Environmental Polychlorinated Biphenyl Contamination near Sites of Manufacture and Use

prepared by

Environmental Science and Engineering, Inc.
P.O. Box 1354, Gainesville, Florida 32604

for

OFFICE OF TOXIC SUBSTANCES
ENVIRONMENTAL PROTECTION AGENCY
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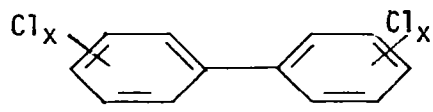
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INTRODUCTION

Polychlorinated biphenyls (PCBs) have a basic chemical structure as follows:



From one to ten chlorine atoms may be attached to the structure resulting in 210 possible isomers. PCB's have certain physical properties; including high heat capacity, chemical stability, and excellent dialectric properties which make them highly desirable for a number of industrial uses. Concern has been expressed in recent years over the widespread dispersion of PCB's in the environment. These compounds are suspected to have significant adverse effects on organisms at the top of the food web, including man. Biomagnification of this contaminant in natural predator-prey food chains has been documented by numerous studies. To complicate the problem, the rate of degradation of PCB's in the environment, particularly the higher substituted isomers, is postulated to be extremely slow. This situation allows for the possibly significant accumulation of these persistent chlorinated organic compounds in the environment. A thorough discussion of the problem and of the environmental data that are available has been presented by Selikoff (1972) and the U.S. Government Interdepartmental Tasks Force (1972).

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In response to the rising concern over PCB's in the environment, the single domestic manufacturer of these materials (Monsanto Co.) has voluntarily taken two actions to reduce the environmental hazard. The trade name for these domestically-manufactured compounds is Aroclor. Beginning in 1970, the production of Aroclor was voluntarily reduced by the manufacturer and supply was discontinued to those users who could not control release to the environment. Production of the more highly chlorine substituted Aroclor mixtures has since been curtailed or discontinued and a lower-substituted, refined mixture (Aroclor 1016) has been introduced. Some researchers have concluded that the lower substituted PCB isomers do not persist as long in the environment as the higher substituted isomers, and that they may be amenable to biological degradation. Aroclors are still employed in electrical capacitors, transformers, vacuum pumps and gas-transmission turbines (Hutzinger, Safe and Zitko, 1974).

There are a number of foreign producers of PCB. The major foreign products are listed in Table 1. Some domestic industries, whose supply of Aroclor has been curtailed by the producer, have turned to foreign supplied PCB's. The extent of the use of these foreign products in the U.S. is not know at the present time.

The Environmental Protection Agency, Office of Toxic Substances has undertaken an environmental monitoring program to assess the magnitude and potential hazard of PCB contamination of the environment and to assess the effectiveness of voluntary controls on use of these compounds. The first phase of this monitoring program has been completed. The initial effort was directed toward the evaluation of PCB contamination

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Table 1. THE WORLD'S MAJOR PRODUCERS OF PCB*

Producer	Country	Tradename of PCB
Monsanto	U.S.A. and Great Britain	Aroclor
Bayer	Germany	Clophen
Prodelec	France	Phenoclor and Pyralene
Kanegafuchi	Japan	Kanechlor
Mitsubishi-Monsanto	Japan	Santotherm
Caffaro	Italy	Fenclor
Sovol	U.S.S.R.	
Chemko	Czechoslovakia	

*after Hutzinger, Safe and Zitko (1974).

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of soils in the vicinity of potential industrial sources and of waste-waters from these sources.

Soil samples have been taken in the vicinity of Yates Manufacturing Co. in urban Chicago, Illinois; Valcast, Inc. in Troy, Michigan, a suburb of Detroit; and the Monsanto Co., Krummrich Plant in Sauget, Illinois, near East St. Louis. Prior to 1972, the Yates Manufacturing Co. used Aroclors 5460, 6090 and 5442 in the production of wax mold material for the investment casting (lost wax) process. These mixtures contain both PCB and polychlorinated terphenyl (PCT). Subsequent to 1972, this company has been purchasing a PCB of foreign origin. Fenclor DK, a product of Caffero Corporation in Milan, Italy, has been substituted for the unavailable Aroclors. Valcast, Inc. is an investment casting facility. It has been reported that Valcast purchases the necessary wax compounding materials from Yates Manufacturing Co. The Krummrich Plant of Monsanto Co. is the only PCB production facility in the U.S. All domestic Aroclors are produced at this location.

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EXPERIMENTAL

Sampling Methods

Surface soil samples were collected at each site from the plant boundary to a distance of approximately one mile at ¼ mile intervals in each direction subject to the physical constraints encountered. At each sample point, five sub-samples were collected within a radius of 10 m to a depth of 2.5 cm over a 100 cm^2 area. This mode of sampling, which was designed to collect a homogeneous surface soil sample representative of the chosen sample point, is illustrated in Figure 1. Vegetative matter was removed with forceps and the five sub-samples were composited and thoroughly mixed. Every attempt was made to select sample points where surface vegetation was sparse and where it appeared unlikely wind scour would be a major soil transport factor. Samples were placed in pre-cleaned, wide-mouth glass jars with aluminum foil-lined closures.

Water samples were collected from a drainage ditch adjacent to the Valcast plant, of Valcast effluent cooling water and from the sanitary sewer system downstream of Valcast. Wastewater samples were taken from the influent and effluent of the Sauget, Illinois sewage treatment plant. The Monsanto facility uses this treatment plant for wastewater treatment. These latter water samples were 24-hour composites of the treatment plant influent and effluent; the former were grab samples. All samples were taken in 1 l. glass containers, composited and stored in 4 l. pre-cleaned glass-stoppered reagent bottles.

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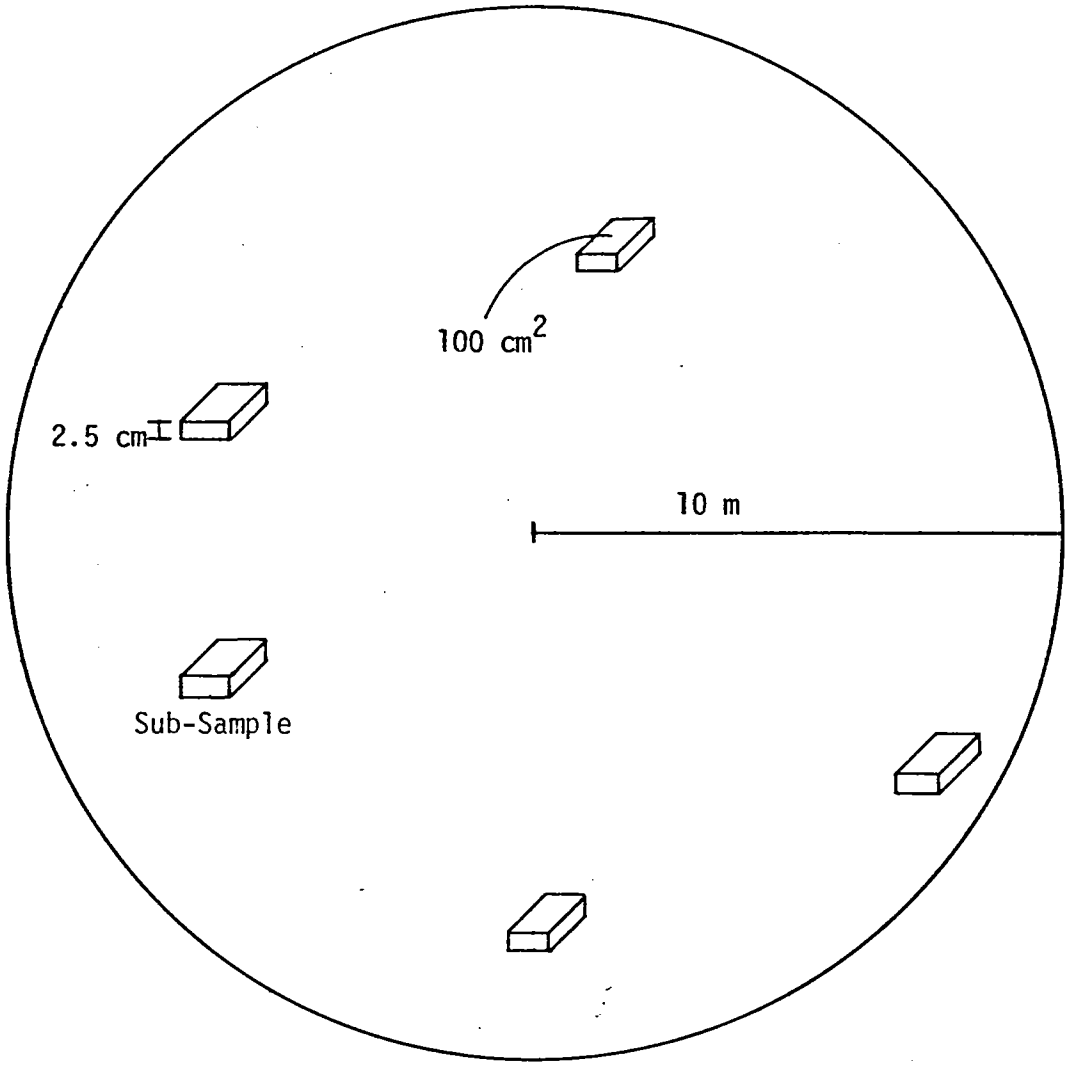


Figure 1. Diagram showing surface soil sampling method. Not to scale. Five sub-samples are composited to yield a single sample.

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Extraction and Concentration

The soil sample is thoroughly mixed and sectioned into quarters. Two of the quarters are rejected and the remaining quarters are again mixed and quartered. This is continued until a representative sample size of about 50 g is obtained. This sample fraction is allowed to air dry at room temperature. PCBs are extracted from the soil using a soxhlet apparatus and 75 ml of 1:1 nanograde hexane/acetone for a 10 g soil sample. The extract is concentrated to 5 ml on a Kuderna-Danish apparatus and dried with sodium sulfate. This concentrate is evaporated to dryness under dry N_2 at room temperature then redissolved in 1 ml of nanograde hexane. This procedure basically follows that described by the EPA (1974).

Water samples are triple extracted with 15 percent nanograde ehtyl ether in hexane, dried with sodium sulfate, concentrated on a Kuderna-Danish apparatus, evaporated to dryness under N_2 and redissolved in 1 ml nanograde hexane. This method is similar to that recommended by the EPA (1973).

Sample Cleanup

Both the soil and water extracts are cleaned-up by the use of a silica-gel column. The column is eluted with nanograde pentane followed by nanograde benzene. The PCBs present in the extract appear in the pentane fraction; pesticides appear in the benzene fraction. The pentane fraction is further treated with concentrated H_2SO_4 to eliminate unstable organic compounds. The silica gel separation procedure is described by Snyder and Reinert (1971). The sulfuric acid cleanup was adapted from Murphy (1972).

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Analytical Methods

All analyses are by gas chromatography. A dual-column Varian 2760 instrument is employed with the following conditions:

a) detection and quantifications

glass column, 6' length, 1/8 ID
1.5%/1.95% OV-17/QF-1 liquid base
Chrom W-HP, 80/100 mesh support
N_2 38 psig inlet pressure, 68 ml/min
detector temperature 225° C
column temperature 200° C
injector temperature 215° C

b) confirmation

stainless steel column, 5' length, 1/8 ID
1.5% OV-101 liquid phase
Chrom G-HP, 100/120 mesh support
N_2 30 psig inlet pressure, 43 ml/min
detector temperature 225°C
column temperature 205° C
injector temperature 210° C

Detection is by electron capture using a tritium detector. Mass spectrographic confirmation has also been obtained on selected samples.

The PCBs are quantified in a two-step process. First, the elution pattern is compared to those of standard mixtures and the closest pattern match is considered to be the PCB present. The relative peak heights in a mixture are a good indication of a match with a standard, as is the presence or absence of later eluting peaks. Good matches with standard Aroclor mixtures have been obtained. The presence of decachlorobiphenyl is easy to ascertain since it is a single peak. Once the elution pattern is subjectively matched to a standard PCB pattern, quantification is conducted by electronic integration of peak areas using a Hewlett-Packard Integrator Model 3380A. The total area of all accepted matching peaks in the elution pattern of the sample is compared

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with the total area of the same peaks in the standard mixture and a sample concentration is calculated.

The Aroclors used for preparation of standards were obtained from the Monsanto Company and the EPA Southeast Environmental Research Laboratory. Fenclor DK was provided by EPA Region V personnel.

Sample Replication and Recovery Studies

A number of replicate analyses were conducted to determine the reproducibility of both the analytical method and the sampling method. The results of these replications are tabulated in Tables 2 and 3. All replications conducted to date are of soil samples. Table 2 lists the results of the analysis of two fractions of a single soil sample. Table 3 lists the results of the analysis of replicate samples and, hence, represents both sampling and analytical variability.

The results of several PCB recovery studies are tabulated in Table 4.

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Table 2. ANALYTICAL REPLICATES OF SOIL SAMPLES

Replicate	Aroclor 1260 (ppm)	Decachlorobiphenyl (ppm)
Y-1	0.86	0.65
	0.51	0.50
	mean 0.69	mean 0.58
V-1	<0.01	
	0.11	
	mean 0.06	
V-2	0.07	
	<0.01	
	0.01	
	mean 0.04	
V-3	0.13	
	0.11	
	mean 0.12	
V-4	0.13	
	0.15	
	mean 0.14	
V-5	<0.01	
	<0.01	
	<0.01	
V-6	<0.01	
	0.03	
	0.04	
	mean 0.02	
M-1	0.06	0.084
	0.13	0.16
	mean 0.10	mean 0.12
M-2	0.21	0.066
	0.30	0.096
	mean 0.26	mean 0.081
M-3	1.8	0.95
	0.95	0.84
	mean 1.4	mean 0.90
M-4		0.49
		0.67
		mean 0.58

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Table 3. SAMPLE REPLICATES OF SOIL SAMPLES

Replicate	Aroclor 1260 (ppm)	Decachlorobiphenyl (ppm)
Y-1	0.53	1.0
	0.40	1.4
	mean 0.47	mean 1.2
M-1	9.8	1.3
	9.3	1.0
	mean 9.6	mean 1.2
M-2	0.12	0.015
	0.46	0.069
	mean 0.29	mean 0.042
M-3	0.23	0.64
	<0.01	0.58
	0.12	mean 0.61

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Table 4. RESULTS OF RECOVERY STUDIES FOR PCBs

PCB Type	Percent Recovery
Aroclor 1242	105
Aroclor 1260	85
Aroclor 1260	122

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RESULTS AND DISCUSSION

PCBs were detected in soils at all three sampling locations. The elution patterns obtained most closely matched Aroclor 1242, Aroclor 1260 and decachlorobiphenyl. Soil concentrations ranged from the detection limit of 0.001 ppm to over 20 ppm.

Data from Vicinity of Yates Manufacturing Co., Chicago, IL.

Figure 2 illustrates: a) a chromatogram of Fenclor DK, the foreign supplied PCB presently used by Yates Mfg. Co. in the production of investment casting wax compounds; b) a chromatogram of a standard Aroclor 1260 and; c) a typical chromatogram of a soil extract taken in the vicinity of the site.

Figure 2a demonstrates that Fenclor DK is nearly pure decachlorobiphenyl. This compound is unusually pure for a technical grade material. One minor peak elutes just before the predominate decachlorobiphenyl peak. The retention time of the minor contaminant peak is such that it may be expected to be an octachlorobiphenyl since it exhibits the same retention time as the latest eluting peak in a standard Aroclor 1260 mixture. It is apparent by comparison of peak retention times with the sample chromatogram, Figure 2c, that decachlorobiphenyl is present in the soil sampled. The measured concentration of decachlorobiphenyl in this particular sample was 1.0 ppm. The presence of decachlorobiphenyl in this sample has been confirmed by mass spectrometry. Figure 3 illustrates a portion of the mass spectrum of the sample and a standard decachlorobiphenyl obtained from a different source.

The sample elution pattern also indicates the presence of Aroclor 1260 as can be seen by comparison with the Aroclor 1260 elution pattern.

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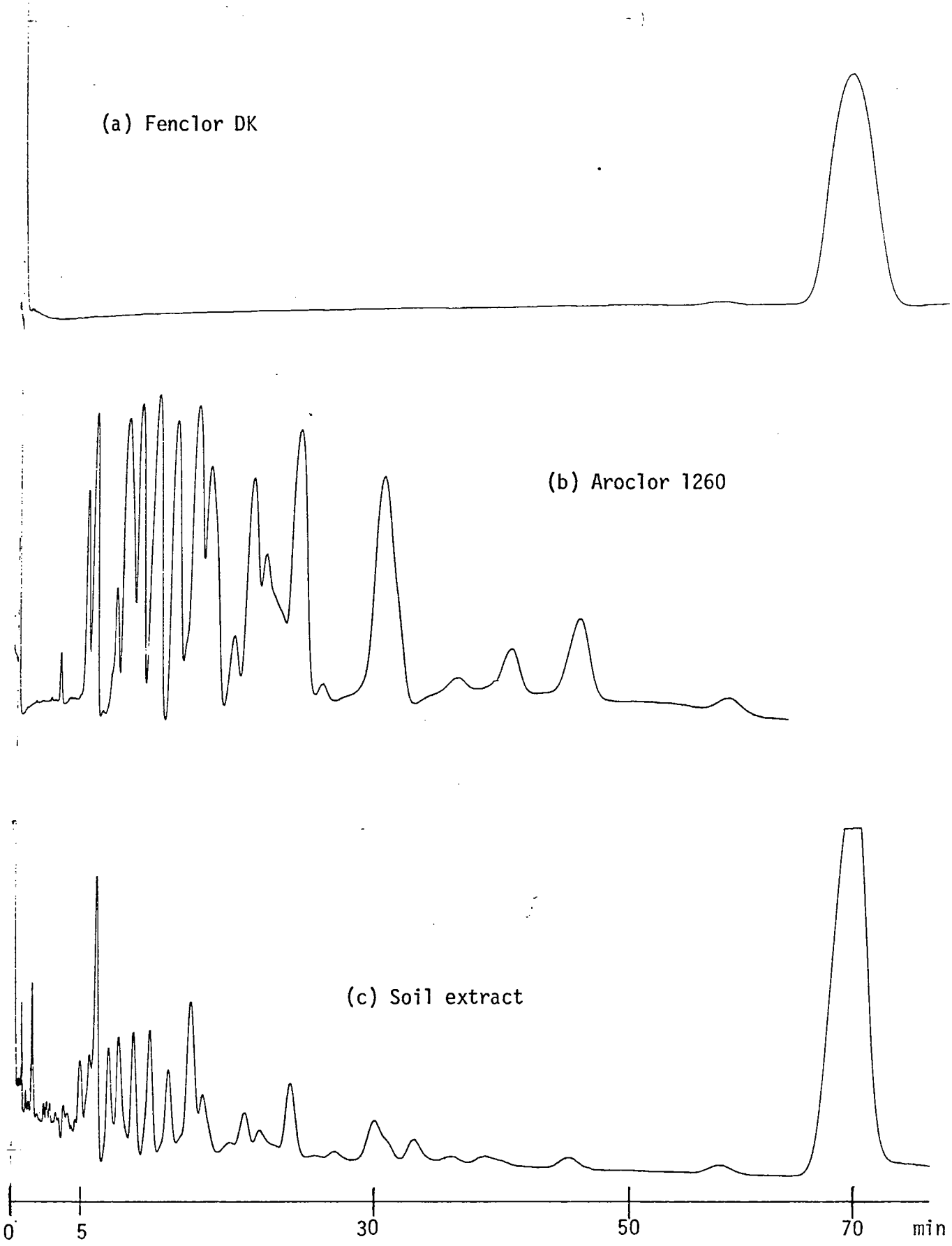


Figure 2. Typical chromatograms of standard PCBs and of a soil sample taken in the vicinity of Yates Mfg. Co., Chicago, Illinois.

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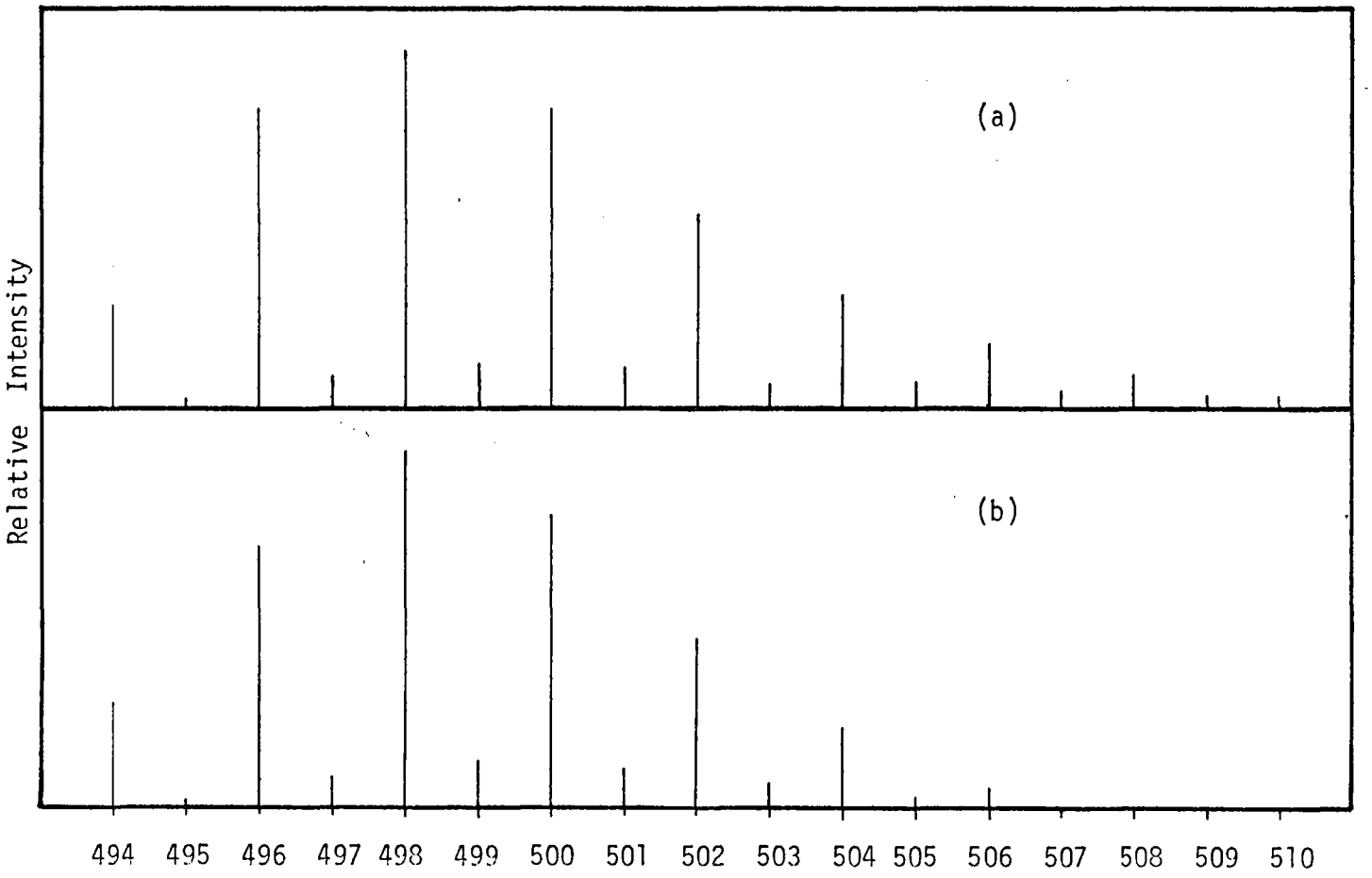


Figure 3. (a) Portion of mass spectrum of sample taken in vicinity of Yates Mfg. Co.
(b) Parent molecule mass cluster of decachlorobiphenyl.

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The concentration of Aroclor 1260 in this sample is 0.53 ppm. There is no indication of degradation of the Aroclor 1260 since virtually all peaks in the sample chromatogram are present in the same relative proportion as in the standard. There are no significant, undefined peaks in the chromatogram following the acid cleanup procedure. Certain of the sample chromatograms indicate the presence of lower weight PCBs, however the quantities present tend to be far less than either Aroclor 1260 or decachlorobiphenyl at this site.

Figure 4 depicts the quantity of Aroclor 1260 measured in samples taken in the vicinity of Yates Mfg. Co. There exists no apparent spatial pattern of Aroclor 1260 distribution over the study site. There is little to indicate the source of the Aroclor 1260 contamination is Yates Mfg.Co. Aroclor 1260 has not, in fact, been used by Yates. Prior to 1972 Aroclors 5442, 5460, and 6090 were purchased by this company. Subsequent to 1972, Fenclor DK and Aroclor 5460 have been in use. Aroclors 5442 and 5460 are terphenyl mixtures with 42 percent and 60 percent chlorine by weight, respectively. Aroclor 6090 is 90 percent terphenyl and 10 percent Aroclor 1221. Aroclor 1221 does not appear in the soils at this site.

Figure 5 illustrates the distribution of decachlorobiphenyl in soils near the Yates facility. The distribution has some elements of a pattern of higher levels closer to the Yates facility with decreasing concentration with distance. Such a pattern may be expected if Yates were the source of the decachlorobiphenyl (or Fenclor DK) contamination.

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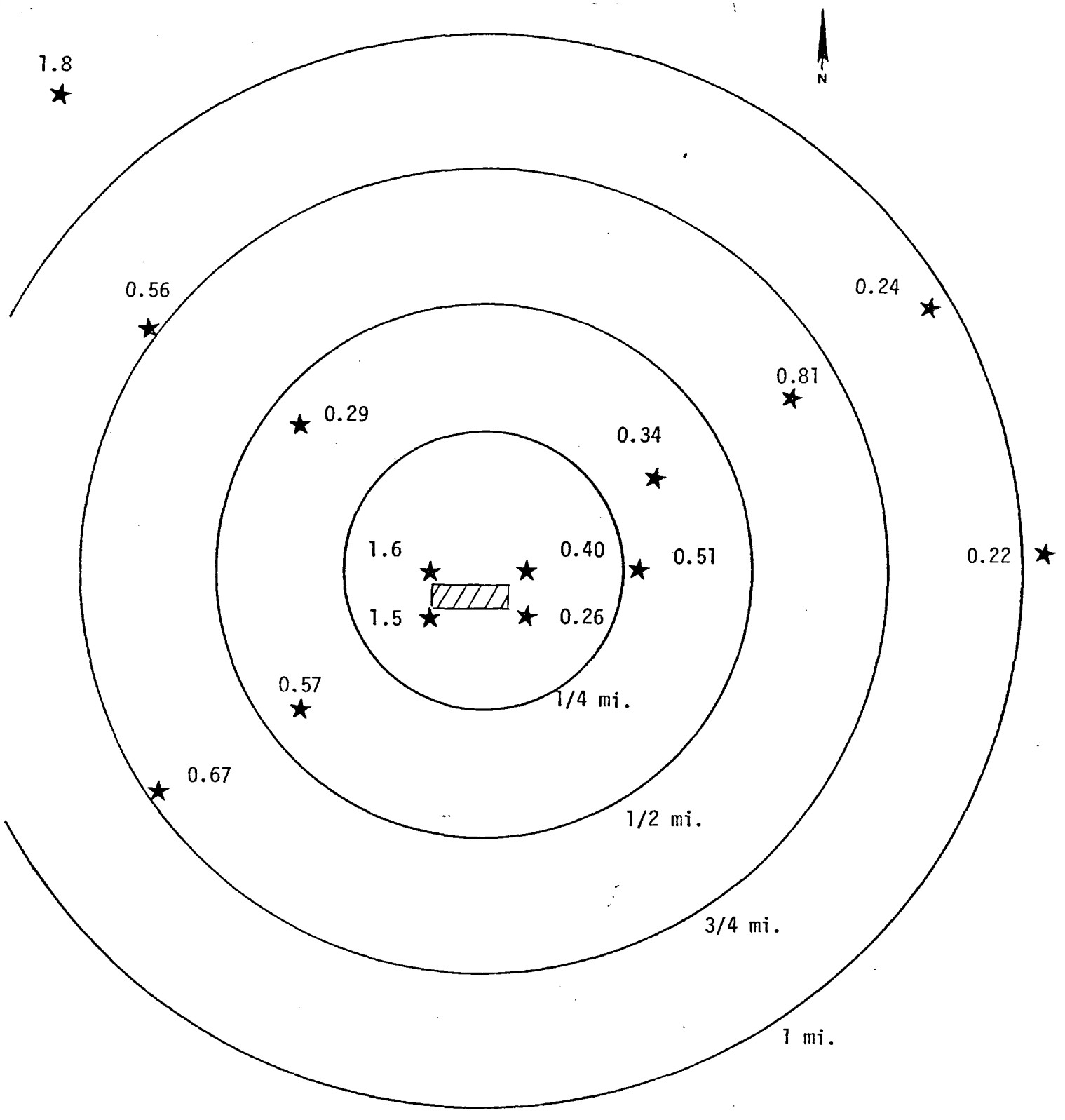


Figure 4. Concentration of Aroclor 1260 in soil as a function of distance from Yates Mfg. Co. Concentrations are expressed in ppm.

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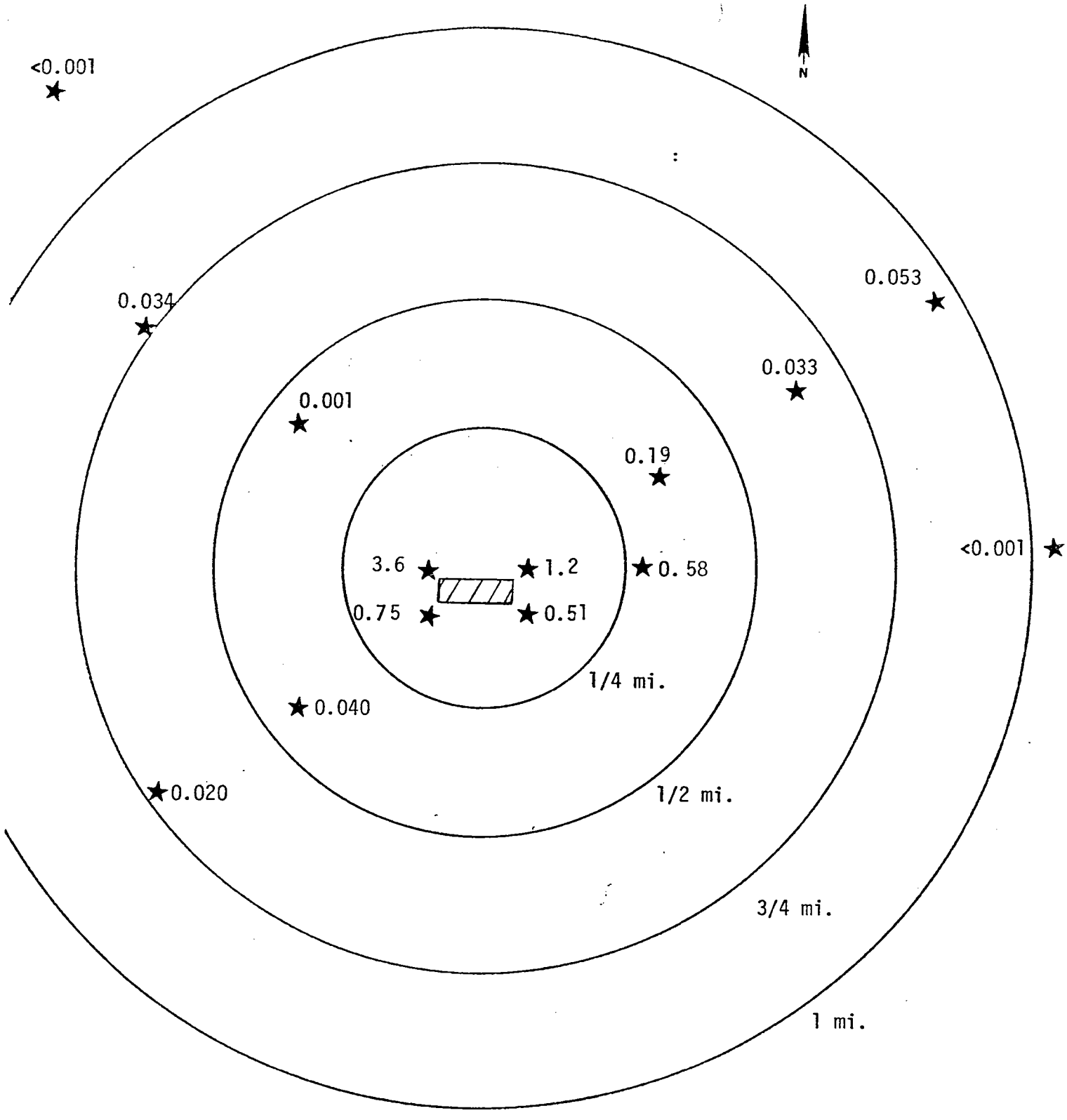


Figure 5. Concentration of decachlorobiphenyl in soil as a function of distance from Yates Mfg. Co. Concentrations are expressed in ppm.

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Data from Vicinity of Valcast Corp., Troy, MI.

Valcast Corp. is an investment casting facility located in a small industrial park in suburban Detroit, MI. PCBs are a constituent of the wax mold compound used to fashion intricate shapes which are to be cast. Figure 6 is a typical chromatogram of a soil sample extract taken from this location. Aroclor 1260 is present in many of the soil samples. Decachlorobiphenyl is absent in the soils. Figure 7 shows the concentrations of Aroclor 1260 observed in the vicinity of Valcast Corp. No distribution pattern is evident. The one observed concentration of 18 ppm Aroclor 1242 appears to be anomalously high, however, replicate analysis yielded values of 16 ppm and 19 ppm, respectively. Aroclor 1242 was not detected in any other soil samples from this area.

A small drainage ditch passes adjacent to the north boundary of the Valcast facility. This ditch serves to remove stormwater runoff in the vicinity and receives discharged cooling water from Valcast and other local small industries. Analysis of the Valcast cooling water at the point of discharge and of water in the drainage ditch failed to detect PCB levels greater than the detection limit of 0.1 µg/l. Two bottom sediment samples taken from this drainage ditch, however, had concentrations as follows:

Aroclor 1242	2.3 ppm	9.4 ppm
Aroclor 1260	6.7 ppm	8.9 ppm
Decachlorobiphenyl	0.09 ppm	0.11 ppm

Three grab samples of water and bottom sludge were taken from the sanitary sewer system about 1/4 mile down stream of the Valcast facility.

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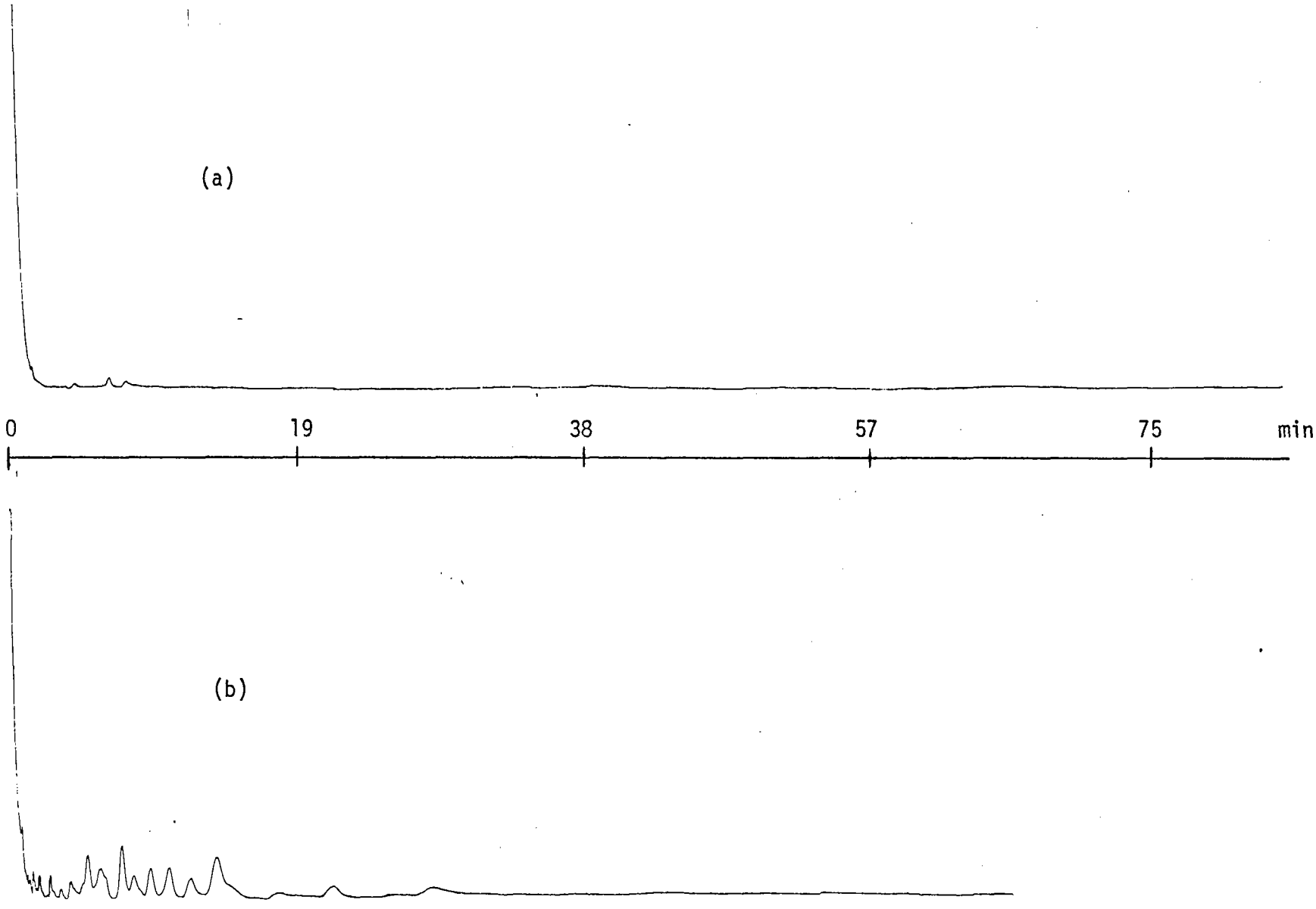


Figure 6. Typical chromatograms of soil samples taken in the vicinity of Valcast Corp., Troy, Michigan--(a) <0.01 ppm, (b) 0.014 ppm Aroclor 1260

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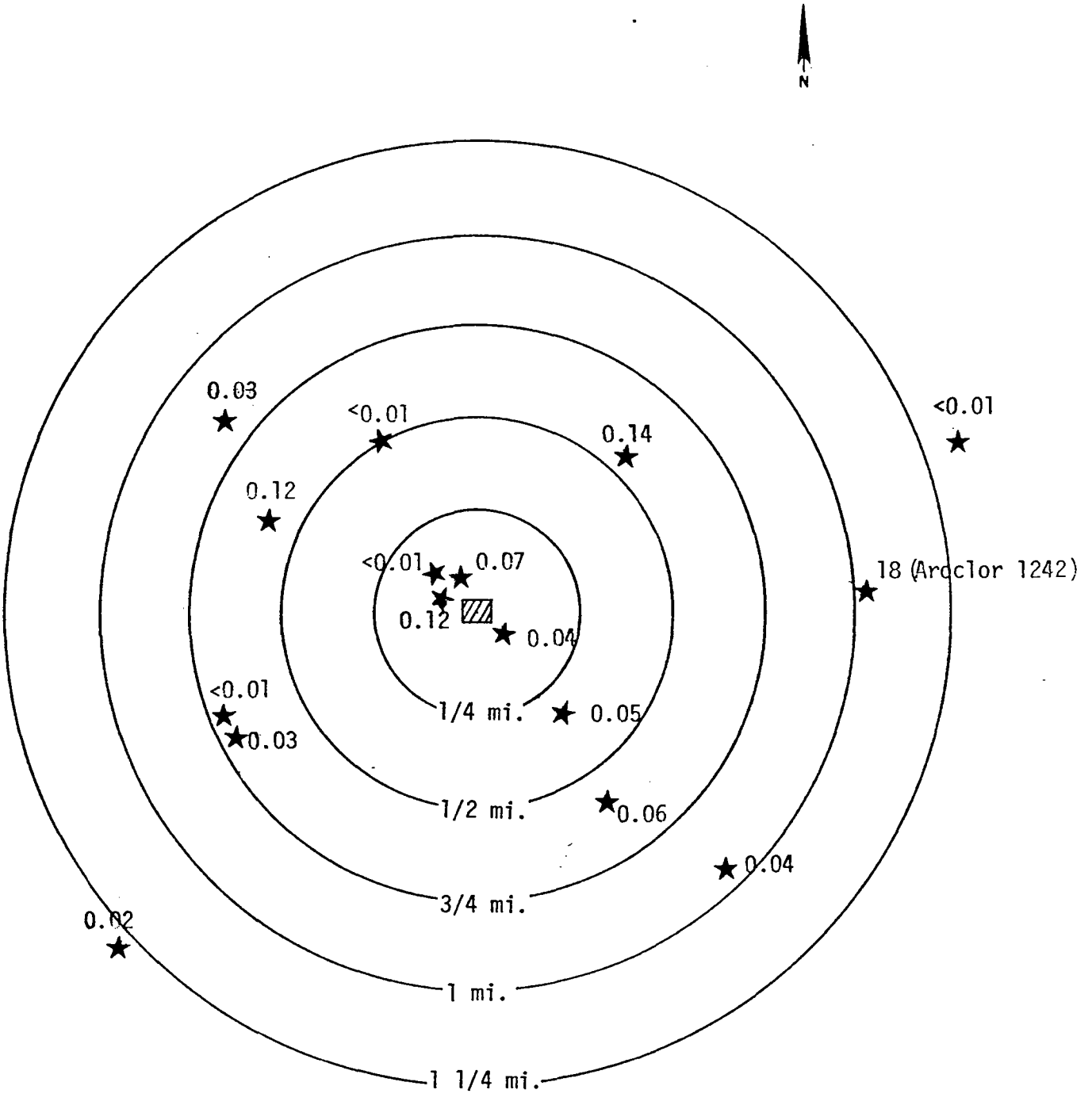


Figure 7. Concentration of Aroclor 1260 in soil as a function of distance from Valcast Mfg. Co. Concentrations are expressed in ppm.

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Table 5. ANALYTICAL RESULTS OF SAMPLES TAKEN FROM THE SANITARY SEWER SYSTEM OF TROY, MICHIGAN, AT INTERSECTION OF ELLIOT AND EXECUTIVE DRIVE.

Date	Time	Type Sample	Aroclor 1260	Decachlorobiphenyl
28 Feb 75	1530	water	<0.1 µg/l	<0.01 µg/l
22 Feb 75	1815	water	<0.1 µg/l	<0.01 µg/l
23 Feb 75	1145	water	7.0 µg/l	4.1 µg/l
22 Feb 75	--	bottom sludge	0.11 ppm	0.034 ppm

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This sewer system serves numerous small industries in the area. Results of the analysis of these samples are tabulated in Table 5. It appears that some Aroclor 1260 and decachlorobiphenyl are finding their way into the sewer system. Insufficient information is available to identify the source of these PCBs.

Data from Vicinity of Monsanto Co., Sauget, IL.

Aroclors have been produced by Monsanto Co. at the Sauget, Illinois site since prior to 1957. PCB mixtures ranging from 16 percent chlorine to 68 percent chlorine have been produced over the years. Production of the higher percent chlorine mixtures has been voluntarily reduced since 1970 and lower substituted isomers have been introduced. Polychlorinated terphenyls and chlorinated benzenes have also been produced at this facility. It is reported (Papageorge, 1975) that terphenyl production was suspended in 1971.

A typical chromatogram of a soil sample taken in the vicinity of the Monsanto facility is shown in Figure 8 along with the chromatogram of several other PCBs run under the same instrument conditions. This particular soil sample contains 11 ppm Aroclor 1242, 9.3 ppm Aroclor 1260 and 1.0 ppm decachlorobiphenyl. Lighter chlorinated Aroclor mixtures are likely present as well. The instrument conditions employed, however, do not give adequate separation of these mixtures to permit accurate quantification. The presence of decachlorobiphenyl at the concentrations observed is interesting. Decachlorobiphenyl has been reported as a component of only one Aroclor mixture, Aroclor 1268 (Hutzinger, Safe and Zitko, 1974). Whether it may be a component of any of the terphenyl mixtures or biphenyl/terphenyl mixtures or a by-product is not known.

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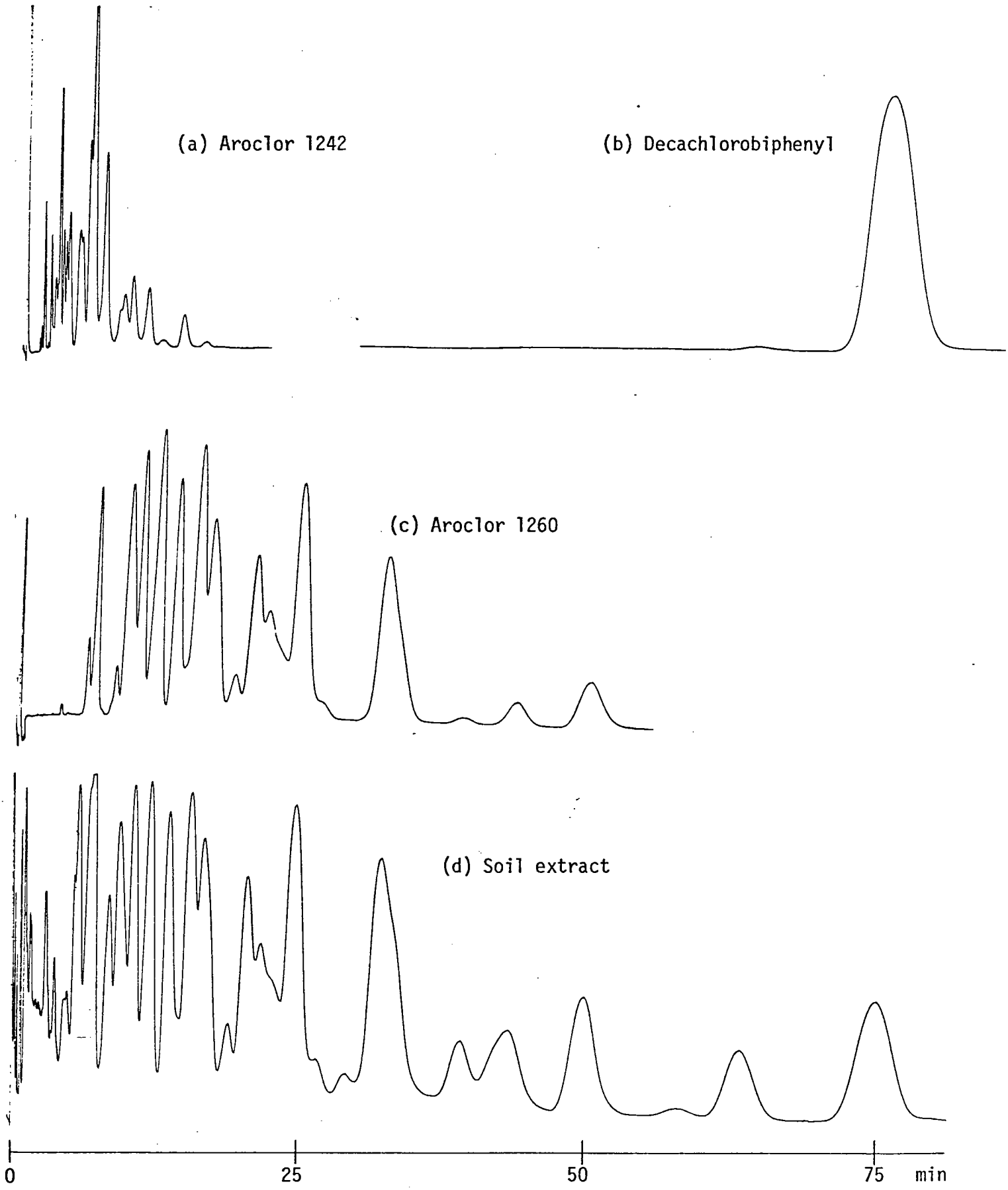


Figure 8. Typical chromatograms of standard Aroclors, decachlorobiphenyl and a soil sample taken in the vicinity of Monsanto Co., Sauget, Illinois.

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The measured soil concentration of Aroclor 1242, Aroclor 1260 and decachlorobiphenyl in the vicinity of the Monsanto facility are depicted in Figures 9, 10 and 11, respectively. Figure 12 shows the long-term average wind direction in the area. The distribution of all PCBs analyzed appears to be higher near the plant site and generally decreasing with distance from the site. Furthermore, there is evidence that generally higher concentrations are present in the soils located to the southeast. This corresponds to the predominant wind direction and may suggest an airborne transport of the PCBs from the facility.

One 15 cm deep core sample was taken 1/2 mile southwest of the Monsanto site. Analysis of the bottom 11 cm of this soil yielded PCB concentrations basically equivalent to the top 4 cm.

A 24-hour composite sample from the influent and the effluent of the Sauget village sewage treatment plant taken beginning 0800 on February 17, 1975, was provided by plant personnel. The Monsanto Co. jointly owns this facility. Analysis of these samples were as follows:

	Aroclor 1242	Aroclor 1260	Decachlorobiphenyl
influent	6.8 µg/l	8.7 µg/l	<0.01 µg/l
effluent	4.5 µg/l	4.7 µg/l	<0.01 µg/l

The treatment plant effects a reduction of 34 percent Aroclor 1242 and 46 percent Aroclor 1260. No decachlorobiphenyl was detected.

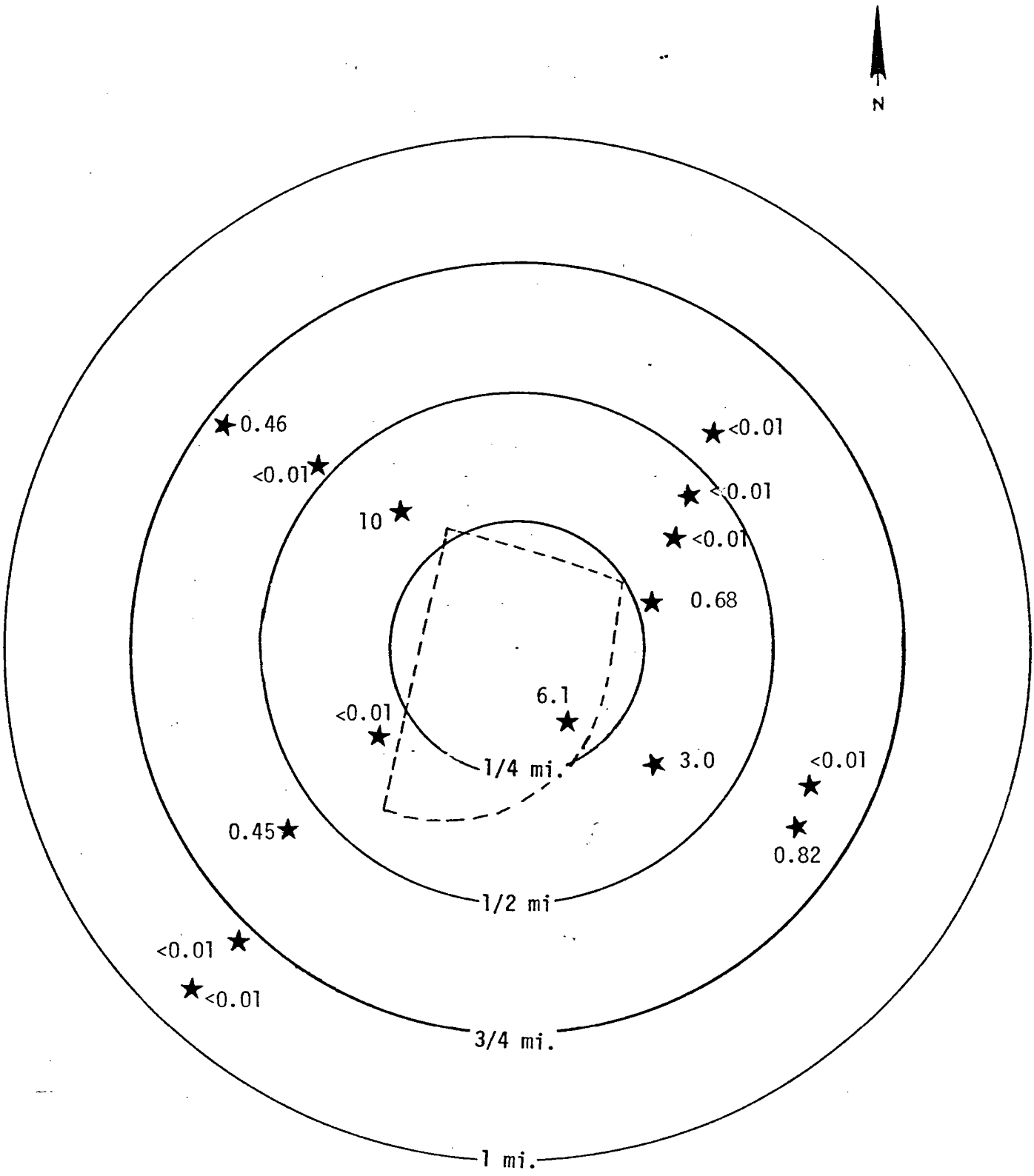


Figure 9. Concentration of Aroclor 1242 in soil as a function of distance from Monsanto Mfg. Co. Concentrations are expressed in ppm.

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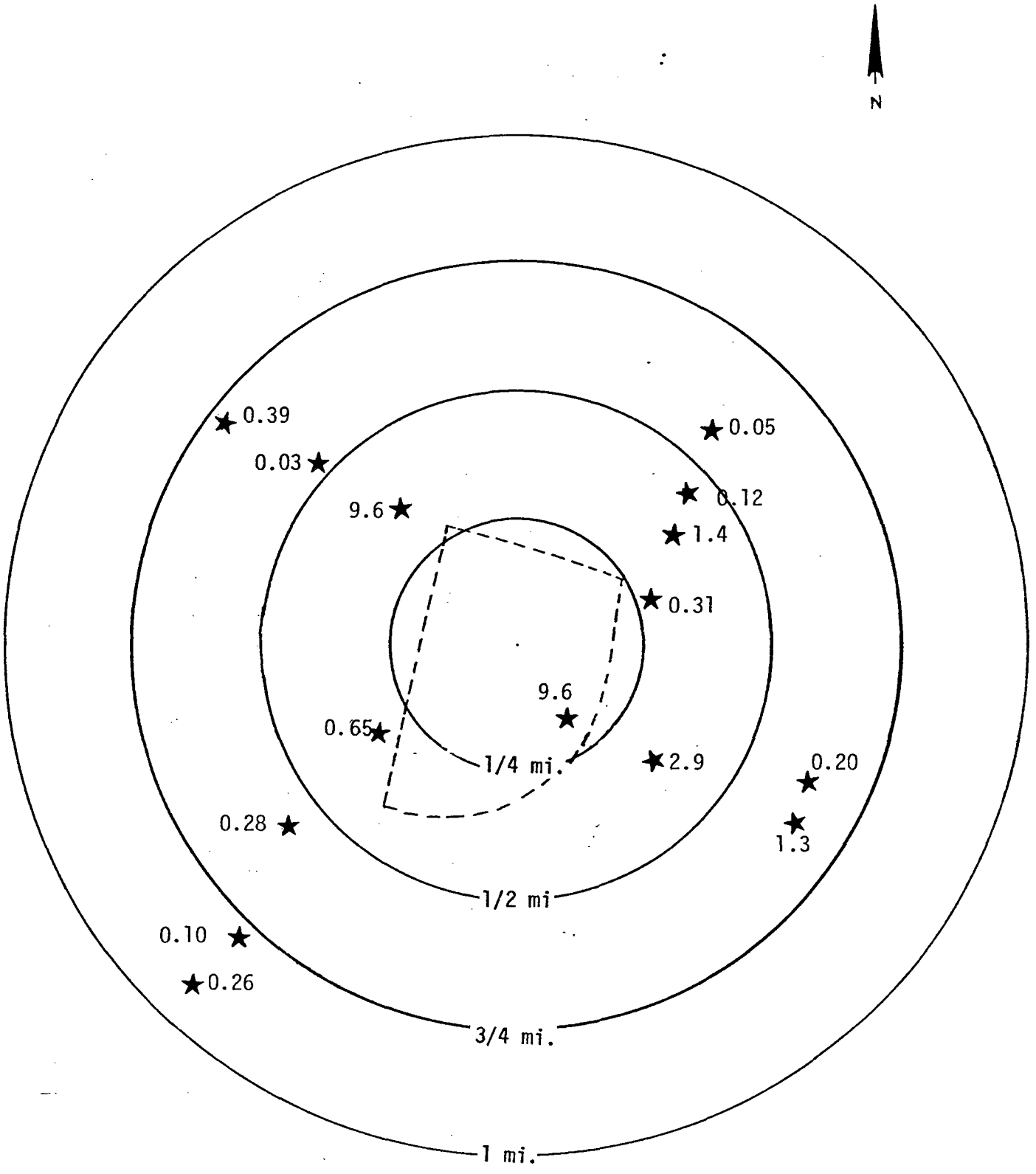


Figure 10. Concentration of Aroclor 1260 in soil as a function of distance from Monsanto Mfg. Co. Concentrations are expressed in ppm.

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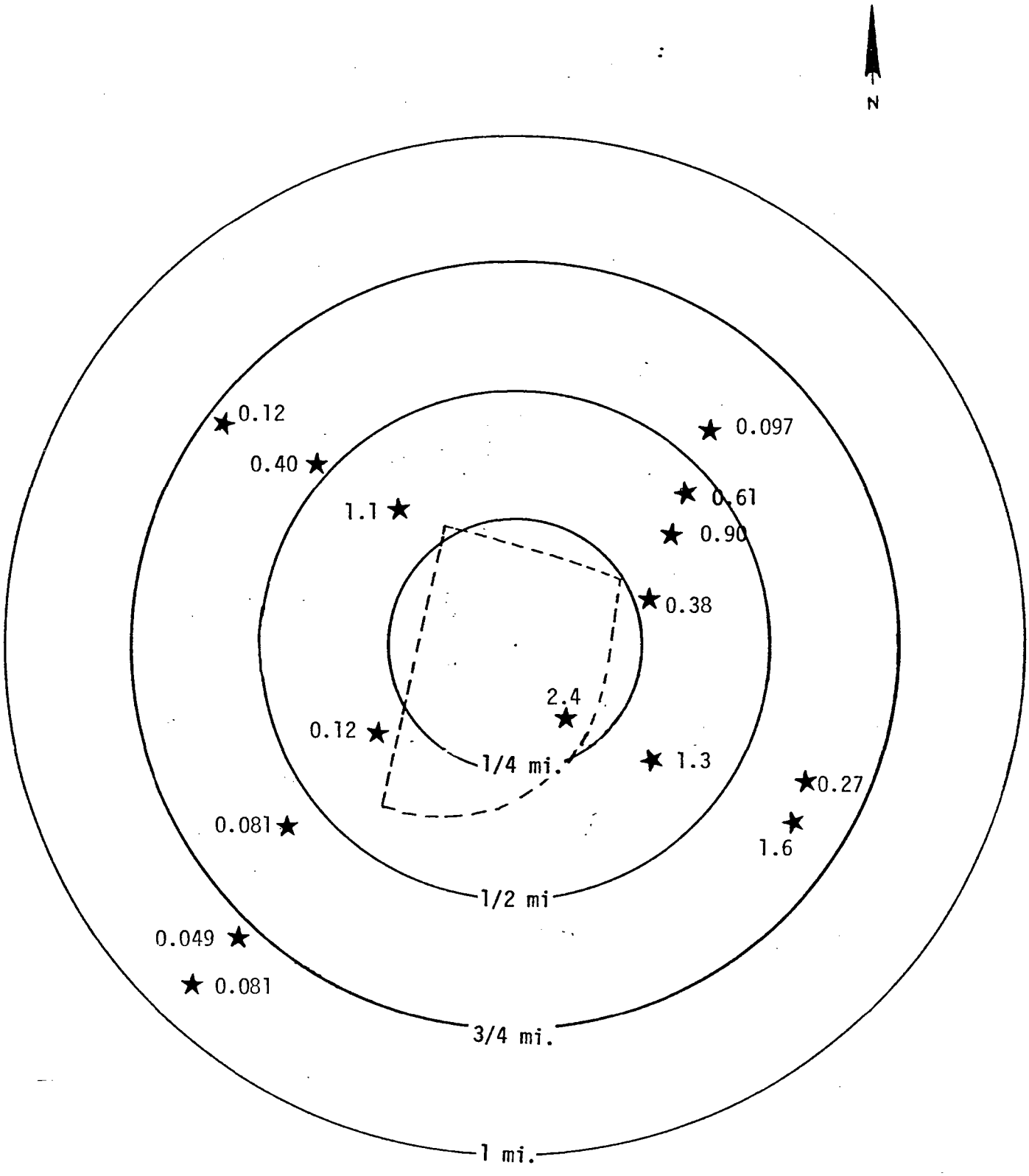


Figure 11. Concentration of decachlorobiphenyl in soil as a function of distance from Monsanto Mfg. Co. Concentrations are expressed in ppm.

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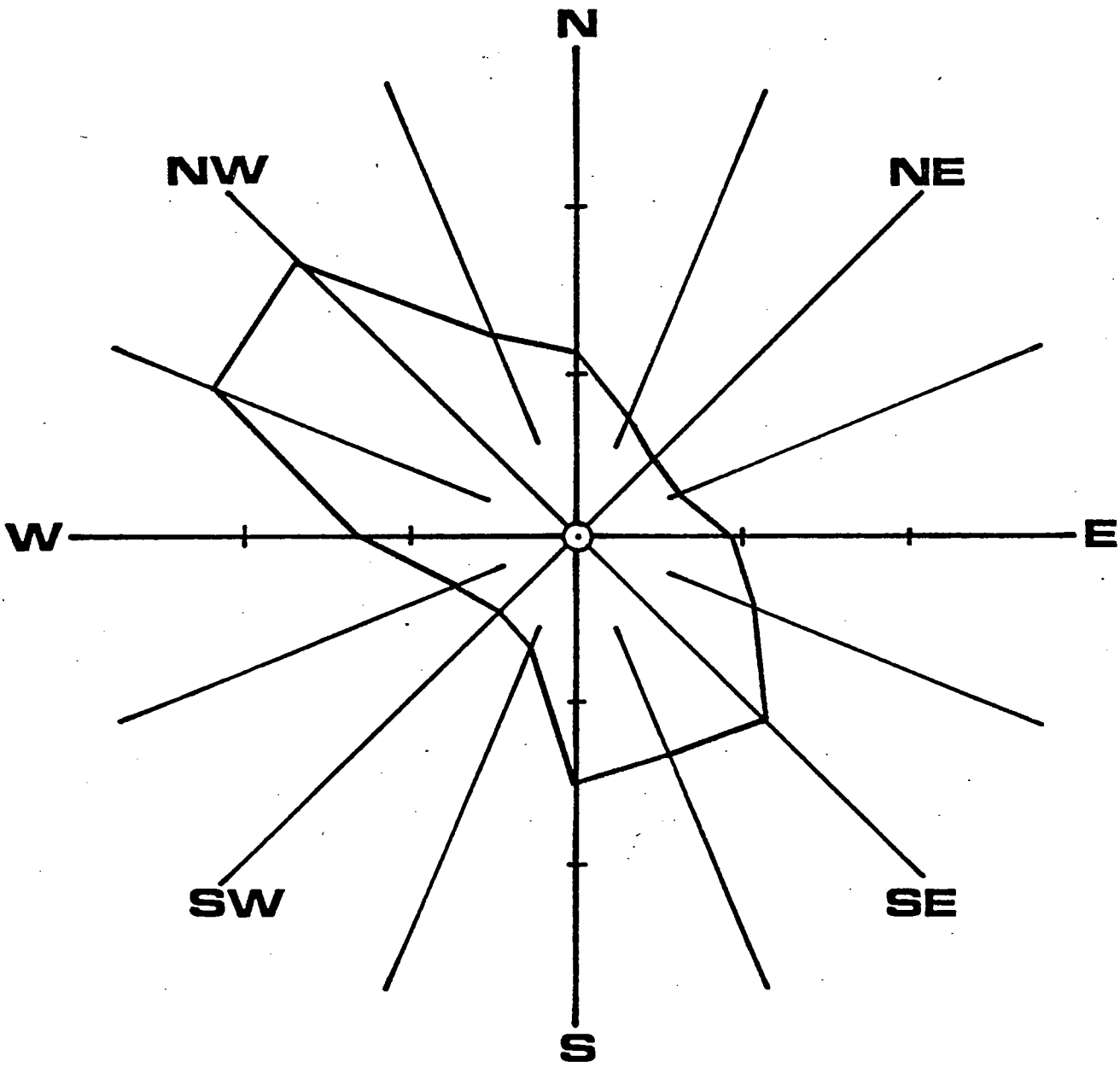


Figure 12. Ten Year Averaged Wind Rose for St. Louis, Missouri, January through March, 1951-1960.

Scale: 1" = 5% Calm Winds = 2.0%

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CONCLUSIONS

1. The distribution of Fenclor DK suggests that this compound may be transported to soils in the vicinity of Yates Mfg. Co. by way of airborne emissions.

2. Aroclor 1260 is present in urban Chicago soils. There is no evidence Yates Mfg. Co. is the source of this soil contamination.

3. Detectable, but lower levels, of Aroclor 1260 are present in soils in Troy, Michigan. There is no evidence Valcast Corp. is the source of the soil contamination. No decachlorobiphenyl (or Fenclor DK) was detected in soils in the area.

4. Significant quantities of Aroclor 1242 (18 ppm) were observed in a single soil sample taken in a residential area near Valcast Corp. The importance of this single data point should not be over-emphasized. However, Aroclor 1242, Aroclor 1260 and decachlorobiphenyl were also detected in drainage ditch sediments at much higher concentrations than the observed PCB levels for soils in the area.

5. The drainage ditch sediments near Valcast Corp. contained detectable levels of decachlorobiphenyl. Its source is unknown. A number of small industries are located in the immediate vicinity, any of which could be the source as well as intermittent discharge, leakage or runoff from the Valcast site or facility.

6. One of three grab samples taken over a period of time from the sanitary sewer system near Valcast Corp. contained detectable levels of Aroclor 1260 (7.0 µg/l and decachlorobiphenyl (4.1 µg/l). The other two samples were below detection limits of 0.01 µg/l and 0.001 µg/l, respectively. Sewer sludge contained both Aroclor 1260 and decachlorobiphenyl. These

materials are apparently discharged to the sewer system on an intermittent basis. The source is unknown.

7. Decachlorobiphenyl is relatively abundant compared to the other PCBs in soils in the vicinity of the Monsanto facility. Decachlorobiphenyl is not reported to be a major component of any of the Aroclor mixtures. It may be a by-product associated with Aroclor production which is eliminated or greatly reduced in the commercial preparations by refinement at the Monsanto facility and may, therefore, find its way into the environment at this location.

8. Aroclors 1242 and 1260 were also present in soils near the Monsanto plant. There appears to be a tendency for higher PCB levels close to the plant with decreasing concentration with distance. There is some evidence that higher soil concentrations exist along the northwest to southeast axis which corresponds to the dominate wind direction at this location. This suggests airborne transport of the PCBs from the plant with subsequent deposition, of at least the heavier isomers of PCB, on nearby soils.

9. PCBs are present in Sauget, Illinois wastewaters. The sewage treatment plant removes less than 50 percent of the Aroclor 1242 and Aroclor 1260. Decachlorobiphenyl is not present in these wastewaters.

10. Aroclor 1260 was found to be present in the soils of all three sites sampled. This product is currently used in electrical transformer fluids and has been used in the past in hydraulic fluids, plasticizers and dedusting agents (Hutzinger, Safe and Zitko, 1974). Aroclor 1242

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was found in comparable concentration near the Monsanto facility and in drainage ditch sediments near Valcast. This product has consistently been the most heavily produced by Monsanto of all the PCB mixtures.

11. Decachlorobiphenyl may be more widely spread than would be anticipated by projection from Monsanto production figures for Aroclors.

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SUGGESTIONS FOR CONTINUED STUDY OF PCBs

1. Conduct total PCB analysis employing the perchlorination technique of selected samples on hand. Determine what fraction of the total PCB present has been quantified as Aroclors 1242 and 1260 and decachlorobiphenyl.

2. Conduct polychlorinated terphenyl (PCT) analysis of selected samples on hand.

3. Assess PCB and PCT levels in various urban and non-urban soils and stream sediments.

4. Conduct analysis of airborne and precipitation-borne PCB and PCT in the vicinity of known users.

5. Evaluate PCB and PCT levels in and near dumps and landfills in the air, water, soil and groundwaters.

6. Investigate the potential for chlorination of biphenyl at normal wastewater and drinking water treatment levels.

7. Investigate the possibility of chlorinated dibenzofuran, dioxin and naphthalene contamination of various foreign and domestic PCB products.

8. Evaluate the rate of degradation of PCBs in the environment.

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