

FATE AND PERSISTENCE OF AMINOCARB  
IN CONIFER FOLIAGE AND FOREST SOIL AFTER  
SIMULATED AERIAL SPRAY APPLICATION

by

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## ABSTRACT

The rate of disappearance of aminocarb residues in spruce foliage and forest soil was studied under natural meteorological conditions. The aminocarb formulation was applied as a simulated aerial spray at a rate of 3.4 l/ha containing 57 g AI. Aminocarb residues were determined by gas chromatographic analysis, using a Tracor Model 550 chromatograph fitted with a Hall detector, over a 69-day period following treatment. Initial concentrations of *ca* 10 ppm aminocarb in foliage and 7 ppm in soil had half-life times of *ca* 6 and 2 days respectively, and had dropped to trace or non-detectable levels within 47 and 27 days. Aminocarb was found to be labile and dissipated rapidly under normal weathering conditions. Details of the extraction and chromatographic determination of aminocarb residues in foliage and soil are included.

RESUME

La vitesse de disparition des résidus de l'aminocarbe du feuillage des épinettes et du sol forestier a été étudiée dans des conditions météorologiques naturelles. On a appliqué une préparation d'aminocarbe contenant 57 g d'I.A., lors d'une pulvérisation aérienne simulée, à raison de 3,4 l/ha. Les résidus ont été dosés par chromatographie en phase gazeuse, à l'aide d'un chromatographe Tracor modèle 550 à détecteur Hall, durant les 69 jours qui ont suivi la pulvérisation. Les concentrations de départ d'environ 10 p.p.m. dans le feuillage et de 7 p.p.m. dans le sol avaient une demi-vie respective de 6 et 2 jours. Elles n'existaient plus qu'à l'état de traces ou étaient complètement disparues en moins de 47 et 27 jours respectivement. L'aminocarbe s'est montré labile; il disparaît rapidement dans les conditions normales de température. Nous incluons les détails de l'extraction et du dosage chromatographique des résidus de l'aminocarbe dans le feuillage et dans le sol.



## INTRODUCTION

Coniferous forests of North America are frequently damaged by numerous species of insect pests. The most common of them are the lepidopterous defoliators followed by various species of sawflies, beetles and weevils. Among the defoliators, spruce budworm, *Choristoneura fumiferana* (Clemens), is a widespread and often serious pest, frequently destroying large areas of spruce and fir forest in Canada. Currently, forests of northeastern United States and adjacent areas of Canada are under attack by the spruce budworm and it is estimated that about 50 million hectares (ha) of valuable spruce and fir stands are endangered by this insect and the potential economic loss could be several billion dollars. These forest stands are the primary supply of pulp and timber for Canada's largest foreign currency source as well as the environmental basis for wildlife habitat, freshwater systems and inland aquatic fauna.

The Canadian Forestry Service of Fisheries and Environment Canada maintains a program of research directed towards the reduction of damage by these pests. The Forest Pest Management Institute (F.P.M.I.) takes a lead role in this program.

Until 1969, economic losses due to these forest insect pests were minimized by aerial application of various organochlorine insecticides such as DDT, BHC, dieldrin, etc. Due to the protracted persistence of these compounds in the environment and their biomagnification in several food chains, they were gradually replaced by a number of compounds of the organophosphate and carbamate types of insecticides. The principal criteria used in selecting new candidate insecticides are: high toxicity to target insects, and minimal or acceptable toxicity to components of the forest

ecosystem such as birds, mammals and fish. The current emphasis is on finding non-persistent insecticides which are environmentally and ecologically acceptable for forest pest control operations. Compounds for aerial application must be adaptable for use in various formulations and modes of application; their cost is an important factor and must be weighed against the value of protection against the pest species. For practical reasons, selected materials must be available in sufficient quantities for aerial application over large areas. Finally, the pest population should be reduced to the desired level with no harmful residual effects.

The carbamate, "aminocarb", (4-dimethylamino-*m*-tolyl methylcarbamate Fig. 1) was manufactured and introduced by Bayer in Germany in 1963 under the registered trade mark Matacil<sup>(R)</sup>. Aminocarb is marketed in North America by Chemagro Chemical Company and has been used increasingly for budworm control because of its effectiveness combined with low mammalian toxicity, high insect toxicity, short residual life in the environment and its relatively harmless decomposition products (Sclagbauer and Sclagbauer 1972).

Aminocarb has been used operationally since 1973 for the control of budworm in Canada with few adverse side effects (Buckner *et al* 1975). To date approximately 4.5 million kilograms of the chemical has been applied aerially on about 5.2 million hectares of forest at the usual operational dosage of 0.070 kg AI/ha when the budworm were primarily 3<sup>rd</sup> or 4<sup>th</sup> instars.



While less persistent chemicals properly dispersed to the required levels under acceptable meteorological conditions may not cause any significant ecological hazard, there is always the probability that toxicants which kill a target pest will present some risk to other components of the ecosystem. With the increasing use of aminocarb for large scale forest insect pest control, it was deemed prudent to investigate its distribution, persistence and ultimate fate in different components of the forest environment and to begin a study of any long term effect on various nontarget species of fauna inhabiting the forest and exposed to the chemical.

The study conducted during the 1976 spray season and reported here was planned with the following objectives: To obtain data on deposit and residue levels of aminocarb and its persistence, half-life and rate of dissipation after application at operational dosage levels. The aminocarb formulation was applied twice, with a 7-day interval between treatments, as simulated aerial spray to single small white spruce trees, [*Picea glauca* (Moench) Voss], and forest soil. Such a study should enable personnel involved in pest management programs and other regulatory agencies to evaluate and assess not only the overall efficacy of the chemical but also its ecological acceptability and facilitate registration of the material for forestry use as required by the Canada Department of Agriculture. In addition, the deposit levels and coverage characteristics of the deposits obtained by the simulated aerial application resemble closely those applied in operational use. When applied to trees growing in their natural environment and exposed to the varying meteorological conditions, the resulting data are useful in evaluating the efficacy of insecticides realistically under existing forest environmental conditions.

## MATERIALS AND METHODS

### 1. Experimental Design

The residue level and persistence studies of aminocarb in foliage and forest soil were determined on a tree farm near Shawville, Quebec. Nine spruce trees, including one control, of near uniform size and shape (1.9 to 2.2 m in height and 7.5 to 8.0 cm d.b.h.) and with abundant foliage were selected for the experiment and numbered. Trees M1 to M8 received the spray application and tree M9 served as the untreated check.

In the tests of aminocarb residues in forest soils, a 4.4 m<sup>2</sup> area near the treated trees consisting of typical forest soil cover, was treated with aminocarb using the same procedure as in application to trees. A similar plot a few meters away served as the untreated check.

Foliage samples from sprayed and check trees, soil samples from the forest floor around the trees, and soil samples from treated and control plots were taken at regular intervals after spray application for studying persistence and dissipation patterns of aminocarb in these substrates.

### 2. Insecticide Formulation

Analytical grade aminocarb and the commercial concentrate containing 0.168 kg AI/l (1.4 lbs/U.S. gal.) were supplied by courtesy of the Chemagro Chemical Company, Kansas City, Missouri. For application, the concentrate was diluted ten times with a mixture containing fuel oil (No. 2) and Arotex<sup>®</sup> 3470 in the ratio of 3:1 by volume to give a formulation containing 1.67% aminocarb (wt/vol). Automate Red B dye (Morton-Williams Canada, Ltd., Ajax, Ont.) was added as a tracer for deposit and droplet density determinations.



### 3. Application of Insecticide

The aminocarb formulation was applied by the technique and device developed by Hopewell (1973) for producing simulated aerial spray deposits. A portable shelter enclosing an area  $2.1 \times 2.1$  m and height of 2.4 m was placed around each tree during application. The required volume for the nominal dosage to be applied was measured into the syringe of the droplet producing device. The unit was raised over the tree in the shelter and the formulation emitted as evenly as possible over the enclosed area during an emission period of *ca* 85 seconds. In each treatment carried out, 1.5 ml of the formulation was emitted over the  $4.41 \text{ m}^2$  for a nominal volume application rate of  $3.4 \text{ l/ha}$  and  $57 \text{ g AI}$ . A similar application was made to the soil plot in an adjoining area clear of trees.

Two applications were made, the first on July 6, and the second on July 14, 1976 from 0900 to 1230 hours under somewhat different meteorological conditions. During the first application, the average daytime temperature was *ca*  $27^\circ\text{C}$  with a wind speed of *ca*  $2 \text{ km/hr}$  and no precipitation; during the second application, the temperature was *ca*  $21^\circ\text{C}$  with an approximate wind of  $8 \text{ km/hr}$  and light rain had fallen just prior to treatment.

Among factors which influence deposition levels at the target area under operational conditions are: wind speed and direction, height of release, air turbulence, humidity, droplet size and formulation. Although a complete monitoring of meteorological conditions existing at application and during the study period was not possible, the daily temperature variation and precipitation, which would have most effect on deposited spray, were recorded and are given in Table I.

#### 4. Sampling

##### Deposit Study

Deposit samples were taken from each tree quadrant (north, east, south and west) on sampling units consisting of a 9 cm diameter petri dish and a 10 x 10 cm Kromekote<sup>(R)</sup> card for the later determination of deposited volume (l/ha) and the droplet density (number/cm<sup>2</sup>) respectively. These units were placed on staked holders 1.8 m above ground level, approximately 0.5 m from the tree stem. Five samples of deposit were taken at ground level in the spray application to forest soil free of overshadowing trees. The deposit samples were returned to the laboratory for colorimetric assessment and drop count.

##### Residue Study

For residue study, foliage and soil samples were collected at prespray, 0, 1, 3, 7, 8, 10, 12, 15, 17, 22, 27, 33, 40, 47, 54 and 69 days post treatment. End twigs from the mid-crown branches of each quadrant of the treated trees were collected using a hand clipper, composited in sealed plastic bags, put in Coleman coolers and transported immediately to the analytical laboratory at the Forest Pest Management Institute (F.P.M.I.) in Ottawa for residue analysis.

Four cores of soil samples were taken near each treated tree (and within the 4.41 m<sup>2</sup> treated area) on each sampling day to a depth of 5 cm using a 10 cm diameter toothed auger. The soil samples collected around the eight trees were mixed well and transported at once to F.P.M.I. in plastic bags. Similarly, eight cores of soil samples were taken randomly on each sampling day from the sprayed soil plot, mixed well and brought to the laboratory in Coleman coolers for residue analysis.

Foliage and soil samples were also collected from the untreated check tree and soil plot.

## 5. Analytical Methodology

### Extraction of Aminocarb

#### Foliage

The foliage samples were cut into small pieces by scissors, macerated in a Hobart chopper and a 20 g aliquot of each sample was homogenized twice in a Sorvall blender for 5 minutes at speed 6 with 100 ml of acetonitrile as extractant each time. The homogenate was filtered under suction using "S and S sharkskin" filter paper. After washing twice with 20 ml portions of the extractant, the residue was discarded. The filtrates were pooled and flash evaporated to *ca* 5 ml. The concentrate was transferred quantitatively to a 500 ml separatory funnel, 200 ml of 0.5N sulphuric acid was added and the sample shaken. The acid layer was extracted three times with 50 ml of ethyl ether saturated with water followed by one extraction with 50 ml of chloroform and both extracts discarded. The pH of the aqueous fraction was adjusted to 7.5 by addition of saturated  $\text{NaHCO}_3$  solution while stirring. The resulting aqueous solution was then extracted four times with 50 ml of benzene. The benzene extracts were pooled, washed thrice with distilled water containing NaCl and dried employing an azeotropic distillation procedure for about 90 minutes using a Dean-Stark condenser.

The colourless dried extract was flash-evaporated to 1 ml and transferred quantitatively to a graduated centrifuge tube for gas-liquid chromatographic (GLC) analysis.



A schematic representation of the extraction and subsequent cleanup procedure for aminocarb residues in spruce foliage is given in Fig. 2.

The moisture content of the chopped foliage was determined (AOAC 1955) using two 10g aliquots.

#### Soil

In the laboratory, each soil sample was passed through a 1.25 cm mesh screen to remove large stones and debris and weighed. The pile of sieved, moist soil was mixed thoroughly by hand. A composite sample of approximately 250 g was sieved (No. 8 mesh, 2.4 mm opening) and 20 g aliquots were used in the extraction as for foliage.

Two 10 g quantities of soil were used for moisture (AOAC 1955) and pH (Atkinson *et al* 1958) determinations.

#### 6. Gas-Liquid Chromatographic (GLC) Analysis

Gas chromatographic analysis of aminocarb present in the extracts of foliage and soil were carried out using a Tracor Model 550 gas chromatograph fitted with a Hall (Model 310) detector. Operating parameters are given in Table II.

The gas chromatograph was standardized on the same day as the samples were analysed by injecting freshly prepared aliquots (2-5  $\mu$ l) of analytical grade aminocarb standards in benzene, measuring the peak heights (Fig. 3) and preparing a calibration curve by plotting peak heights *vs* concentration (Fig. 4). The calibration was checked intermittently. The foliage and soil extracts were concentrated by dry

air-evaporation to the desired concentration for GC analysis. Quantitative results of the extracted sample were obtained by measuring the peak height after injection ( $4\ \mu\text{l}$ ), under the same operating conditions, and reading the concentration from the calibration curve (Fig. 4).

#### 7. Solvents and Chemicals

All organic solvents used were either pesticide grade or freshly distilled in glass. The benzene, especially, was made anhydrous by distilling with a Dean-Stark condenser.

All the chemicals used in the analysis met the American Chemical Society specifications. The anhydrous sodium sulphate was reagent grade, heated at  $150^\circ$  overnight and stored in air-tight glass-stoppered bottles.

Laboratory sources of contamination of chemicals, glassware, solvents, etc., was found to be negligible during the period of study.

## RESULTS AND DISCUSSION

### 1. Analytical Methodology

The analytical procedure followed in the current study included; solvent extraction, partitioning, distillation, concentration and final quantitation using gas-liquid chromatography. The electron capture chromatographic method developed earlier for aminocarb quantitation (Sundaram *et al* 1976) was not adequately sensitive for foliar extracts, with a minimum detection limit (MDL) of 0.5 ppm, due to the presence of impurities with a similar retention time (RT) to aminocarb. The present use of a Hall detector in nitrogen mode for the final quantitation of aminocarb not only increased sensitivity (MDL 0.2 ppm) but also simplified the technique by eliminating the derivatization used in the former method. For analysing soils, both GLC techniques, using EC (MDL 0.10 ppm) and Hall (MDL 0.20 ppm) detectors, are suitable although the former is preferable if a higher sensitivity is required.

Average recoveries of aminocarb in spruce foliage and forest soil obtained from the untreated check tree (M9) and soil plot, spiked with known amounts of analytical grade material, and analysed by the method described were 92±6% and 91±5% (Table III) respectively for foliage and soil. The MDL of the chemical from both substrates was 0.20 ppm for 20 g material. Recoveries were low (84%) at 0.3 ppm fortification level probably due to appreciable loss of the chemical in the extraction and subsequent partition procedures.

The GLC response to the standard was sharp, symmetrical and well defined (Fig. 3) with RT of 3.25 minutes. The background interference in the chromatogram (Fig. 5) of the foliage fortified with the toxicant



was small, showing that the extraction, partition and quantitation methods employed in this study are reasonably good and the method developed is adequate for the analysis of aminocarb residues present in conifer foliage and forest soil at above 0.2 ppm concentration level.

Frequent replacement of the pyrolysis tube and the isopropanol/water solvent system in the instrument was necessary to maintain analytical precision. Moreover, the response of the Hall detector was temperamental and often nonlinear, resulting in considerable delay in the overall analysis. Apart from these problems associated with instrumentation, the program went smoothly and yielded useful data.

## 2. Distribution of Spray Deposits

The spray deposits and droplet densities actually applied to each of the treated trees and on the soil plot, as determined by colorimetric assessment of the petri dish and Kromekote card samples, are given in Table IV and the average values from these data in Table V. Both applications were closely duplicated with averages of 4.3 and 4.5  $\ell/\text{ha}$  (72 and 75 g AI/ha) and 43 and 51 drops/cm<sup>2</sup> on first and second treatments respectively. However, there was considerable variation between deposits on samples from quadrants of individual trees and between tree averages. This variance from the nominal 3.4  $\ell/\text{ha}$  rate applied may be attributed to several factors, e.g., air turbulence if wind conditions were less than ideal, experience and skill of the operator handling the equipment, inherent characteristics of the device and technique.

In any case, all the area and tree foliage within the shelter during each treatment received adequate deposits of aminocarb, only two samples showing less than 10 drops/cm<sup>2</sup>. The average measured deposit on the soil plot was 3.1 l/ha (52 g AI/ha) and 34 drops/cm<sup>2</sup>.

### 3. Distribution of Aminocarb Residues in Foliage and Forest Soil

Amounts of aminocarb residue found in the treated foliage and forest soil are recorded in Tables VII to IX. The concentration levels are expressed in ppm moist weight (as sampled) including variables such as water and volatile components. Ecological interpretations under actual field conditions and also on a dry weight basis may be made from these data. The residue data were not corrected for less than 100 percent recovery from the spiked samples mentioned earlier.

The check and prespray foliage and soil samples were analysed but did not contain any detectable insecticide residues and are not recorded here.

The results in Tables VII to IX show that 2 hours after spray application, the concentration of aminocarb found in "as sampled" foliage, partially overshadowed soil, and exposed forest soil were 9.74, 4.08 and 5.22 respectively. The initial residue levels found in the foliage (9.74 ppm) were approximately 14 times higher than the residue levels (0.7 ppm) found in foliage sampled after a conventional aerial spray operation carried out in 1974 (Sundaram *et al* 1976). Although the emitted dosage (70 g AI/ha) in 1974 and the period of sampling after

application were rather different, (14 *vs* 2 hours) the deposit levels observed in foliage using the simulated aerial application technique were much greater. This may be accounted for by the much greater percent recovery on target area in the simulated spray method (*ca* 100%) as compared to an expected 25% or less as applied operationally. The residue levels found in soil partially overshadowed by foliage and in the open were 4.08 and 5.22 ppm respectively. The difference in the two values reflects the partial screening of the applied spray by foliage of the small single trees treated: in operational application a much greater proportion would be deposited on the tree canopy with only traces reaching the ground.

#### 4. Persistence of Aminocarb in Spruce Foliage

The persistence of aminocarb in spruce foliage collected from treated trees was determined during a period up to 69 days after application. The zero day concentration in wet foliage was 9.74 ppm (Table VII) which decreased rapidly after a week to 2.46 ppm. The initial concentration after the second application was 11.73 ppm which diminished to traces (< 0.20 ppm) after an interval of 40 days.

A plot of the concentration of the insecticide residues in foliage (as sampled and oven dry) *vs* time (Fig. 6) showed a curvilinear decrease. Using the initial concentrations obtained for the first and second applications, the half-lives ( $T_{\frac{1}{2}}$ ) were read from the curve and are recorded below:



<u>Application</u>	<u>Initial Conc. (ppm)</u>	<u>T<sub>1/2</sub> (days)</u>
I	9.74 (as sampled)	3.2
	19.10 (oven dry)	4.1
II	11.78 (as sampled)	6.3
	26.07 (oven dry)	6.9

The half-life times of aminocarb for foliage as sampled were short and ranged from 3.2 (first application) to 6.9 days (second application). These values are in agreement with the half-life of 5.6 days observed earlier (Sundaram *et al* 1976) for aminocarb under aerial spraying conditions.

It is evident from this study that the aminocarb residual half-life is to a certain extent dependent upon the initial concentration observed in substrate; the higher the initial concentration, the greater the half-life and vice versa. As expected, the half-lives for the oven dry foliage samples were slightly higher than the "as sampled" material.

These studies showed that although high concentrations of aminocarb had occurred in conifer foliage shortly after application, the material dissipated rapidly, indicating that it is highly labile and would be most unlikely to harm the forest environment. A combination of factors, e.g., growth dilution, weathering, rainfall and metabolism of the chemical by the conifer probably caused the concentration of aminocarb deposited on spruce foliage to decrease rapidly with time.

##### 5. Persistence of Aminocarb in Forest Soil

Although the forest floor receives a significant portion of the aerially applied spray materials, the fate of aminocarb in forest soil has not been studied. In the 1974 application of aminocarb at the applied dosage of 70 g AI/ha, the forest floor soil did not contain detectable levels (0.1 ppm) of the chemical (Sundaram *et al* 1976). In the current study, the forest floor surrounding the treated trees contained 4.08 and 5.59 ppm (to a depth of 5 cm) immediately after successive applications of 72 and 75 g AI/ha at a one-week interval. The soil in the open area plot contained 5.22 and 7.45 ppm of aminocarb immediately after successive deposits of 53 and 50 g AI/ha, also applied at one-week interval (Tables VIII and IX). At the end of one week after the first application, more than 75% of the aminocarb added to the forest floor soil had disappeared. The oven dry soil samples, as expected, contained a higher concentration of aminocarb than the "as sampled" soils. The residue levels in both soil samples decreased exponentially with time and disappeared below the detection limit (0.20 ppm) after 22 days.

Graphed data of aminocarb concentration in ppm *vs* time (days) representing the dissipation pattern of the chemical in the two forest soils (partly overshadowed and open) are shown in Figs. 7 and 8. The half-lives for the chemical in these two soils were read from the degradation curves and are recorded below:

<u>Application</u>	<u>Type of Soil</u>	<u>Initial Conc. (ppm)</u>	<u>T<sub>1/2</sub> (days)</u>
I	Overshadowed (Fig. 7)	4.08 (as sampled)	2.4
		4.69 (oven dried)	2.7
II		5.59 (as sampled)	3.0
		6.82 (oven dried)	3.0
I	Open (Fig. 8)	5.22 (as sampled)	0.9
		5.93 (oven dried)	1.2
II		7.45 (as sampled)	2.1
		8.87 (oven dried)	2.3

As observed earlier for foliage, the half-lives in the oven-dried soil samples are usually higher than "as sampled" (wet) soils. In general, the half-life times are very short, showing that the chemical is degraded rapidly from the forest soil by physicochemical and biological processes. The extent to which the soil characteristics listed in Table VI influenced the decomposition of aminocarb was not examined and its metabolic pathway in different forest substances is still obscure.

Persistence of pesticides in the environment is dependent upon various factors such as physicochemical, biological and structural properties of the chemical; formulation, dosage, and mode of application; various environmental and climatic conditions; and finally, the type and composition of the substrate material to which the chemical is exposed. The current studies provided conclusive evidence as to the lability of aminocarb in conifer foliage and forest soil under normal weathering conditions as exist in a forest environment and confirmed that aminocarb is environmentally safe and ecologically acceptable for budworm control operations in Canada.



#### 6. Mechanism of Dissipation of Aminocarb

The behaviour of aminocarb in the forest environment begins with its initial distribution and continues through its subsequent movement, persistence and fate in each component of the environment. The emphasis in the current study has been the quantity and persistence of the chemical in conifer foliage and forest soil. Although the compound is currently utilized extensively in insect control programs, no reports are available on the mechanism of its dissipation.

The results in Table VII show that most of the aminocarb intercepted by spruce foliage dissipated within forty days. This rapid disappearance is considered to have resulted from a combination of physical, metabolic and environmental factors. Physical factors include vapour pressure of the material and weathering, i.e., sunlight, rain, temperature, etc.; metabolic factors include dilution by tree growth, migration and molecular alteration, while environmental factors include microorganisms and micrometeorological conditions. Earlier studies (Sundaram *et al* 1976, Sundaram and Hopewell 1977) showed physical factors such as growth dilution and climatic conditions, in addition to volatilization and photolysis, as observed in other pesticides (Yule and Duffy 1972, Sundaram 1974, Varty and Yule 1976), did play an important role in the loss of aminocarb from conifer foliage.

Aminocarb found in forest soil samples (Tables VIII and IX) also decreased rapidly with time probably also due to a combination of factors such as; volatilization, leaching, surface runoff, photolysis and microbial activity. During the study period, microbial activity could have been low because of the dry soil (average moisture content 18%,

Table VI). However, dissipation of the chemical also depends on its chemical characteristics, the soil type and pH.

The residue data of aminocarb compiled in Tables VII to IX were used to plot  $\log C$  (residue concentrations found in wet and dry foliage and soil samples) against time. The plots were nearly linear (Figs. 9 to 11) showing that the dissipation of aminocarb in spruce foliage and forest soil depended upon its concentrations in these substrates and accordingly followed the first-order kinetic expression as observed earlier (Ebeling 1963, Ruzieka *et al* 1967, Sundaram *et al* 1972) in other similar studies.

## CONCLUSIONS

The results clearly demonstrated the rapid dissipation of aminocarb in spruce foliage and forest soil under normal weathering conditions as exist in a forest environment, when the material was applied as a simulated aerial spray deposit. Whereas the insecticide was found to be labile, and insect mortality depends on the magnitude of the concentration deposited on target, the toxicity of the compound and the duration of persistence, it is concluded that satisfactory budworm population reduction requires a higher dosage level, e.g., 100 g AI/ha, than the currently emitted dosage of 55 g AI/ha. Higher dosage levels should be assessed quantitatively for possible immediate and long range environmental and ecological hazards. The activity and effective life of aminocarb formulations in forest pest control could possibly be improved by use of adjuvants, e.g., surfactants, UV light protectors and stickers.

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*Toxicol.* 8 (1) : 10.

Table I

Meteorological Data at the Shawville Experimental Site - Summer, 1976

Days After Application	Time of Sampling (hrs)	Temp. at Sampling °C	Intersample Temp. °C			Precipitation (mm)
			Max.	Min.	Avg.	
0*	1130	27	30	15	23	
1	1130	27	31	17	24	T
3	1130	21	29	11	20	
7	0900	12	26	12	19	21
7†	1215	15	26	12	19	**
8	1140	21	21	13	17	T
10	1400	22	27	12	20	3.5
12	1115	23	26	13	20	-
15	1110	19	30	8	19	2.0
17	1130	22	29	11	20	-
22	1120	19	29	8	18	-
27	1110	19	26	6	16	T
33	1115	17	28	7	18	22
40	1120	15	29	12	20	16
47	1225	21	31	8	20	-
54	1055	11	29	7	18	18
69	1055	21	26	1	14	25

T Traces (< 1 mm)

\* First spray on July 6 and the second one (†) on July 13, 1976.

\*\* Rain ceased before second application.



Table II  
Gas Chromatographic Parameters

GC	:	Tracor (Model 550)
Detector	:	Hall (Model 310) (Nitrogen mode)
Column	:	1.83 m x 6 mm O.D. Pyrex glass packed with 6% SE 30 on Chromosorb W, H.P., mesh 80/100
Solvent	:	15% Isopropanol in distilled water
Rate	:	1 ml/min
Temp (°C)		
Oven	:	210
Outlet	:	280
Inlet	:	215
Carrier gas (ml/min)	:	He, 100
Gas flow (ml/min)	:	H <sub>2</sub> , 150
Attenuation	:	2
Range	:	1
Recorder	:	Linear instruments, 1 mV
Chart speed (cm/hr)	:	76
Retention time (min)	:	3.25

Table III

Recovery Studies of Aminocarb added to Spruce Foliage and Forest Soil  
as Determined by GLC

Fortification Level (ppm)	Foliage		Soil	
	Average Recovery* (%)	Coefficient of Variation (%)	Average Recovery* (%)	Coefficient of Variation (%)
0.3	84	8	94	5
0.5	94	6	88	4
1.0	95	4	96	4
2.0	94	5	95	5
	Mean 92 $\pm$ 6%		Mean 91 $\pm$ 5%	

\* Three replicates of each.

Minimum detection limit 0.20 ppm.

TABLE IV

Aminocarb Applied Deposit Rates to White Spruce Trees and Forest Soil, as Determined by Dye Tracer from Samples on Petri Dishes and Kromekote Cards

Tree No.	Quadrant	First Application			Second Application		
		ℓ/ha	g AI/ha	Drops/cm <sup>2</sup>	ℓ/ha	g AI/ha	Drops/cm <sup>2</sup>
1	N	6.3	106	50	7.0	118	88
	E	9.1	153	30	2.1	35	13
	S	6.7	113	56	7.0	118	72
	W	7.1	119	55	3.2	54	11
	Avg.	<u>7.3</u>	<u>122</u>	<u>50</u>	<u>4.8</u>	<u>80</u>	<u>46</u>
2	N	2.9	49	24	4.2	71	43
	E	6.5	109	63	2.5	42	34
	S	1.5	25	10	2.3	39	31
	W	8.0	134	63	4.6	77	43
	Avg.	<u>4.7</u>	<u>79</u>	<u>40</u>	<u>3.4</u>	<u>57</u>	<u>38</u>
3	N	1.4	24	16	2.2	37	17
	E	4.1	69	25	2.9	49	32
	S	1.3	22	20	10.3	173	86
	W	2.2	37	21	4.5	76	45
	Avg.	<u>2.2</u>	<u>38</u>	<u>21</u>	<u>5.0</u>	<u>83</u>	<u>45</u>
4	N	2.6	44	50	4.7	79	109
	E	2.8	47	37	1.8	30	12
	S	11.5	193	103	2.9	49	28
	W	3.5	59	8	4.9	82	61
	Avg.	<u>5.1</u>	<u>85</u>	<u>50</u>	<u>3.6</u>	<u>60</u>	<u>82</u>
5	N	2.7	45	47	4.5	76	65
	E	5.0	84	33	3.8	64	40
	S	2.0	34	40	1.3	22	17
	W	3.6	60	49	4.2	70	39
	Avg.	<u>3.3</u>	<u>55</u>	<u>42</u>	<u>3.4</u>	<u>58</u>	<u>40</u>
6	N	2.8	47	43	6.2	104	85
	E	1.9	32	15	1.9	32	9
	S	7.5	126	55	4.4	74	51
	W	2.3	39	28	6.7	113	69
	Avg.	<u>3.6</u>	<u>60</u>	<u>35</u>	<u>4.8</u>	<u>80</u>	<u>54</u>
7	N	5.0	84	59	5.1	86	46
	E	3.8	64	59	4.2	71	31
	S	2.5	42	26	2.3	39	30
	W	3.8	64	51	8.8	148	62
	Avg.	<u>3.8</u>	<u>63</u>	<u>49</u>	<u>5.1</u>	<u>85</u>	<u>42</u>
8	N	7.0	118	75	3.0	50	38
	E	3.0	50	48	8.1	136	76
	S	3.5	59	30	3.8	64	51
	W	3.4	57	65	7.7	129	87
	Avg.	<u>4.2</u>	<u>70</u>	<u>54</u>	<u>5.6</u>	<u>94</u>	<u>63</u>
Soil	N	2.4	40	33	3.2	54	38
	E	4.3	72	43	3.3	55	25
	S	1.7	29	23	2.8	47	35
	W	3.5	59	40	1.9	32	18
	Centre	3.9	66	40	3.9	66	37
	Avg.	<u>3.2</u>	<u>53</u>	<u>36</u>	<u>3.0</u>	<u>50</u>	<u>31</u>



Table V

Average Spray Deposit Rates of Aminocarb Applied to Foliage and Forest Soil as Determined by Dye Tracer

Tree No.	First Application			Second Application			Average of Two Applications		
	ℓ/ha	g AI/ha	Drops/cm <sup>2</sup>	ℓ/ha	g AI/ha	Drops/cm <sup>2</sup>	ℓ/ha	g AI/ha	Drops/cm <sup>2</sup>
1	7.3	122	50	4.8	80	46	6.0	101	48
2	4.7	79	40	3.4	57	38	4.0	68	39
3	2.2	38	21	5.0	83	45	3.6	61	33
4	5.1	85	50	3.6	60	82	4.3	73	66
5	3.2	55	42	3.4	58	40	3.3	57	41
6	3.6	60	35	4.8	80	54	4.2	70	45
7	3.8	63	49	5.1	85	42	4.4	74	46
8	4.2	70	54	5.6	94	63	4.9	82	59
Average	4.3	72	43	4.5	75	51	4.4	74	47
Soil	3.2	53	36	3.0	50	31	3.1	52	34

Table VI  
Soil Characteristics

Soil Texture	Loamy Sand
pH	6.1
Clay Content (%)	2
Silt Content (%)	3
Sand Content (%)	95
Organic Matter (%)	4
Average Moisture Content (%)	18

Table VII

Aminocarb Residues in Spruce Foliage Exposed to Simulated Spray Deposit  
as Determined by GLC

Days After Application	Moisture Content %	Aminocarb Conc. (ppm)	
		As Sampled	Oven Dried
Prespray	47	N.D.	N.D.
0*	49	9.74	19.10
1	57	7.77	18.07
3	51	5.00	10.20
7	58	2.46	5.86
7**	55	11.73	26.07
8	54	11.08	24.10
10	61	8.33	21.36
12	57	6.82	15.86
15	51	4.70	9.59
17	49	3.43	6.72
22	52	2.60	5.42
27	58	1.32	3.14
33	62	0.60	1.58
40	57	0.20	0.47
47	53	T	T
54	58	N.D.	N.D.
69	61	N.D.	N.D.

\* First spray on July 6, 1976. Samples were taken *ca* one hour after application.

\*\* Second spray on July 13, 1976. Samples were taken *ca* two hours after application.

N.D. Not detected. T. Traces < 0.20 ppm.



Table VIII

Aminocarb Residues Found in Forest Soil Partially Overshadowed by Foliage of Treated Trees

Days After Application	Moisture Content %	Aminocarb Conc. (ppm)	
		As Sampled	Oven Dried
Prespray	11	N.D.	N.D.
0	13	4.08	4.69
1	19	2.71	3.35
3	15	1.81	2.13
7	24	1.05	1.38
7	18	5.59	6.82
8	16	4.38	5.21
10	20	2.62	3.28
12	18	1.98	2.41
15	19	1.02	1.26
17	14	0.72	0.84
22	13	0.22	0.25
27	16	T	T
33	28	T	T
40	17	N.D.	N.D.
47	14	N.D.	N.D.
54	27	N.D.	N.D.
69	26	N.D.	N.D.

See the footnotes in Table VII.

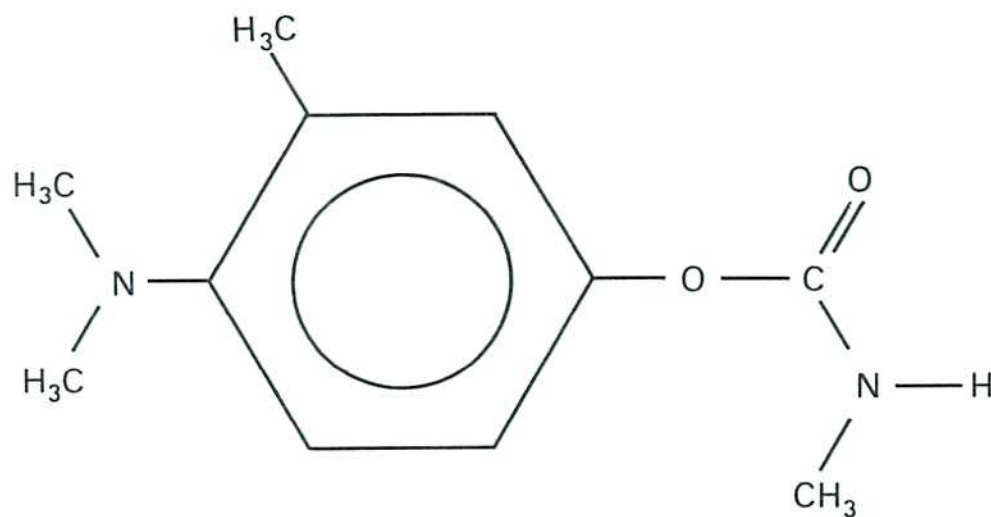
Table IX

Aminocarb Residues Found in Forest Soil Clear of Overshadowing Foliage during Application

Days After Application	Moisture Content %	Aminocarb Conc. (ppm)	
		As Sampled	Oven Dried
Prespray	12	N.D.	N.D.
0	12	5.22	5.93
1	18	2.89	3.52
3	14	1.12	1.30
7	22	0.72	0.92
7	16	7.45	8.87
8	14	5.27	6.13
10	18	3.02	3.68
12	17	1.78	2.14
15	18	0.92	1.12
17	12	0.45	0.51
22	12	0.27	0.31
27	15	T	T
33	26	T	T
40	15	N.D.	N.D.
47	13	N.D.	N.D.
54	25	N.D.	N.D.
69	24	N.D.	N.D.

See the footnotes in Table VII.

MATACIL<sup>®</sup>— AMINOCARB



4-Dimethylamino -*m*- tolyl methylcarbamate

Fig. 1. Structural Formula of Aminocarb



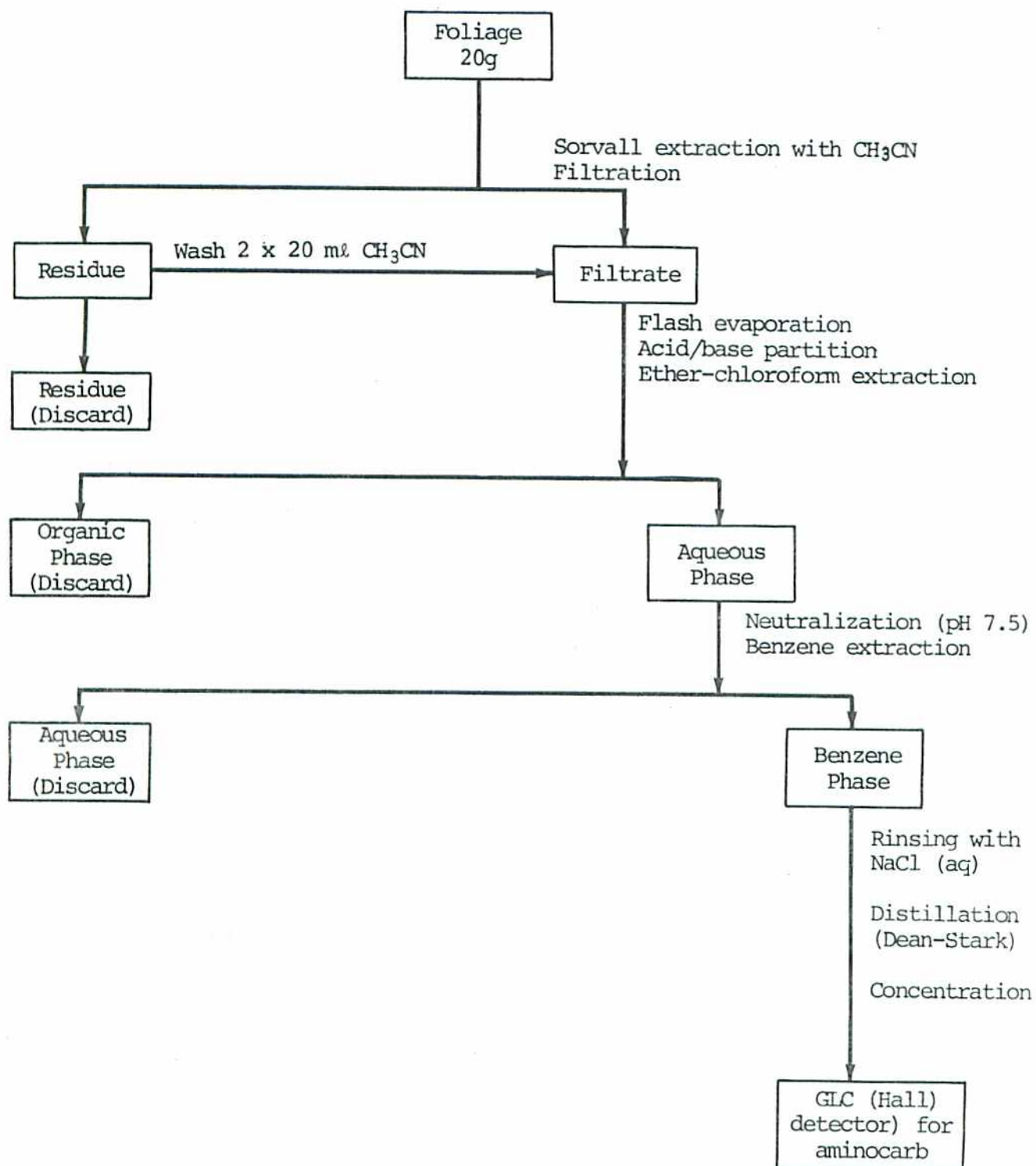


Fig. 2. Extraction and Cleanup Procedure for Aminocarb from Spruce Foliage.

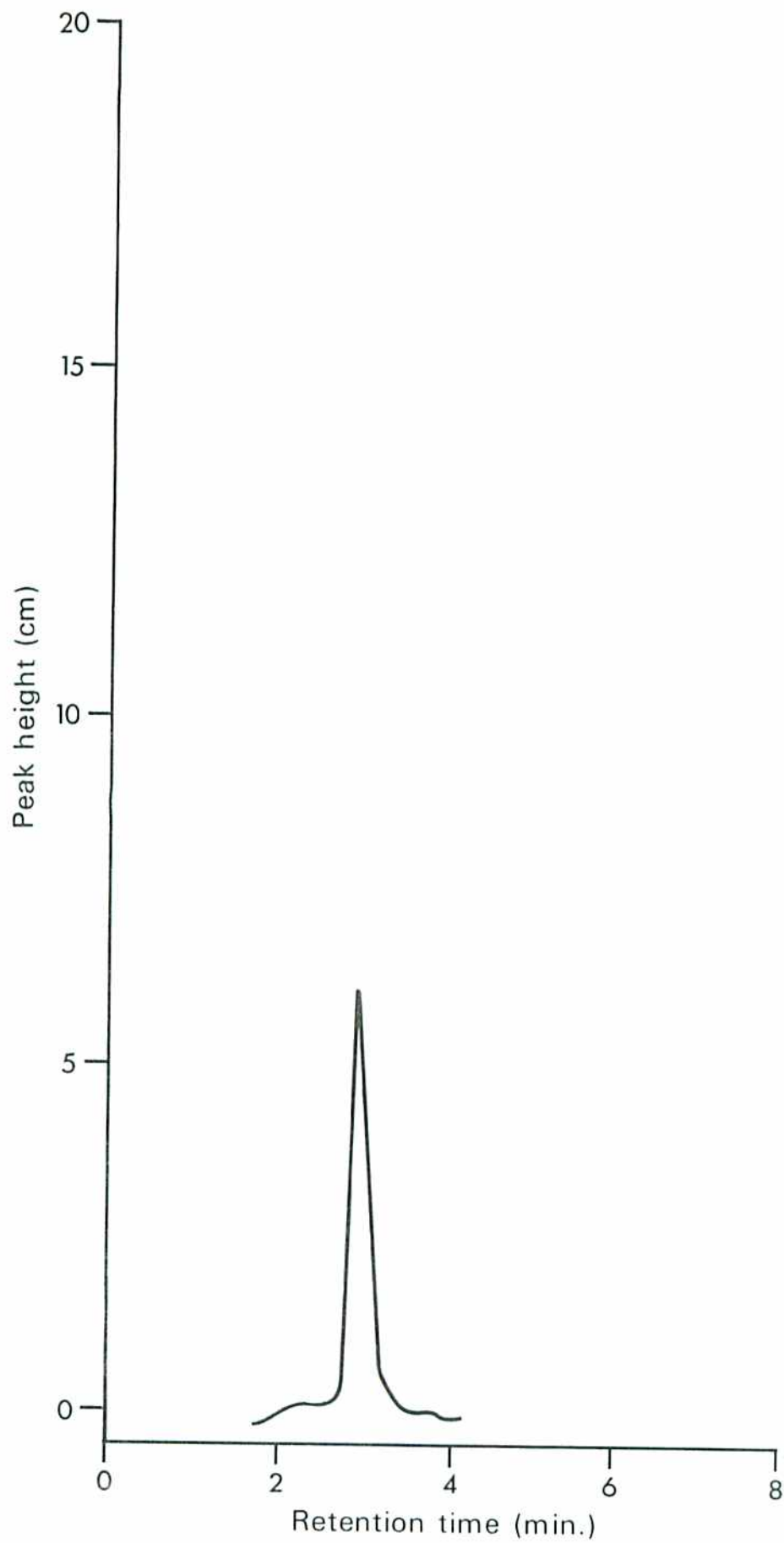


Fig. 3. Chromatographic Response of Aminocarb Standard Solution.

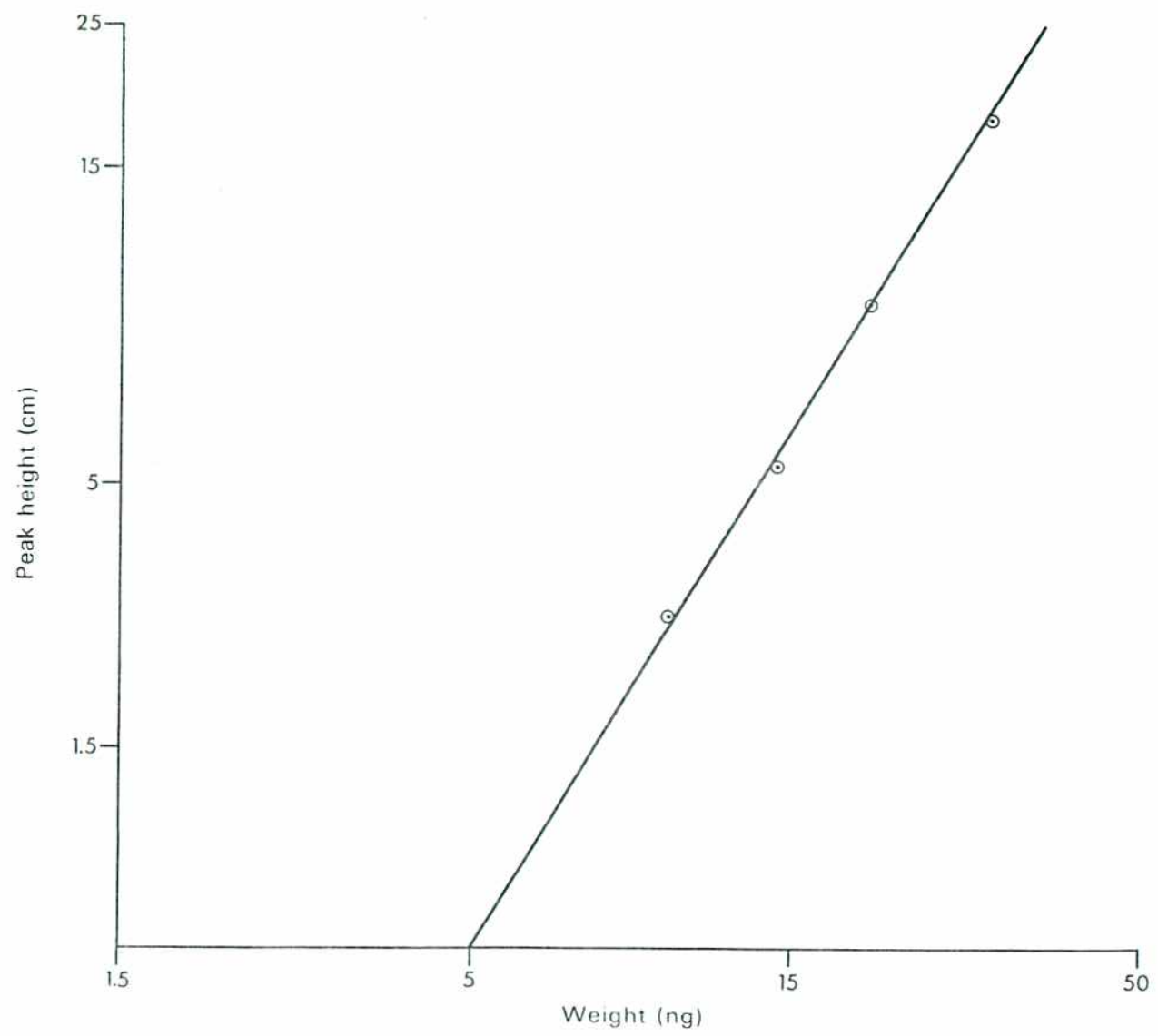


Fig. 4. GLC Calibration Curve for Aminocarb.



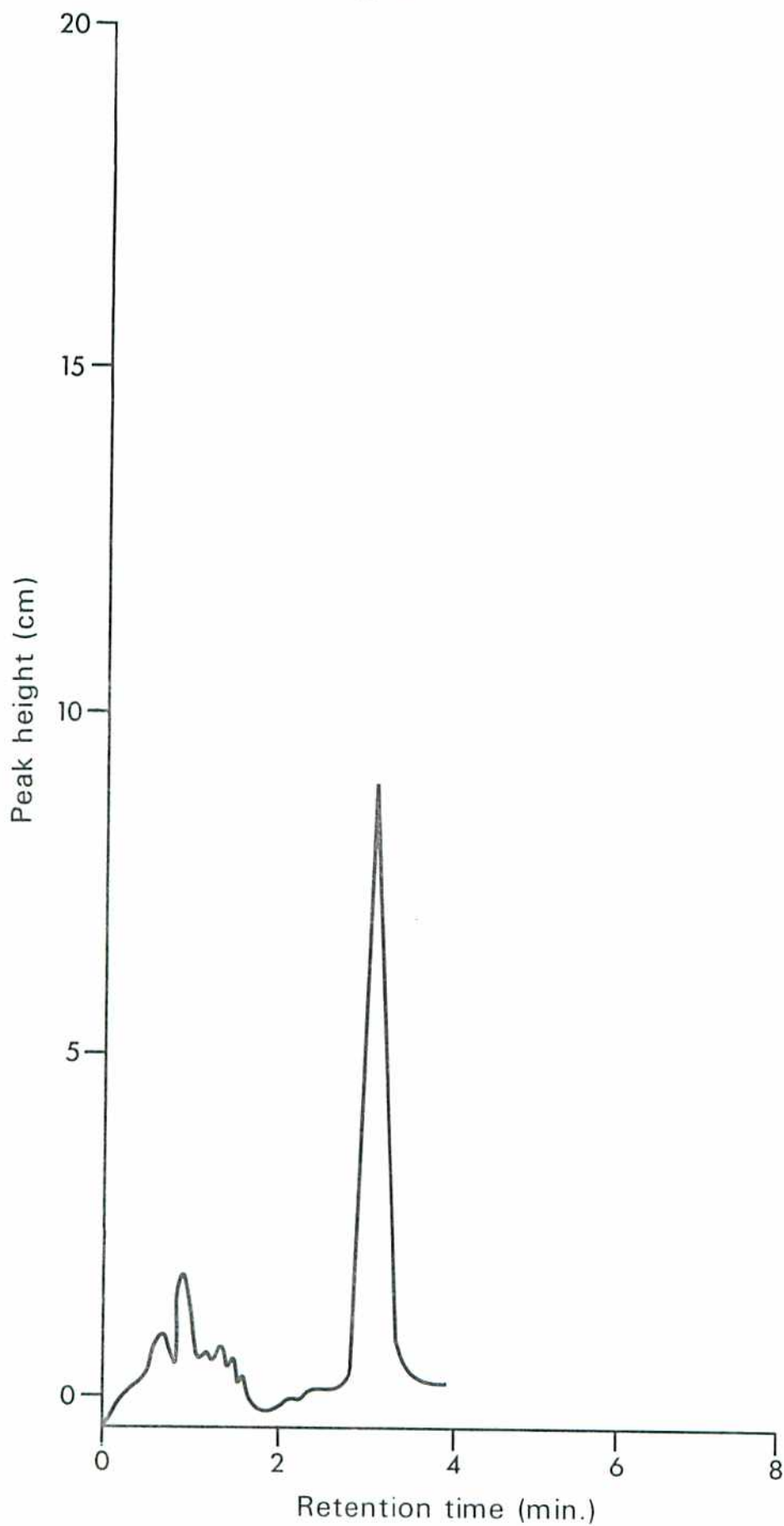


Fig. 5. Chromatogram of Cleanup Extract of Spruce Foliage Fortified with Aminocarb.

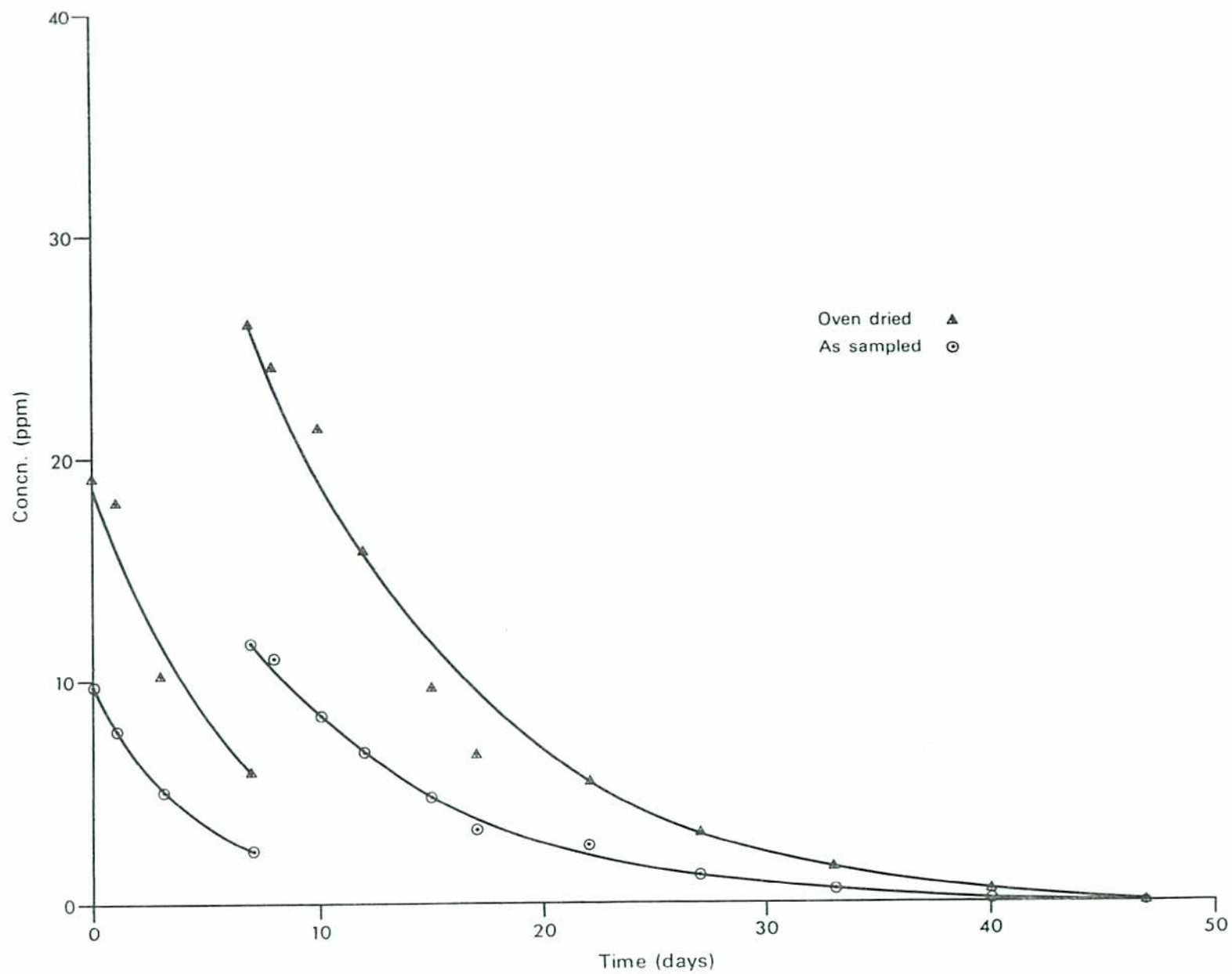


Fig. 6. Dissipation of Aminocarb in Spruce Foliage.

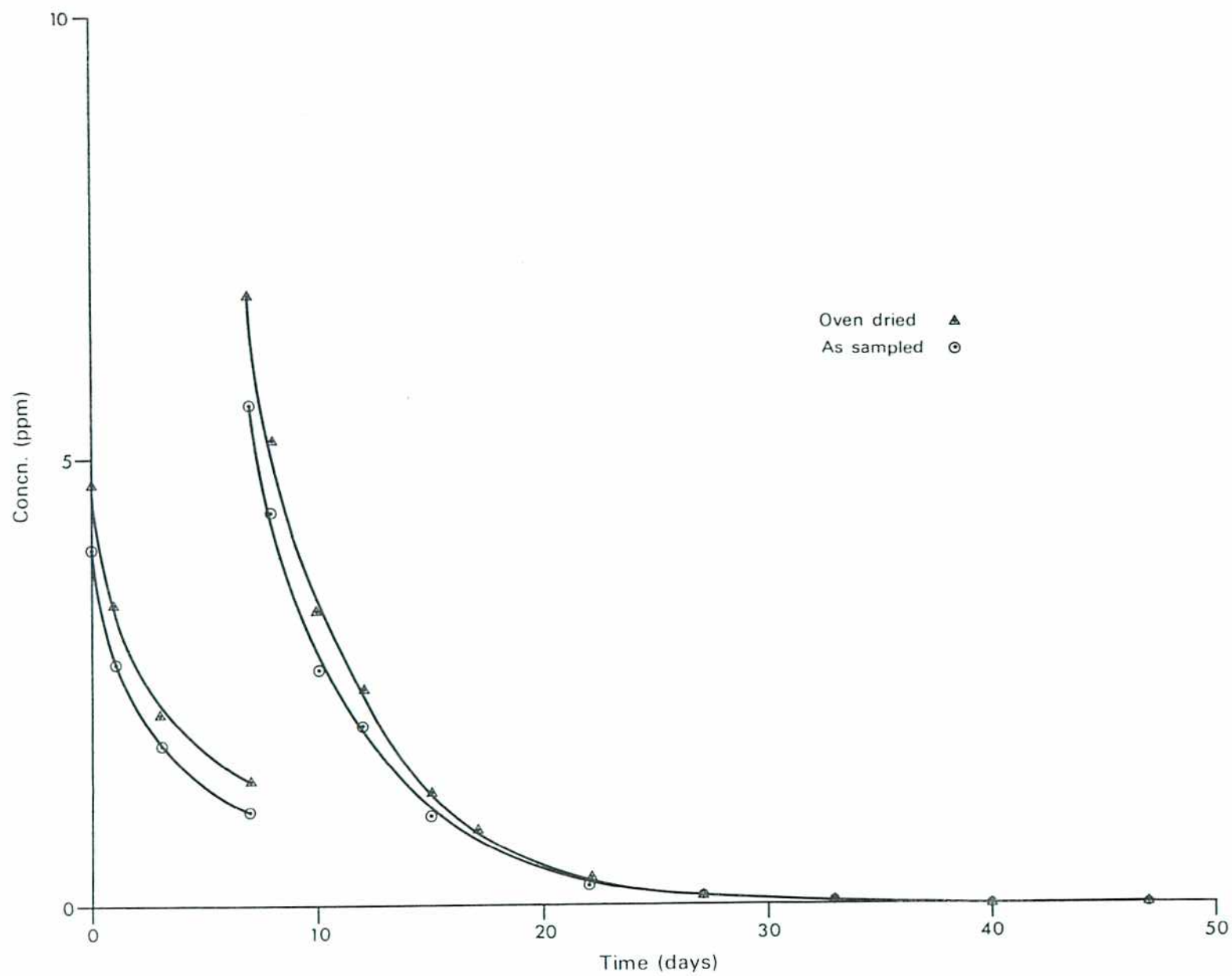


Fig. 7. Dissipation of Aminocarb in Partly Overshadowed Forest Soil.



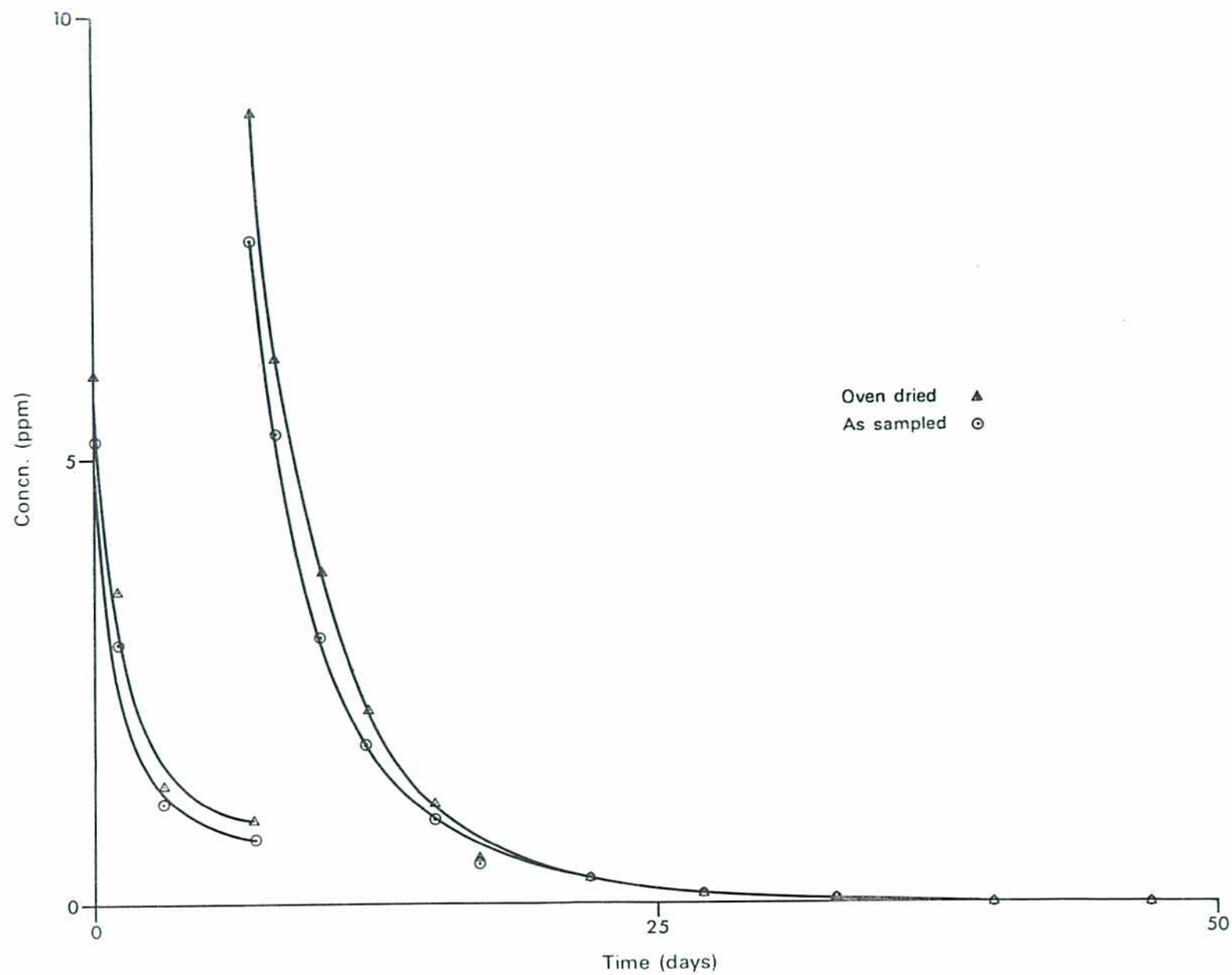


Fig. 8. Dissipation of Aminocarb in Open Forest Soil.

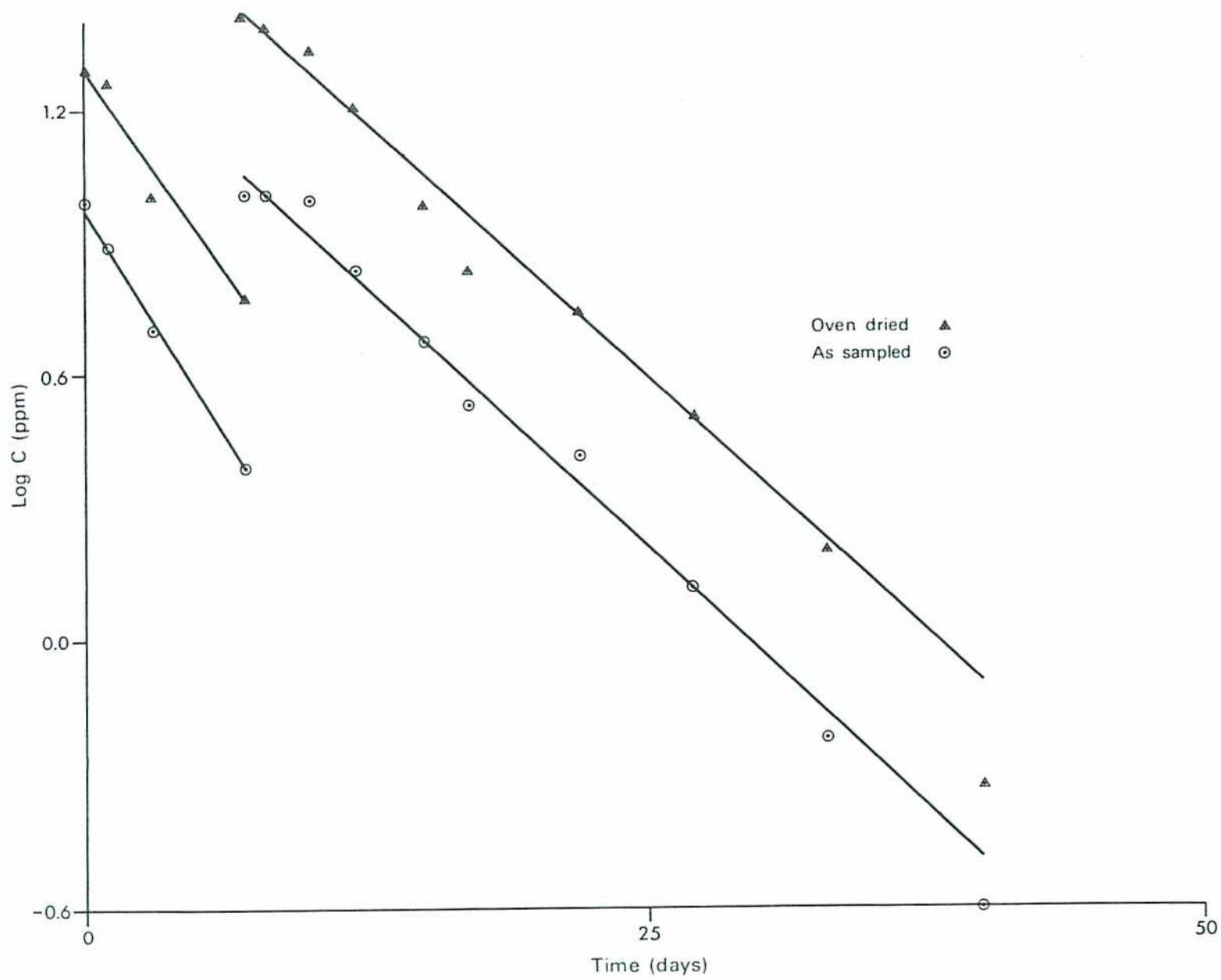


Fig. 9. Kinetic Behaviour of Aminocarb Dissipation in Spruce Foliage.

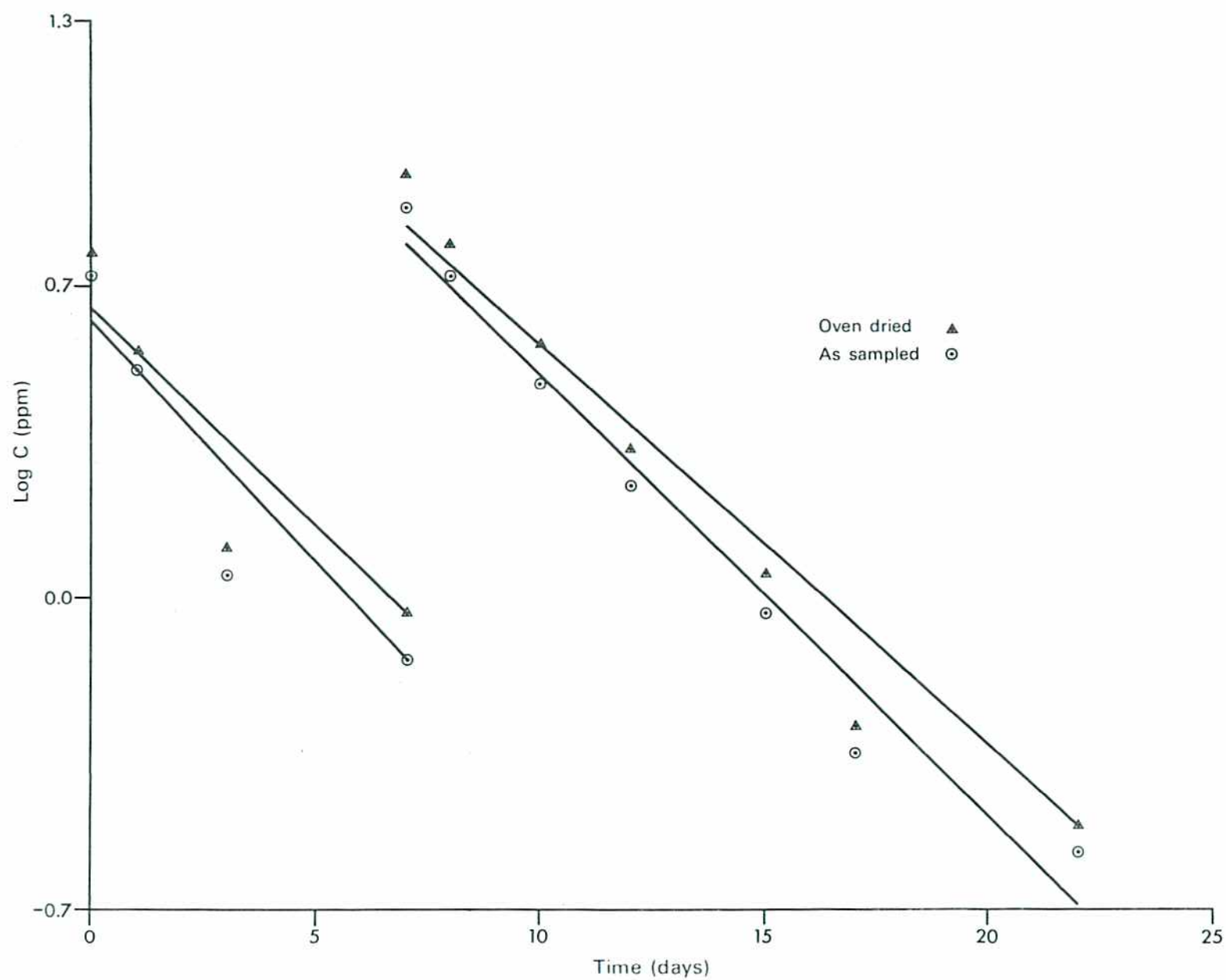


Fig. 10. Kinetic Behaviour of Aminocarb in Overshadowed Forest Soil.



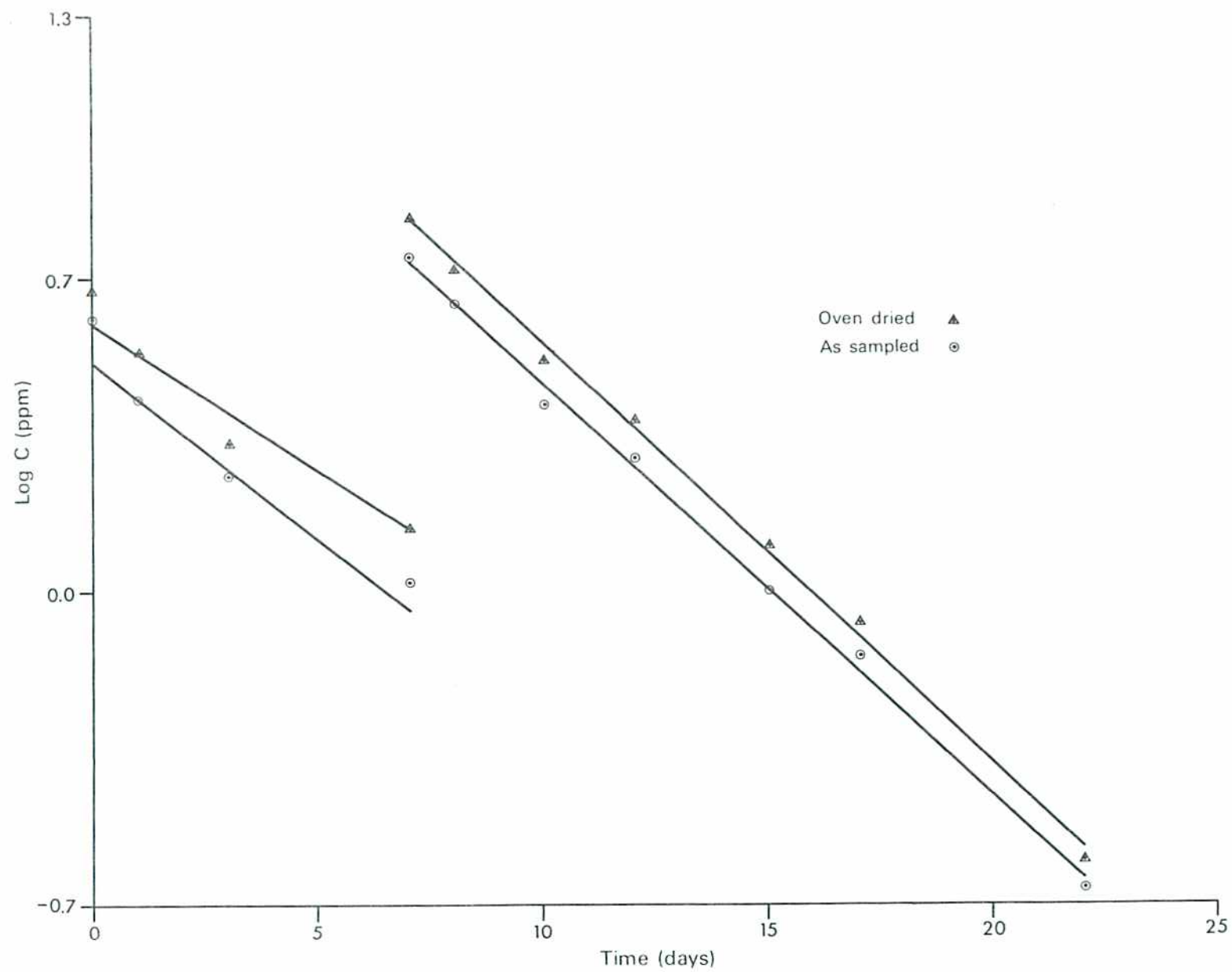


Fig. 11. Kinetic Behaviour of Aminocarb in Open Forest Soil.