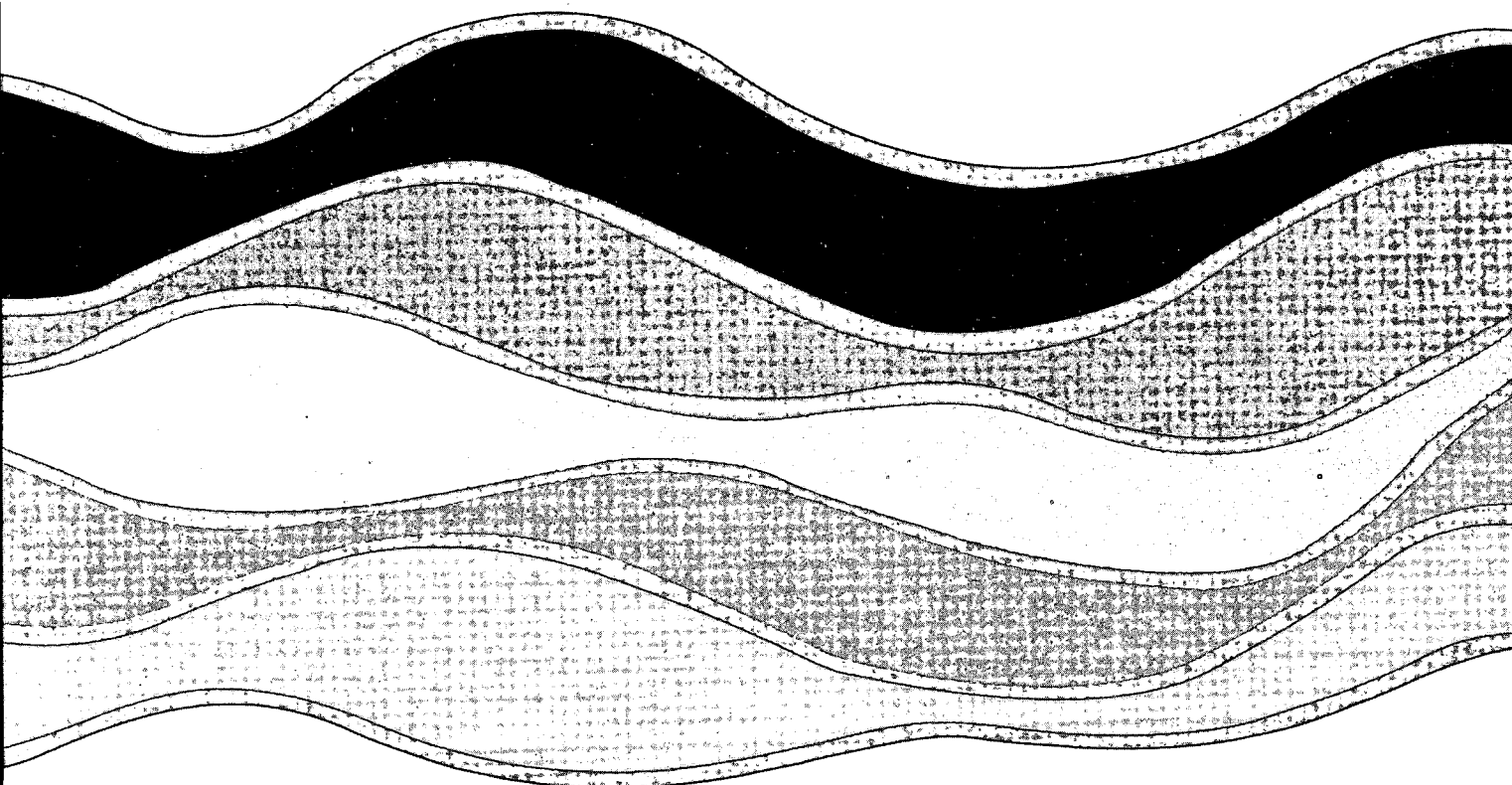
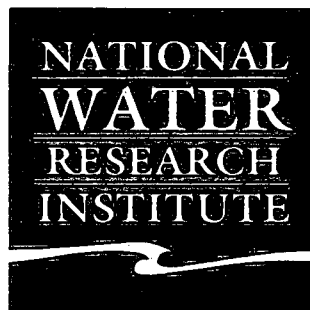
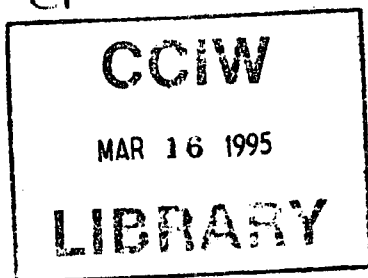


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**VOLATILIZATION OF METOLACHLOR
FROM WATER**

**Y.L. Lau, G.J. Pacepavicius, D.L.S. Liu
and R.J. Maguire**

NWRI CONTRIBUTION NO. 95-54

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VOLATILIZATION OF METOLACHLOR FROM WATER

Y.L. Lau, G.J. Pacepavicius, D.L.S. Liu and R.J. Maguire

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Department of the Environment
Burlington, Ontario L7R 4A6 Canada**

NWRI Contribution No. 95-54

MANAGEMENT PERSPECTIVE

The herbicide metolachlor [2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide] is the most heavily used herbicide in Ontario, accounting for about 25% of all herbicides used, on a weight basis. Both the Ecosystem Interpretation Branch of Environment Canada and the U.S. Environmental Protection Agency have noted that more information is required on the occurrence of metolachlor in water, its toxicity to aquatic organisms, and its persistence and fate, in order that an assessment can be made of the hazards of its use to aquatic ecosystems.

Our earlier work has shown that metolachlor is fairly stable to biological, chemical and photochemical degradation, and it is commonly assumed that volatilization of metolachlor from water is not a significant pathway because of its relatively high aqueous solubility and low vapour pressure at 20-25 °C. The volatilization of metolachlor from water was studied in the laboratory and in an outdoor open-channel experiment. As expected, volatilization was not significant at temperatures ≤ 25 °C. However, at temperatures ≥ 30 °C, there was significant volatilization (e.g., half-life of 20 days at 40 °C in unstirred solutions). Such conditions may occur in Canada in the summer in small ponds or irrigation ditches. In tropical countries, such high temperatures are common, and significant losses of metolachlor from water through volatilization may be expected. Additional experiments indicated that aeration of water significantly accelerated volatilization losses. Such air-stripping may be important in turbulent streams and rivers. The experiments reported here indicate the importance that ecosystem-specific characteristics can have on the persistence of environmental contaminants.

The results of this work, and our earlier work on toxicity, biological, chemical and photochemical degradation will be communicated to the Ecosystem Interpretation Branch for its consideration in setting a full Canadian Water Quality Guideline for metolachlor for the protection of aquatic life. The results will also be communicated to the Pesticides Division of the Commercial Chemicals Evaluation Branch, and to the U.S. Environmental Protection Agency, for their consideration in the periodic re-registration of metolachlor under the Pest Control Products Act and the U.S. Federal Insecticide, Fungicide and Rodenticide Act, respectively.

SOMMAIRE À L'INTENTION DE LA DIRECTION

Le metolachlore [2-chloro-N-(2-éthyl-6-méthylphényl)-N-(2-méthoxy-1-méthyléthyl)acétamide] est l'herbicide le plus utilisé en Ontario; il représente, en poids, environ 25% de tous les herbicides utilisés. La Direction de l'interprétation des écosystèmes d'Environnement Canada et l'Environmental Protection Agency des États-Unis ont indiqué que de plus amples informations sont requises sur la présence de metolachlore dans l'eau, sa toxicité pour les organismes aquatiques, de même que sur sa persistance et son devenir, de manière à évaluer les dangers qu'il représente pour les écosystèmes aquatiques.

Nos travaux précédents ont montré que le metolachlore est relativement stable à la dégradation biologique, chimique et photochimique, et on pense généralement que la volatilisation du metolachlore présent dans l'eau n'est pas une voie importante, étant donné la solubilité relativement élevée de ce composé dans l'eau et sa faible tension de vapeur à 20-25 °C. On a étudié la volatilisation du metolachlore dans l'eau au laboratoire et dans un canal découvert à l'extérieur. Comme prévu, la volatilisation n'était pas importante à des températures ≤ 25 °C. Toutefois, à des températures ≥ 30 °C, la volatilisation était notable (p. ex. demi-vie de 20 jours à 40 °C en solution non agitée). De telles conditions peuvent se présenter au Canada, en été, dans de petits étangs ou dans des canaux d'irrigations. Sous les tropiques, ces températures élevées sont courantes et se traduisent probablement par des pertes importantes de metolachlore dans l'eau par volatilisation. D'autres expériences ont indiqué que l'aération de l'eau accélérât significativement les pertes par volatilisation. Un tel entraînement par l'air peut être important dans les cours d'eau turbulents. Les expériences rapportées indiquent l'importance que peuvent

avoir certaines caractéristiques des écosystèmes sur la persistance des contaminants dans l'environnement.

Les résultats de ces travaux et de travaux antérieurs sur la toxicité de cette substance et sur sa dégradation par des processus biologiques, chimiques et photochimiques seront communiqués à la Direction de l'interprétation des écosystèmes qui les examinera pour établir une recommandation concernant la protection de la vie aquatique contre le metolachlore dans le cadre des Recommandations pour la qualité des eaux au Canada. Les résultats seront également communiqués à la Division des pesticides de la Direction de l'évaluation des produits chimiques commerciaux, ainsi qu'à l'Environmental Protection Agency des États-Unis, qui les examineront dans le cadre de la réhomologation périodique du metolachlore aux termes de la Loi sur les produits antiparasitaires, au Canada, et de la Federal Insecticide, Fungicide and Rodenticide Act, aux États-Unis.

ABSTRACT

The volatilization of metolachlor [2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide] from water was studied in the laboratory and in an outdoor open-channel experiment. As expected, volatilization was not significant at temperatures ≤ 25 °C. However, at temperatures ≥ 30 °C, there was significant volatilization (e.g., half-life of 20 days at 40 °C in unstirred solutions). This increased volatility reflected the rapid increase of the Henry's law constant with temperature. Additional experiments indicated that aeration of water significantly accelerated volatilization losses. Such air-stripping may be important in very turbulent streams and rivers and when water flows over hydraulic structures such as weirs. The experiments reported here indicate the importance that ecosystem-specific characteristics can have on the persistence of environmental contaminants.

RÉSUMÉ

La volatilisation du metolachlore [2-chloro-N-(2-éthyl-6-méthylphényl)-N-(2-méthoxy-1-méthyléthyl)acétamide] présent dans l'eau a été étudiée au laboratoire et dans un canal découvert à l'extérieur. Comme prévu, la volatilisation n'était pas importante aux températures ≤ 25 °C. Toutefois, à des températures ≥ 30 °C, la volatilisation était notable (p. ex. demi-vie de 20 jours à 40 °C en solution non agitée). Cette augmentation de la volatilité tient à l'augmentation rapide de la constante de la loi de Henry avec la température. D'autres expériences ont indiqué que l'aération de l'eau accélérerait significativement les pertes par volatilisation. Un tel entraînement par l'air peut être important dans des cours d'eau très turbulents et lorsque l'eau coule sur des structures hydrauliques tels des barrages. Toutes ces expériences indiquent l'importance que peuvent avoir certaines caractéristiques des écosystèmes sur la persistance des contaminants dans l'environnement.

Introduction

In today's intensively competitive market, a sustainable agriculture must maintain adequate productivity and reasonable profit for farmers. For this reason, pre-emergence and post-emergence herbicides are widely used to maintain agricultural productivity (Suntio *et al.*, 1988; Marcheterre *et al.*, 1988). Metolachlor [2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide] is one of the most widely used and important herbicides in the world (Chang *et al.*, 1994). In Canada, metolachlor is now the most heavily used herbicide in the Province of Ontario. In 1993, approximately 1,300,000 kg of metolachlor (as active ingredient) were used in Ontario on a variety of crops (Hunter and McGee, 1994). This amounted to 31% of all pesticides used that year in Ontario.

Inevitably, a small fraction of applied herbicides finds its way into the aquatic environment. The occurrence of metolachlor in surface waters has been noted. Concentrations have been reported in the range of 0.3-4.4 $\mu\text{g/L}$ for the period 1975-1985 at various locations along the Mississippi River and in the range of 0.8-1.8 $\mu\text{g/L}$ in the Sacramento River in California (Chesters *et al.*, 1989). Mean annual concentrations were up to 4.1 $\mu\text{g/L}$ at the mouths of the Grand, Saugeen, and Thames Rivers in Ontario in 1981-1985 (Frank and Logan, 1988). The mean concentration in 24 Yamaska River (Québec) basin samples in 1986-1987 was 1.7 $\mu\text{g/L}$ (Maguire and Tkacz, 1993).

There is little information in the open literature on the aquatic fate and persistence of metolachlor (U.S. Environmental Protection Agency, 1987; Kent *et al.*, 1991). Metolachlor has been shown to be fairly stable in natural waters, with little loss due to biological degradation or purely chemical degradation over periods of up to 6 months at room temperature (LeBaron *et al.*, 1988; Kochany and Maguire, 1994; Liu *et al.*, 1995). Although sunlight photodegradation is faster than purely chemical degradation, it appears also to be a relatively slow process, with estimated near-surface half-lives in lake water of 22 calendar days in summer and 205 calendar days in winter at 40° N latitude (Kochany and Maguire, 1994). Because metolachlor has a relatively high water solubility (530 mg/L at 20 °C), low vapour pressure (1.3×10^{-5} mm Hg) and thus a low Henry's law constant (0.9×10^{-8} atm m³/mol), it is considered to be essentially involatile under the usual environmental conditions (Chesters *et al.*, 1989; LeBaron *et al.*, 1988; Thomas, 1982). Experiments reported herein confirmed that assumption, with no volatilization of metolachlor from water at 22 °C over a period of two months. However, the possibility that volatilization could be important at higher temperatures was suggested by a further experiment. In this experiment, originally designed to study the biological degradation of metolachlor in an outdoor open-channel, the metolachlor concentration declined much more quickly than could be explained by biological, chemical or photochemical degradation. Adsorption to container walls was considered unlikely because of the relatively high aqueous solubility of metolachlor. Because the temperature in the outdoor experiment was sometimes greater than 35 °C, it was concluded that the rapid loss of metolachlor could have been due to volatilization at higher temperatures. Therefore, a series of laboratory experiments was carried out to investigate the effects of temperature as well as other environmental parameters such as turbulence and wind

on the rate of volatilization of metolachlor from water. The results are presented in this report.

Experimental Method

Materials

Metolachlor was obtained from Ciba-Geigy Canada Ltd. (Mississauga, Ont.). Pesticide grade organic solvents were obtained from Caledon Laboratories (Georgetown, Ont.). The sodium sulfate used for drying organic extracts was heated to 500 °C for 24 h before use. All glassware was rinsed with pesticide grade solvents before use. Organic-free water was prepared with a Milli-Q system from Millipore-Waters Co. (Mississauga, Ont.). The natural water used in the open-channel outdoor experiment was from nearby Hamilton Harbour.

Open-channel experiment

The open-channel experiment was conducted in an outdoor recirculating flow facility. A centrifugal pump delivered Hamilton Harbour water from a tank (450 mm x 450 mm x 550 mm high) into the upstream end of a trough (200 mm x 200 mm x 2 m long). The water flowed down the trough and emptied back into the tank. The depth of water in the trough was about 8 cm. All surfaces in contact with the water were manufactured from stainless steel. The water was spiked with a technical metolachlor standard in acetone to a concentration of approximately 14 mg/L. The pump was then started to initiate the open-channel flow. The average flow velocity in the trough was approximately 20 cm/s. Periodically, the pump was stopped and the

water loss through evaporation was monitored through a stilling well attached to the tank. Water was added to bring the volume in the system back to its original level and then, after a brief mixing period, samples were taken for metolachlor analyses. Procedures for sampling and for analysis of metolachlor are given in the next section describing the laboratory experiments. Measurements were made over a three-week period in the summer of 1993.

Laboratory experiments

Preliminary volatilization experiments were done in test tubes. Forty mL of organic-free water containing 68 mg/L metolachlor were added to 50 mL test tubes, and incubated in the dark at 22 °C for periods up to 64 days. The solutions were poisoned with 25 mg/L HgCl_2 to inhibit biological degradation. At each sampling interval (1 hour, 1, 2, 4, 8, 16, 32 and 64 days), the contents of the test tubes were weighed to calculate water loss, and then the entire contents were extracted three times with 2 mL of hexane each time. The hexane extracts were combined and dried by passage through sodium sulfate. Two mL of toluene was added as a "keeper", and the volume of the extract was reduced to 2 mL and analyzed without further cleanup. The experiment was conducted in triplicate, and the appropriate reagent blanks and procedural blanks were performed to check for chemical and biological degradation. Subsequent analysis showed constant recoveries of metolachlor over a 64 day period, indicating no loss by volatilization. Further laboratory experiments described below did not employ the HgCl_2 microbial inhibitor because it had been shown that metolachlor was stable to chemical and biological degradation over at least 100 days (Kochany and Maguire, 1994; Liu *et al.*, 1995).

In further experiments, glass beakers containing organic-free water were spiked with metolachlor, and the solutions were sampled periodically to measure any changes in concentration. The beakers and contents were weighed prior to sampling to account for water loss through evaporation. The experiments were carried out at four different water temperatures, and under the following conditions: unstirred, stirred (magnetically), with air bubbling, and with a fan blowing air over the beakers. The experimental conditions are listed in Table 1. For the experiments at 30 °C and 40 °C, the temperatures were maintained by immersing the beakers in a water bath. The other experiments were carried out at room temperature and the values listed in Table 1 represent the average room temperatures during the tests. In test no. 9, the wind blowing over the beaker had a speed of approximately 40 cm/s and did not create any visible disturbances on the water surface. In test no. 2, air was bubbled through the solution via a 3 mm diameter tubing. The air flow rate was not measured but was estimated to be less than 10 ml/minute.

At each sampling interval, three 10 mL samples were removed from the system and then 10 mL of dichloromethane was added to each sample. The samples were extracted with 3 x 10 mL aliquots of dichloromethane, dried by passage through sodium sulfate, and concentrated, with solvent exchange, to 2 mL of toluene, and analyzed without cleanup.

Analysis for metolachlor

Analyses for metolachlor were performed with a Hewlett-Packard 5890 gas

chromatograph with a single splitless injector - dual column - dual detector (nitrogen-phosphorus and flame ionization) technique. Injection volume was 2 μL . Both columns were DB-5 (Chromatographic Specialties, Brockville, Ont.). Column dimensions were 0.25 mm i.d. x 30 m in length, with 0.25 μm film thickness. Injector and detector temperatures were 200 and 300 $^{\circ}\text{C}$, respectively. The initial column temperature was 80 $^{\circ}\text{C}$ (2 minute hold), and the program rate was 10 $^{\circ}\text{C}/\text{minute}$ to 150 $^{\circ}\text{C}$, then 4 $^{\circ}\text{C}/\text{minute}$ to 280 $^{\circ}\text{C}$, then 8 $^{\circ}\text{C}/\text{minute}$ to 300 $^{\circ}\text{C}$, followed by a 5 minute final hold. The helium carrier gas was maintained at 1 mL/minute with electronic pressure programming. A standard mixture of metolachlor was prepared and used to calibrate retention times and detector responses. The presence of metolachlor was taken to be confirmed if it eluted within the appropriate chromatographic "window" on both columns. Quantification was done with the flame ionization detector.

Results and Discussion

The results of the open-channel experiment are shown in Figure 1. The half-life was about 9 days, much faster than would be expected on the basis of biological, chemical or photochemical degradation. It should be noted that none of the common degradation products identified in earlier studies (Kochany and Maguire, 1994; Liu *et al.*, 1995) was found in the water samples. It should also be noted that the water temperature in this experiment reached at least 35 $^{\circ}\text{C}$, and that there was air entrainment as the water plunged from the trough into the tank. Therefore, additional laboratory experiments were conducted to investigate the effects of temperature, turbulence and wind on the volatilization rate.

Results from the still-water experiments in organic-free water at four different temperatures are shown in Figure 2. The metolachlor concentrations, C , have been non-dimensionlized with the initial concentrations, C_0 , and Figure 2 is a semi-log plot of C/C_0 versus time. There was very little or no loss of metolachlor from water over a period of 45 days at 18 °C or 25 °C. There was, however, significant loss of metolachlor from water at temperatures \geq 30 °C. At 40 °C, half of the metolachlor was lost in about 20 days. As was the case with the open-channel outdoor experiment, no degradation products were detected. Consequently, it was concluded that the loss was due to volatilization and that the volatitlity of metolachlor increased substantially with temperature.

The loss of metolachlor followed first-order kinetics with, as expected, no dependence of the rate constant on initial metolachlor concentration. Practically identical results were obtained in tests 5-7, all at 30°C, in which C_0 varied over two orders of magnitude.

The increase in volatility of metolachlor with increasing temperature likely reflected an increase in the Henry's law constant (the ratio of vapour pressure to aqueous solubility) of metolachlor with temperature. Mackay and Wolkoff (1973) developed a model for estimating the volatilization of a chemical from water in which the first-order rate constant, k , is given by the following expression:

$$k = \frac{EM_i 10^6 H}{18GP_w} \quad (1)$$

where E is the water evaporation rate, M_i is the molecular weight of the chemical (284 for metolachlor), G is the mass of the solution, P_w is the partial vapour pressure of water, H is the Henry's law constant, and the number 18 is the molecular weight of water.

The rate constants for the tests at 25, 30 and 40°C were calculated from the concentration-time data in Figure 2. The values of H were then obtained from equation (1). These values are listed in Table 2 together with the value of H at 20°C which is calculated from published values of vapour pressure and water solubility (Chesters et al., 1989). The increase of H with temperature is very evident.

There are few studies on the variation of H with temperature for environmental contaminants. In one such study, ten Hulscher *et al.* (1992) determined the values of H for several chlorobenzenes, PCB congeners and PAH compounds over a temperature range of 10 to 55 °C and found that the data could be modelled with the van't Hoff equation for temperature effect on an equilibrium constant, *i.e.*,

$$\ln H = a - \frac{b}{T} \quad (2)$$

in which T is the absolute temperature and a and b are constants.

Figure 3 is a plot of $\ln H$ vs. $1/T$ using the data from Table 2. The data fit fairly well to a straight line, indicating that the temperature dependence of H for metolachlor can also be described by equation (2).

The effect of variables other than temperature was also assessed. Figure 4 shows the effect of wind in increasing the rate of loss of metolachlor from water at 40 °C. The rate constant was more than doubled when wind was blown over the beaker, even though the wind velocity was not large enough to produce any visible disturbances on the water surface. Coincidentally, the rate of water loss also increased. Similarly, in tests 3 and 4, the solution which was stirred showed both increased metolachlor loss and water loss.

The effect of bubbling air through the solution on the rate of volatilization of metolachlor was also assessed. Figure 5 shows the results of tests 1 and 2, both at 18 °C. It is apparent that air bubbling was very efficient in stripping the metolachlor from the solution. After 26 days, almost 80% of the metolachlor had been lost, whereas in the still water case (test 1), there was practically no loss. As noted above, in the open-channel outdoor experiments, air was entrained in the water as it plunged from the trough into the tank. Consequently, the rapid loss of metolachlor from water in the open-channel experiment was probably due to air-stripping as well as the effect of higher temperatures.

In summary, although metolachlor is generally regarded as involatile under the usual environmental conditions, at temperatures greater than 30 °C its volatility from water is greatly

increased. Such conditions may occur in Canada in the summer in small ponds or irrigation ditches. In tropical countries, such high temperatures are common, and significant losses of metolachlor from water through volatilization may be expected. Moreover, air-stripping of metolachlor from water may be important in turbulent streams and rivers and whenever water flows over hydraulic structures where air entrainment often occurs. The experiments reported here indicate the importance that ecosystem-specific characteristics can have on the persistence of environmental contaminants.

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Table 1. Summary of experimental conditions in laboratory experiments.

Test No.	Temperature (°C)	Initial Metolachlor Concentration (mg/L)	Test Condition	Water Evaporation Rate (g/day)
1	18	10.5	Still water	19.7
2	18	10.6	Air bubbling	18.8
3	25	7.7	Still water	6.7
4	25	7.6	Stirred	13.7
5	30	0.4	Still water	47.5
6	30	3.9	Still water	48
7	30	39.9	Still water	47.5
8	40	11.4	Still water	121.7
9	40	11.4	Blowing wind	225.2

Table 2. Values of Henry's Law constant estimated from experimental data.

Temperature (°C)	40	30	25	20
H (atm m ³ /mol)	1.98×10^{-7}	3.8×10^{-8}	1.39×10^{-8}	$0.9 \times 10^{-8}^a$

^acalculated from published values of vapour pressure and water solubility

Figure Captions

Figure 1. Decrease in metolachlor concentration with time in open-channel experiment.

Figure 2. Effect of temperature on metolachlor loss rate.

Figure 3. Variation of Henry's law constant for metolachlor with temperature.

Figure 4. Effect of "wind" on volatilization of metolachlor from water at 40 °C.

Figure 5. Effect of air bubbling on rate of volatilization of metolachlor from water at 18 °C.

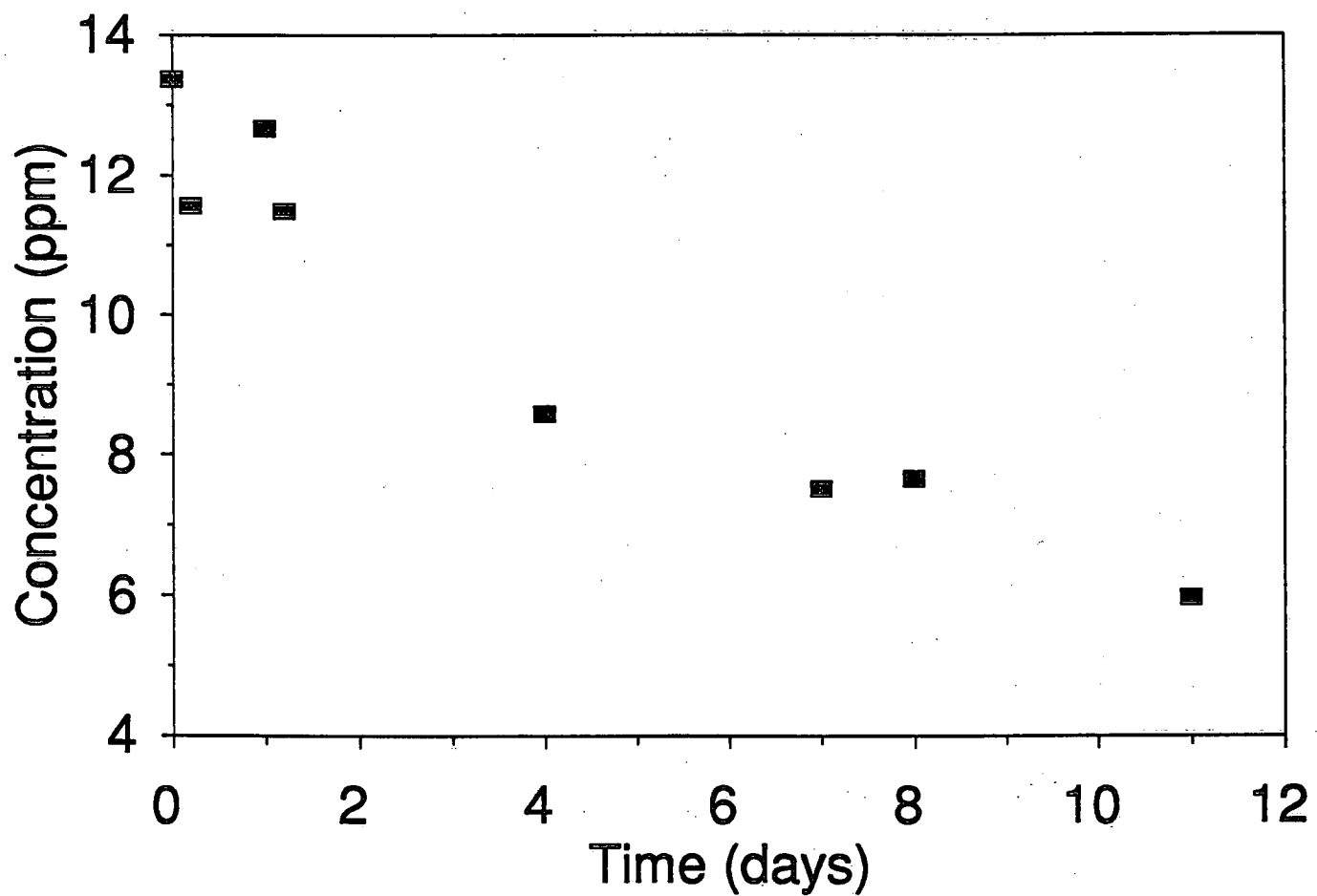
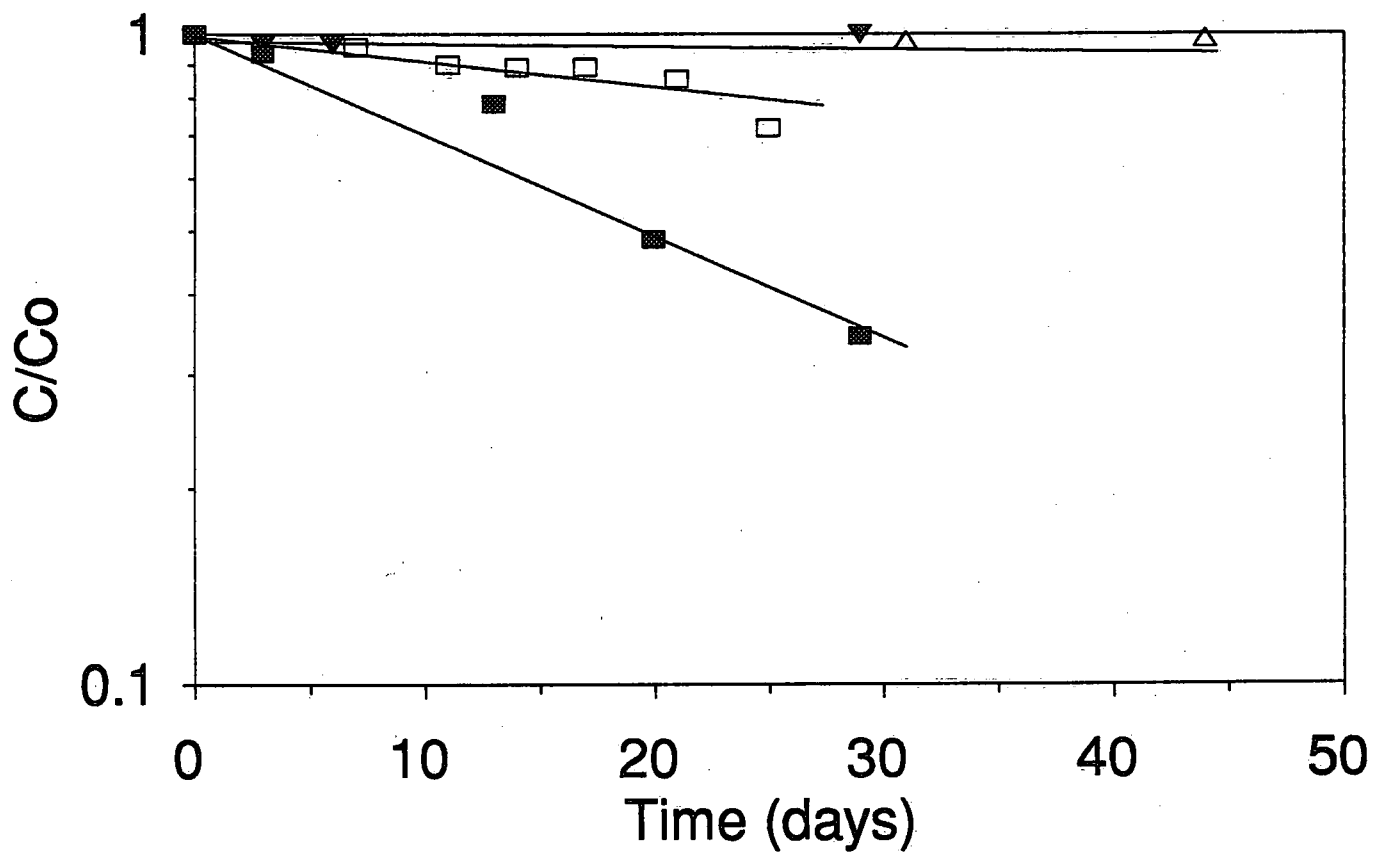


Figure 1. Change in metolachlor concentration with time in open-channel experiment.



▼ 18 deg △ 25 deg □ 30 deg ■ 40 deg

FIGURE 2.

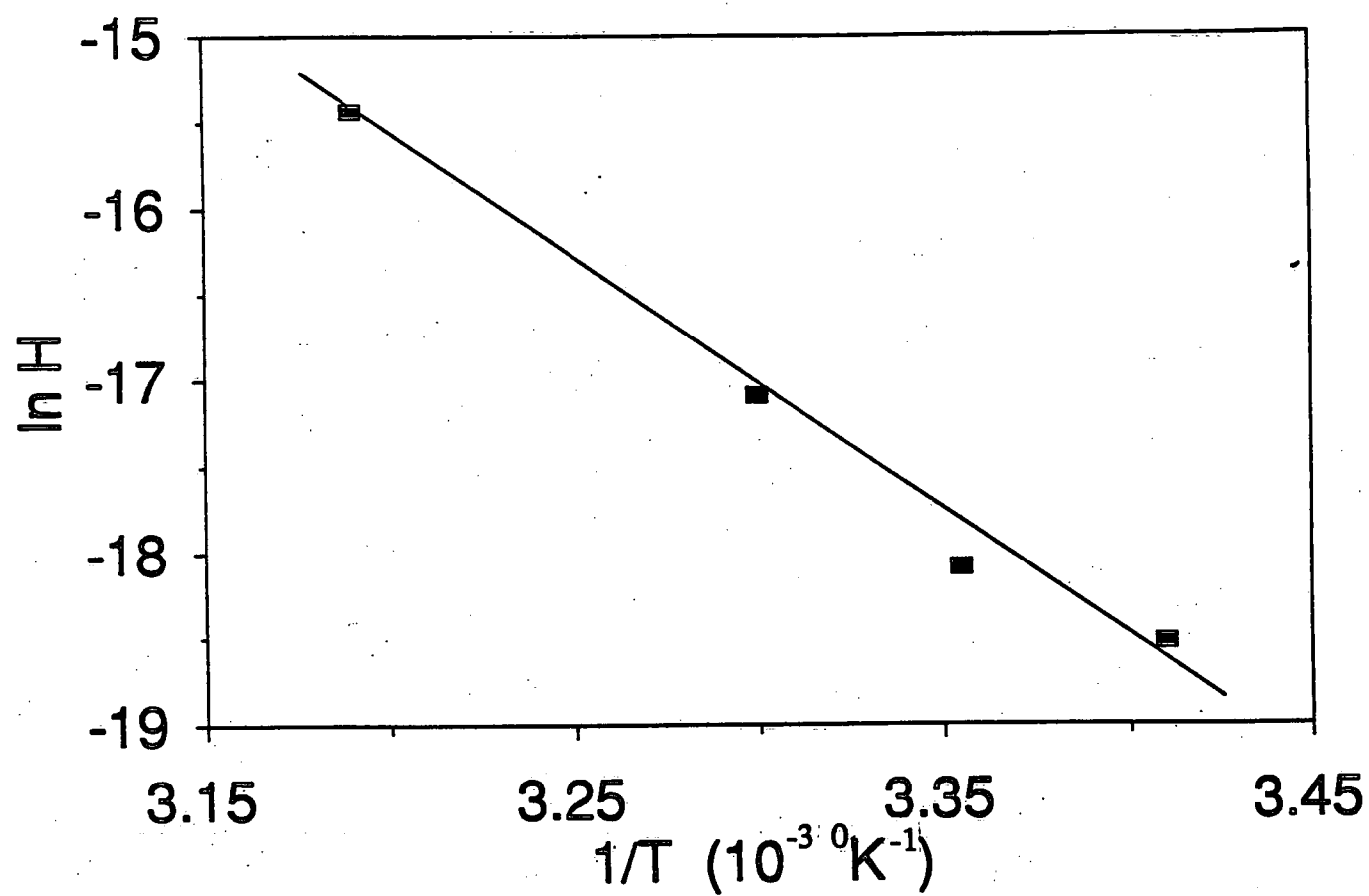


FIGURE 3.

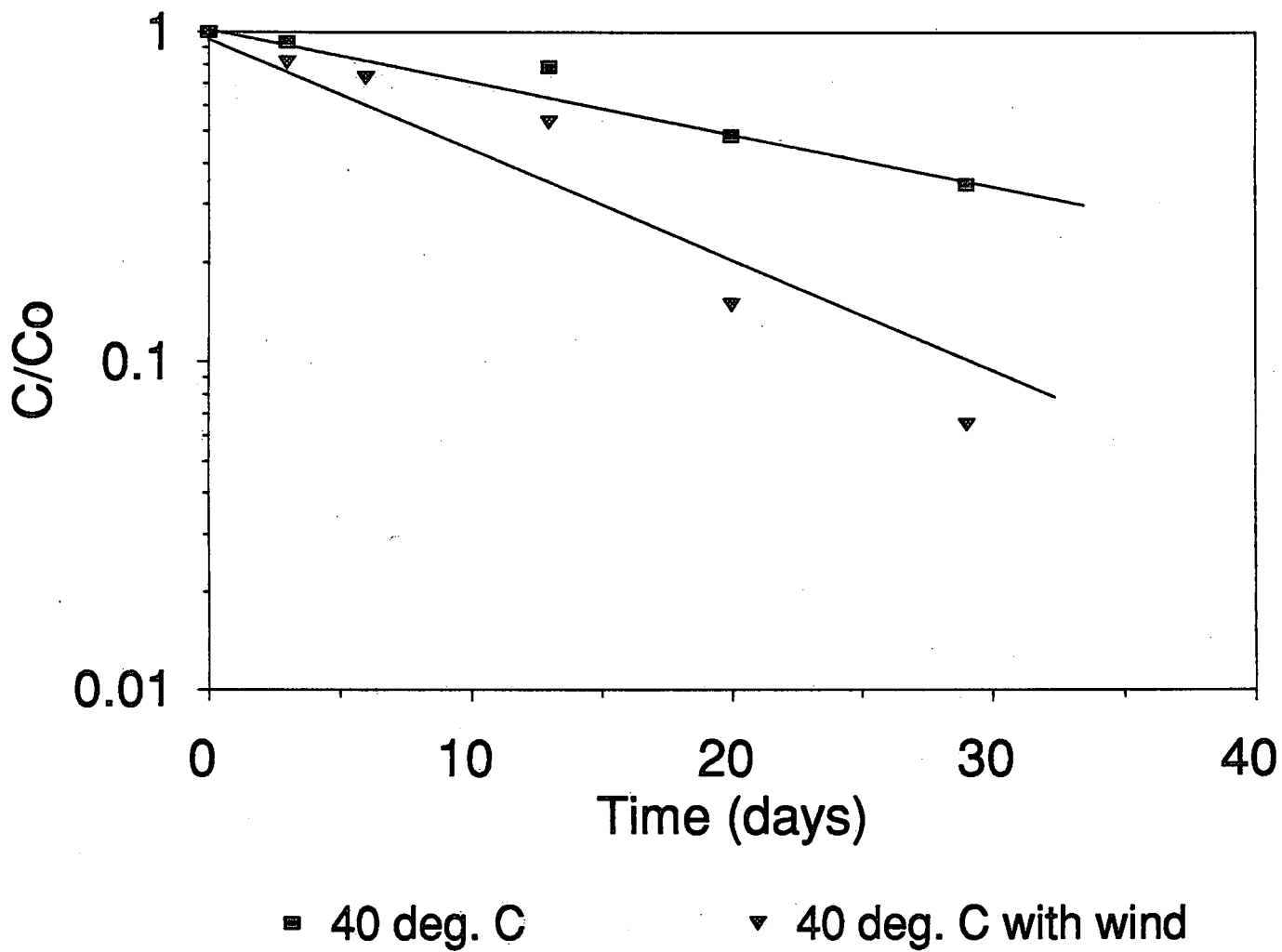


FIGURE 4.

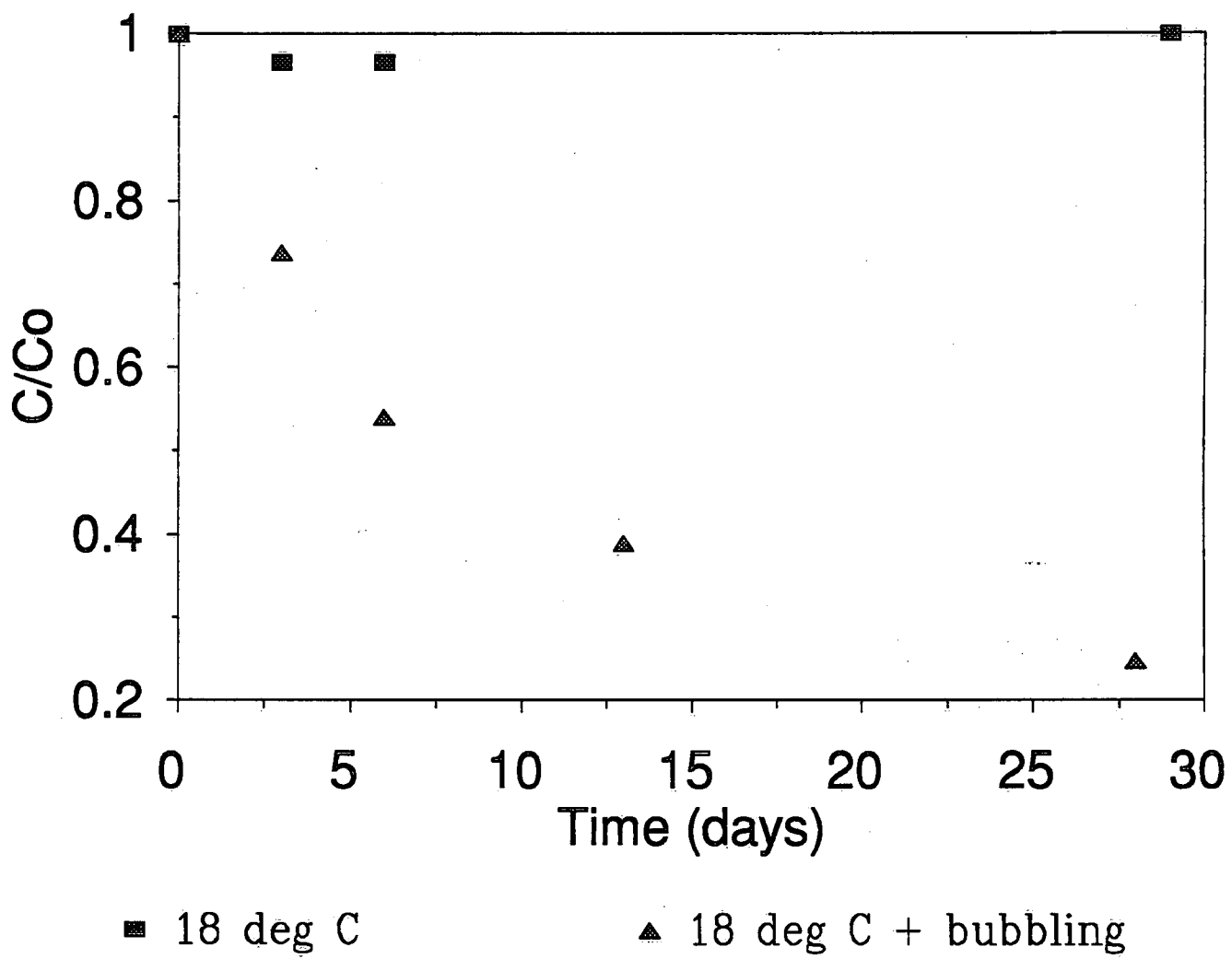
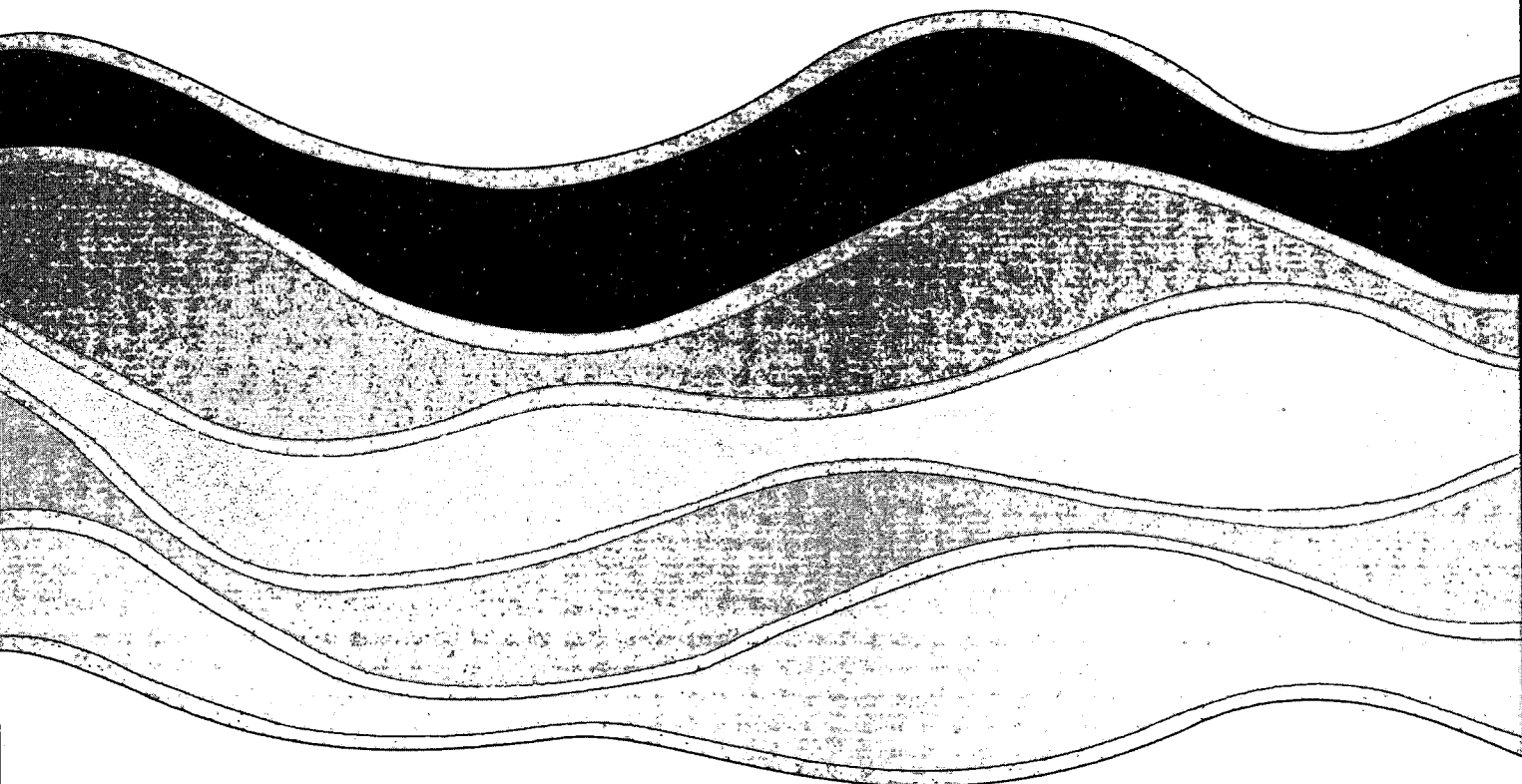


FIGURE 5.

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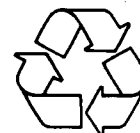
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