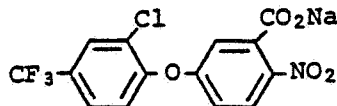
DATE OF REVIEW: August 2, 1979

TEST TYPE: Rotational Crop Uptake Study: Collard, Carrot,
Corn and Barley

CONCLUSIONS:

Rotational field studies are required to support registration of formulated products intended for field use. This study followed EPA guidelines for rotational crops, with the exception that residues were not tested in mature grain. Residues present at final sampling from preemergence and postemergence application of RH-6201, respectively, were: 0.011 and 0.049 ppm in collard foliage, 0.027 and 0.064 ppm in carrot tops, 0.010 and 0.045 ppm in carrot roots, 0.019 and 0.036 ppm in barley foliage, 0.037 and 0.008 in corn foliage, and 0.011 ppm and no detectible residue in immature corn ears.

MATERIALS AND METHODS:



RH-6201

-2-

Silt loam soil (pH = 8.3, CEC = 20.9 meq/100 g, organic matter = 6.1%, sand = 0.0%, silt = 41.5%, clay = 50.3%) was treated with RH-6201 labeled with carbon-14 either in the carboxyl group or in the trifluoromethyl group at a rate of 1 lb active ingredient per acre. It was applied to the soil as both a preemergence and a postemergence spray for germinating 'Adelphia' soybean.

After the soybean harvest, the treated plots were fertilized, rototilled, raked and planted (118 days after treatment) with 'Georgia LS' collard and 'Long Imperator' carrot. 'Wong' barley and 'Y cytoplasm' corn were planted at 134 and 148 days after treatment, respectively.

Rotation crops were sampled at 30-40 days, intervals after planting through final harvest. Collard greens, carrot tops, barley foliage, and corn foliage were homogenized by manually pulverizing or grinding with dry ice in a food chopper. Carrot roots and immature corn ears were ground with dry ice in a blender. Crop samples were combusted, and then counted with a liquid scintillation spectrometer.

Duplicate soil samples were taken at a depth of 0-3 inches 118 days after treatment, and at depths of 0-2 and 2-6 inches 246 days after treatment. They were combusted at 900°C for four minutes in an oxygen atmosphere and counted with a liquid scintillation spectrometer.

REPORTED RESULTS:

A. Radioactive Residues in Rotation Crops

Table 1 shows the level of radioactive residues (mean of two values) of labeled RH-6201 throughout the season in the various rotation crops. Residues were highest in crops immediately following soybean. Residues found in most rotation crop samples were less than 0.05 ppm with the exception of carrot tops from soil treated with trifluoromethyl-labeled RH-6201. No explanation is available for the slightly higher residues in these carrot tops.

Corn and barley grain did not develop because of the extremely wet conditions and diseases that were experienced late in the growing season.

B. Radioactive Residues in Soil

Table 2 shows the levels of radioactive residues (means of two data points) of labeled RH-6201 in the soil 118 and 246 days after treatment. There was less residue remaining in the soil at 246 days after treatment than at 118 days after treatment and, in general, residue levels decreased with soil depth.

DISCUSSION:

1. This study followed EPA guidelines in that the pesticide was aged at least 120 days under aerobic conditions for crops rotated immediately after harvest.
2. Because grain did not develop in barley and corn, no data are available, and conclusions therefore could not be made concerning residue levels in mature grain.
3. Data on precipitation, water table, and field grade required for field data under EPA guidelines were not given.
4. This study did not use ring-labeled RH-6201. This does not permit conclusions concerning metabolic fate in soils and crops of aromatic portions of this compound.

Acifluorfen

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Pages 136 through 137 are not included in this copy.

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- _____ The document is a duplicate of page(s) _____
- _____ The document is not responsive to the request.

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

CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Augenstein, L.L. (1976). ¹⁴C Residue Studies on Rotation Crops Grown in Plots Treated with ¹⁴C-RH-6201 at Newton, Pa. (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-76-21)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

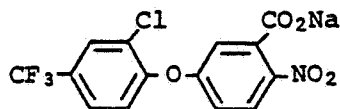
REVIEWED BY: Ronald L. Schaefer, Staff Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 3, 1979

TEST TYPE: Rotational Crop Uptake Study: Turnip, Kale, Winter Wheat

CONCLUSIONS:

Rotational field studies are required to support registration of formulated products intended for field use. This study follows EPA guidelines for rotational crops, with the exception that residues were not tested in mature grain. Residue levels present at final sampling from trifluoromethyl- and carboxyl-labeled RH-6201, respectively, were: 0.04 and 0.11 ppm in turnip tops, 0.02 and 0.04 ppm in turnip roots, 0.05 and 0.15 ppm in kale foliage, and 0.06 and 0.28 in winter wheat foliage.

MATERIALS AND METHODS:

RH-6201

There is no mention of soil type, chemical or physical analysis. A field plot was treated with RH-6201 labeled with carbon-14 either in the carboxyl group or the trifluoromethyl group at a rate of 2 lb active ingredient per acre and applied as a spray on a prepared but

-2-

bare seed bed. The field remained fallow for 56 days after treatment. It was then rototilled to a depth of 4 inches, fertilized, raked lightly, and 'Purple Top White Globe' turnip and 'Vates Blue Curled' kale planted. 'Red Coat' winter wheat was planted 98 days after treatment. Turnip and kale were sampled at 28, 56 and 69 days after planting and final harvest; winter wheat was sampled 27 days after planting. Crop samples were combusted, and then radioassayed with a liquid scintillation spectrometer. Duplicate soil samples were taken at depths of 0-2, 2-4, and 4-6 inches at 56 and 125 days after treatment, and combusted at 900°C for 4 hours in an oxygen atmosphere. Carbon dioxide was released and counted as described earlier.

REPORTED RESULTS:

A. Radioactive Residues in Rotation Crops

Table I shows the levels of radioactive residues (mean of two values) of labeled RH-6201 present in the various rotation crops. In turnip tops and kale, the 56-day residue of RH-6201 labeled in the trifluoromethyl group was higher than the 28- or 69-day samples (0.32 versus 0.13 and 0.04 ppm, respectively for turnip tops and 0.77 versus 0.15 and 0.05 ppm, respectively for kale). The 56-day residue of RH-6201 labeled in the carboxyl group was lower than the 29- or 69-day residues.

Residue levels were considerably lower in turnip roots (0.01-0.06 ppm) than in turnip tops (0.03-0.32 ppm).

B. Radioactive Residues in Soil

Table II shows the levels of residues (means based on two data points) of labeled RH-6201 in the soil 0, 56, and 125 days after treatment. There was less residue remaining in the soil as time and depth increased.

DISCUSSION:

1. EPA guidelines indicate a pesticide should be aged aerobically 30 days after treatment for assessing circumstances of crop failure. This experiment, according to the authors, represents the most rapid plant back into treated soil which might be encountered under actual situations (56 days in this case).
2. A small-grain crop was studied in accordance with EPA guidelines, but was not grown long enough to produce grain.
3. Soil characteristics were not given for the field in which the research was done. This important piece of data, missing from the research, is required by EPA guidelines.

4. Precipitation, water table, and field grade values required for field data under EPA guidelines were not given.
5. This study did not use ring-labeled RH-6201. In the opinion of the reviewer, this does not allow for conclusions concerning the metabolic fate in soils and crops of aromatic portions of this compound.

TABLE I

Radioactive Residues in Rotation Crop

Crop	Component	PSI/TSI ^a	Site of ¹⁴ C Label:	CF ₃ Group	COO ⁻ Group
			lb ai/A:	2.0	2.0
				ppm ^b	ppm ^b
Turnips	top	28/84		0.13	0.25
	top	56/112		0.32	0.03
	root	56/112		0.06	0.01
	top	69/125		0.04	0.11
	root	69/125		0.02	0.04
Kale	foliage	28/84		0.15	0.40
	foliage	56/112		0.77	0.02
	foliage	69/125		0.05	0.15
Winter Wheat	foliage	27/125		0.06	0.28

^a PSI = Planting-to-Sampling Interval in days; TSI = Treatment to Sampling Interval in days.

^b ppm calculated as RH-6201

TABLE II

Radioactive Residues in Soil

TSI ^a	Depth of Sample	Site of ¹⁴ C Label:	CF ₃ Group	COO ⁻ Group
		lb ai/A:	2.0	2.0
			ppm ^b	ppm ^b
0	0-2"		3.87	5.36
56 ^c	0-2"		0.65	0.90
56 ^c	2-4"		0.20	0.02
56 ^c	4-6"		0.25	0.04
56 ^d	0-4"		0.82	0.73
125	0-4"		0.46	0.22
125	4-6"		0.01	0.02

^a TSI = Treatment-to-Sampling Interval in days

^b ppm calculated as RH-6201

^c Samples collected prior to rototilling

^d Samples collected after rototilling

561

141

CHEMICAL: Blazer, sodium salt of acifluorfen carbofluorfen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Rogerson, Thomas D. (1978). ¹⁴C Residue Studies on Rotation Crops from Plots Treated with ¹⁴C-RH-6201 at Newtown, Pa. (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-78-7)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: Ronald L. Schaefer, Staff Scientist, Enviro Control, Inc., Rockville, Md.

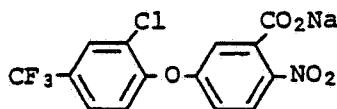
DATE OF REVIEW: August 6, 1979

TEST TYPE: Rotational Crop Uptake Study: Lettuce, Turnip, Cotton, Corn, Kale and Winter Wheat

CONCLUSIONS:

Rotational field studies are required to support the registration of formulated products intended for field use. Part 1 of this study follows EPA guidelines precisely. It was reported that labeled RH-6201 residue levels, at the end of the growing season for rotational crops planted one year after treatment, declined to 0.04-0.08 ppm in lettuce, 0.01 ppm in turnip roots, 0.01-0.04 ppm in turnip foliage, 0.01-0.03 ppm in cotton seed, and 0.01-0.03 ppm in corn grain.

The second part of the study did not follow EPA guidelines because kale and winter wheat were planted 56 and 98 days after treatment, respectively (compared to a 120-day period for crops rotated immediately after harvest).

MATERIALS AND METHODS:

RH-6201

-2-

Silt loam soil (pH = 7.1; CEC = 1.54 meq/100 g; organic matter = 4.7% sand = 0.0%; silt = 45.6%; and clay = 33.2%) was treated with trifluoromethyl-labeled or carboxyl-labeled RH-6201 at the rate of 1.0 lb (postemergence) or 2.0 lb (preemergence) active ingredient per acre and planted with 'Adelphia' soybean. One-half of each field plot used for a fallow-field residue-decline study (reported elsewhere) was rototilled and raked smooth 56 days after treatment. 'Vates Blue Curled' kale was then planted on one-half of this plot, and 'Red Coat' winter wheat was planted 98 days after treatment. The remaining plots were used for a soybean residue-decline study (reported elsewhere), and rototilled and raked smooth 309 days after treatment, at which time 'Black Seeded Simpson' lettuce and 'Purple Top White Globe' turnips were planted. '10 Chief' corn was planted and 'Stoneville' cotton seedlings (ca. 4-5 weeks old) were transplanted 345 days after treatment on these plots. Crops and crop components were sampled at periodic intervals as indicated in the tables. Each plant and soil sample was combusted, and radioassayed in a liquid scintillation spectrometer.

REPORTED RESULTS:

- A. Tables I and II show the levels of radioactive residues (mean of two values) of labeled RH-6201 present at various sampling dates in lettuce, turnip, cotton, and corn (Table I) and in kale and winter wheat (Table II). Residue levels were generally less than 0.1 ppm (in a few instances residues were in the range of 0.10 to 0.22 ppm in lettuce, turnip tops, and winter wheat) and did not decrease as the season progressed.

In general, there was no apparent difference in residue levels in crops from plots treated with carboxyl-labeled or trifluoromethyl-labeled RH-6201.

B. Radioactive Residues in Soil

Tables III and IV show the levels of radioactive residues (mean of two data points) of labeled RH-6201 in the soil for various sampling dates at depths of 0-3, 3-6, and 6-12 inches. Residue levels declined slightly during the course of the experiment and were lower as depths increased.

DISCUSSION:

1. This study was in two parts. One part, a rotational crop study with lettuce, turnip, cotton, and corn planted in a field one year after treatment of a soybean crop with labeled RH-6201, followed EPA guidelines precisely. The other part of the study compared residue levels in kale and winter wheat planted in a field that was left fallow 56 and 98 days after treatment with RH-6201. EPA guidelines require a 120-day period for crops rotated immediately after harvest.

-3-

2. Application was made at the rate of 1.0 lb per acre (postemergence) or 2.0 lb per acre (preemergence). It is not clear to the reviewer which application rate was used for determining residues in crop samples.
3. This study did not use a ring-labeled form of the experimental compound. In the opinion of the reviewer, this does not allow for conclusions concerning metabolic fate in soils and crops of aromatic portions of this compound.
4. Most climatological data were given as required by EPA guidelines for field studies. However, no information was presented on field grade and water table.

Acifluorfen

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CHEMICAL: Blazer, sodium salt of acifluorfen, carbofluorfen (formerly RH-3602, RH-6201)

FORMULATION: Technical

CITATION: Rogerson, T.D. (1978). ¹⁴C - Residue Studies on Rotation Crops from Plots Treated with ¹⁴C - RH-6201 at Newtown, Pa., in 1977 (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34H-78-23)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: Ronald L. Schaefer, Staff Scientist, Enviro Control, Inc. Rockville, Md.

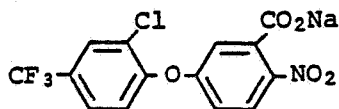
DATE OF REVIEW: August 13, 1979

TEST TYPE: Rotational Crop Uptake Study: Lettuce, Turnip, Oat, Corn and Winter Wheat

CONCLUSION:

Rotational field studies are required to support registration of formulated products intended for field use. This study followed EPA guidelines for rotational crops. At final sampling, the highest residues (from labeled RH-6201) in marketed portion of crops were: 0.02 ppm in winter wheat grain, 0.01 ppm in lettuce, 0.01 ppm in turnip roots, 0.03 ppm in oat grain, and 0.04 ppm in corn grain.

MATERIALS AND METHODS:



RH-6201

Soil characteristics are given in Technical Report No. 34H-78-5, a study not included in the data package. Soil plots were treated with RH-6201 labeled with carbon-14 either in the trifluoromethyl group or in the nitrophenyl ring at a rate of 1 lb active ingredient per acre.

One-half of one plot was used for a fallow-field residue decline study. The soil was rototilled to a depth of six inches, raked smooth 142 days after treatment, and planted with 'Red Coat' winter wheat.

Other plots were treated similarly 311 days after treatment and planted with 'Black Seeded Simpson' lettuce and 'Purple Top White Globe' turnip. 'Orbit' oat and '10 Chief' corn were planted 335 and 353 days, respectively, after treatment. Crops were sampled periodically, as shown in Table III.

Duplicate foliage and grain samples were homogenized with dry ice, combusted, and then radioassayed by liquid scintillation spectrometry.

REPORTED RESULTS:

Radioactive Residues in Rotation Crops

Table III shows the level of radioactive residues (mean of two values) of labeled RH-6201 throughout the season in the various rotation crops (Table I is a summary of Table III). In all but two cases, residue levels were less than 0.05 ppm (the two exceptions were the first sampling of winter wheat foliage and oat stalk; each had 0.08 ppm). In most cases, residue levels were either 0.01 ppm or not detectable. There appeared to be no difference in residue levels in crops from soil treated with nitrophenyl ring- or trifluoromethyl-labeled RH-6201 in cases where the two labeling sites were compared.

DISCUSSION:

1. This study followed EPA guidelines in that the pesticide was aged at least 120 days for crops rotated immediately after harvest, and one year for crops rotated on a yearly basis. In addition, the required types of crops grown (grain, leaf, and root crop) complied with the guidelines.
2. Soil characteristics were not available to the reviewer since they were not included in the data package.
3. Water table and field grade values were not given.

Acifluorfen

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Pages 151 through 153 are not included in this copy.

The material not included contains the following type of information:

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

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Cooke, P.K. (1978), Evaluation of RH-6201 Against Environmentally Important Microorganisms (unpublished report prepared by Rohm and Haas Co., Philadelphia, Pa., page 196)

TRADE SECRET CLAIM: Yes

REASON FOR REVIEW: Registration of Blazer 2L and Blazer 2S

REVIEWED BY: M. Edwards, Environmental Scientist, Enviro Control, Inc., Rockville, Md.

DATE OF REVIEW: August 13, 1979

TEST TYPE: Effects of Pesticides on Microbes

CONCLUSIONS:

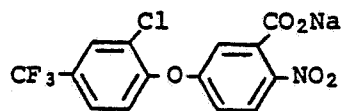
1. No conclusions were presented in this report. The data indicate, in the opinion of the reviewer, that the herbicide RH-6201 has less bacteriostatic potential than the commercial microbicide Kathon 886.
2. This study was acceptable for the endpoint being measured (growth/no growth), although additional measurements could have been made in support of visual scoring of the culture. However, the study by itself falls short of the EPA requirements for determining the effects of pesticide on microbial population, rather than on microbial functions, and does not allow a determination to be made as to whether effects on microbes will be permanent or temporary.

MATERIALS AND METHODS:

Minimum inhibitory concentrations (MIC) of RH-6201 were determined against the following selected microorganisms of ecological significance:

-2-

<u>Organism</u>	<u>ATCC #</u>	<u>Distinguishing Characteristics</u>
<u>Azotobacter vinelandii</u>	12837	Aerobic nitrogen-fixing bacterium
<u>Clostridium pasteurianum</u>	6013	Obligately anaerobic nitrogen-fixing bacterium
<u>Nostoc sp.</u>	-	Nitrogen-fixing blue-green alga
<u>Bacillus subtilis</u>	15841	Polysaccharide-decomposing bacterium
<u>Cellulomonas sp.</u>	21399	Cellulose-decomposing bacterium
<u>Pseudomonas aeruginosa</u>	10145	Bacterium utilizing organic compounds
<u>Pseudomonas fluorescens</u>	-	Hydrocarbon-metabolizing bacterium
<u>Streptomyces albus</u>	3004	Protein-degrading actinomycete
<u>Aspergillus foetidus</u>	16878	Pectin-decomposing fungus
<u>Chaetomium globosum</u>	6205	Cellulose-decomposing fungus
<u>Nitrosomonas europea</u>	19718	Ammonium-ion oxidizing bacterium
<u>Nitrobacter agilis</u>	14123	Nitrite-oxidizing bacterium



RH-6201

Test media, and conditions and periods of incubation varied depending with the organism under investigation. The minimum inhibitory concentration was determined by using a two-fold liquid serial dilution procedure to test the effects of RH-6201 up to 1000 ppm. Most tests were conducted in duplicate and for most organisms the effects on cultures were determined visually. Effects of the compound on growth of

Nitrobacter agilis and Nitrosomonas europea were determined by analyzing the nitrite levels of the culture media. A commercial microbicide (Kathon 886; 5-chloro-2 methyl-4- isothiazolin-3-one and 2-methyl-4-isothiazolin-3-one in a 3:1 ratio) was used as a positive control in each of the experiments.

REPORTED RESULTS:

Table 1. summarizes the data from these studies. Growth of Nostoc sp., Azotobacter vinelandii, Nitrobacter agilis, Nitrosomonas europea, Pseudomonas aeruginosa, Pseudomonas fluorescens, Asperigillus foetidus and Chaetomium globosum was not observed to be inhibited at the highest concentration of RH-6201 tested. No growth of Bacillus subtilis and Streptomyces albus was observed at the highest RH-6201 level (1000 ppm), and growth of Cellulmonas sp. was prevented at 500 ppm. The most sensitive organism tested was Clostridium pasterianum, for which 125 ppm was found to prevent growth.

DISCUSSION:

1. Protocols and procedures described appear adequate for the purpose of the study. Since tubes were scored only for the presence or absence of growth, visual determinations alone could be used. Visual determinations would not have been acceptable, however, if the study had attempted to evaluate percent growth inhibitions at different RH-6201 concentrations.

Ideally, visual determinations would have been confirmed by measurements of oxygen consumption, carbon dioxide, enzyme activity (dehydrogenase and/or phosphatase), or nitrogen-cycle reactions.

2. In the reviewer's opinion, a weakness in the value of this study, as alluded to above, is that no efforts were made to determine the concentrations at which partial growth inhibition would occur. This level could be considerably lower than the level causing complete inhibition. Data from the positive control does provide at least a partial basis for a comparison of the relative bacteriostatic potential of the compound.
3. The range of soil microorganisms used in this study, including bacteria, actinomycetes and fungi, is a good representation of ecologically important soil microorganisms.

4. The paper made no attempt to explain or discuss the results obtained, which are summarized Table I. It is interesting to note that Clostridium pasteurianum was the most sensitive microbe to RH-6201, and the least sensitive to Kathon 886. The reasons for the difference in sensitivity of this obligately anaerobic nitrogen-fixing bacterium were not noted.

Table I

Minimum Inhibitory Concentrations of RH-6201 vs.
Ecologically-Important Microorganisms
(Summary of Data from Tables II-XI)

Test Organism	ATCC No.	MIC, ppm AI	
		RH-6201	Kathon 886*
Alga:			
<u>Mostoc sp.</u>		>1000	0.32
Bacteria:			
<u>Azotobacter vinelandii</u>	12837	>1000	2
<u>Bacillus subtilis</u>	15841	1000	4
<u>Cellulomonas sp.</u>	21399	500	4
<u>Clostridium pasteurianum</u>	6013	125	63
<u>Nitrobacter agilis</u>	14123	>1000	0.06
<u>Nitrosomonas europea</u>	19718	>1000	0.06
<u>Pseudomonas aeruginosa</u>	10145	>1000	4
<u>Pseudomonas fluorescens</u> (API strain)		>1000	4
<u>Streptomyces albus</u>	3004	1000	0.5-1
Fungi:			
<u>Aspergillus foetidus</u>	16878	>1000	8
<u>Chaetomium globosum</u>	6205	>1000	16

*Kathon 886 (5-chloro-2-methyl-4-isothiazolin-3-one and 2-methyl 4-isothiazolin-3-one) was included in order to provide a comparison with a broad-spectrum industrial microbicide.

CHEMICAL: Blazer, sodium salt of acifluorofen, carbofluorofen (formerly RH-36201, RH-6201)

FORMULATION: Technical

CITATION: Fisher, J.D. (1978). Accumulation and Elimination of RH-6201 Residues in Bluegill and Catfish (unpublished report by EG & G Bionomics and Union Carbide Corp., Environmental Services Division, for Rohm and Haas Co., Philadelphia, Pa., Technical Report No. 34-28).

TRADE SECRET: Yes

REVIEWED BY: David Arieti, Staff Scientist, Enviro Control, Inc. Rockville, Md.

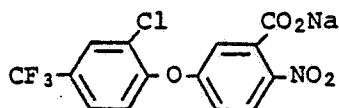
DATE OF REVIEW: August 21, 1979

TEST TYPE: Fish Accumulation

CONCLUSION:

This experiment demonstrated that initial accumulations of label from RH-6201 subside (but not completely) in bluegills over a 30-day period. The label almost disappears after exposure ceases. No conclusion can be reached about the catfish study because it was incomplete.

MATERIALS AND METHODS:



RH-6201

A. Bluegill Study (EG & G Bionomics)

Radiolabeled carbon-14 in either the trifluoromethyl group or in the nitrophenyl ring of RH-6201 was introduced into aquariums containing 300 bluegill sunfish (*Lepomis macrochirus*) weighing 3.2 ± 1.0 g. A control aquarium was also used. Fish were continuously exposed to RH-6201, 0.5 mg per liter, for 30 days and then were transferred to clean 50-liter aquariums containing well water for 14 days during which half-lives were observed.

-2-

Water was aerated continuously and maintained at $18^{\circ} \pm 1^{\circ}\text{C}$. Carbon-14 residues in the water, and in the muscle, viscera, and whole fish were sampled on days indicated in Tables III and IV during the exposure and depuration periods. On days 3, 10, 22, and 30 of exposure, 50 fish were eviscerated, and pooled samples were shipped to Rohm and Haas for metabolite identification. Fish and water controls were sampled on days 1 and 30. Duplicate sets of three fish were sampled on days 14 and 30 of exposure to quantitate hexane and methanol extractable carbon-14 residues in muscle. Prepared samples of muscle and viscera were counted in a scintillation counter.

B. Catfish Study (Union Carbide Corp.)

Sandy loam soil (pH 5.2, CEC 8.4, organic matter 2.1%, sand 70%, silt 18%, clay 12%) was aged for 40 days under aerobic conditions before the experiment.

RH-6201 was labeled at the trifluoromethyl group or at the nitrophenyl ring, was added to well water and to aged soil as if applied to land at 2.0 lb per acre, and was allowed to age for 3 days.

Catfish (Ictalurus punctatus) weighing 6.2 ± 1 g were to be exposed to regulated contamination with labeled RH-6201 (above) or to no contamination for 28 days in three identical exposure pools indoors. Owing to high mortality from infestation in all groups including controls, the ring-labeled experiment was terminated on day 20, and surviving fish were transferred to the depuration system. The trifluoromethyl-labeled experiment completed the 28 days of exposure before depuration.

Residues were determined in soil, water, and fish samples on dates indicated in Tables V and VI.

Degradation products in catfish were characterized by Rohm and Haas Co. from samples taken during the static catfish study. Samples of soil, water, and fish were prepared for radioassay and TLC by extraction procedures using a solution of acetonitrile/water/hydrochloric acid for soil and fish, and ether/hydrochloric acid for water. Samples were counted in a scintillation counter or were analyzed by TLC. Half-lives of RH-6201 were also determined.

REPORTED RESULTS:

A. Bluegill Study (Refer to Table I and Figures 1 and 2)

With an average of trifluoromethyl-labeled residue in the water at 0.50 ppm, the highest residue was found on day 7 of exposure for both muscle and viscera at less than 0.13 and 6.8 ppm, respectively, while for whole fish the highest level was less than 1.1 ppm. The bioconcentration factor was 14.0 for viscera and less than 1.0 for muscle and for whole fish.

160

During depuration, carbon-14 residues remained undetectable in muscle and decreased by 80% and 59% in viscera and whole fish (Table I).

Residues of nitrophenyl ring-labeled RH-6201 averaged 0.56 ppm in the water. In muscle the highest level was 0.50 ppm on day 3, while the highest residue for viscera and whole fish was 23 and 3.8 ppm, respectively, on day 7. The bioconcentration factors were 41 for viscera, 7 for whole fish, and less than 1.0 for muscle. During depuration, muscle concentrations were below detectable limits while concentrations in viscera and whole fish decreased by 96% and 95% (Table I).

Figures 1 and 2 show rapid uptake of carbon-14 by whole fish during the first week of exposure, which was not accounted for by muscle. An equally rapid loss of carbon-14 occurred during the second week of exposure, again not referable to muscle, and a steady decline thereafter until day 30 of exposure (inconsistent with a 28-day exposure period). Depuration produced a further sharp initial decline of whole fish carbon-14. Although the amounts of muscle carbon-14 remained quite steady, extraction data indicate some compartmental shifts during the period of observation, described in the text of the report.

B. Catfish Study (Refer to Tables I, V and VI)

Mortalities averaged 20% (Summary Table 4), and the author asserted that the epizootic did not affect movements of carbon-14. Radioanalysis data are grouped as edible or inedible tissues. Major uptakes were by inedible tissues and were continuous throughout exposure, unlike the bluegill uptakes. The label position apparently made no difference. Bioconcentration factors for catfish exposed to trifluoromethyl-labeled RH-6201 residues were 3 for edible and 8 for inedible tissues, and 6 for whole fish; exposures to ring-labeled residues were shorter and the factors somewhat lower.

C. Metabolite Characterization (Refer to Tables II and VIII)

From the data it appears that the acid form of RH-6201, designated as RH-5781, is the most prevalent in the soil/water system with 69% after the 40-day aging period, while unknowns comprise less than 12%.

With the fish exposure experiment, unknown compounds in soil comprised 81% using trifluoromethyl-labeled RH-6201. In water, unknowns comprised 17.1% with trifluoromethyl-labeled RH-6201 and 5.4% with ring-labeled RH-6201.

In catfish, the unknown compounds comprised 21% with the trifluoromethyl-labeled RH-6201 and 18.1% with ring-labeled RH-6201. Half-lives were calculated to be 4.6 and 4.2 days for trifluoromethyl- and ring-labeled RH-6201, respectively (see Figure 1 on text page 1225).

DISCUSSION:

1. Results of the bluegill study indicated that ring-labeled RH-6201 was picked up more readily than trifluoromethyl-labeled RH-6201.
2. In all bluegill studies, the viscera accumulated virtually all the carbon-14 and little was transported into the muscles.
3. During exposure and again during depuration, there were decreases of carbon-14 in the bluegills to below detectable levels.
4. The objective of the study was to define kinetics of carbon-14 in bluegills exposed to labeled residues of RH-6201. This objective was fulfilled under the limited conditions of this study.
5. The catfish study met the objective of determining residues following depuration, but in the opinion of the reviewer the study was incomplete for the following reasons:
 - a. There was a high mortality owing to an epizootic affecting all groups which affected some of the data and perhaps all of them.
 - b. There were only three tanks, two experimental and one control. The control was not treated exactly as either of the experimental tanks.
 - c. Parts of the experiment were curtailed arbitrarily.
6. The catfish experiment deviated from the EPA guidelines in that the soil was aged for 40 days instead of the 28-day maximum in the guidelines.
7. The unknown labeled compounds need to be identified because they amounted to 20% in the catfish and 74-81% in the water.

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